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HANFORD ENGINEER WORKS TECHNICAL MANUAL

SECTION C - SEPARATIONS

The uranium metal, as discharged from the piles in the 100 Areas, contains the alpha emitting product, plutonium, in concentration in the neighborhood of 150 - 250 grams per metric ton, along with similar amounts of beta and gamma fission elements. It is the purpose of the Separations Plant to effect the separation of this product from the uranium metal and fission elements, and to prepare a concentrated, relatively pure solution of plutonium nitrate as the final product of the Hanford Plant.

This section of the manual discusses the chemistry of the separations process, describes the buildings and equipment provided for carrying out the various steps in the operation, and presents the detailed operating procedures used. There are included, in many instances, references to other documents presenting a more detailed view of a specific point in the process.

The material included in this section was prepared between April and November, 1945. Some discrepancies may be found due to the differences in viewpoint and process at the times of preparation of the various chapters. Material revised subsequent to November 1945 is indicated by the letter R with the date in the lower left hand corner of the page. Where a new page has been added the letter I is used with the date.

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HANFORD ENGINEER WORKS TECHNICAL MANUAL

SECTION C - SEPARATIONS

CHAPTER I - FUNDAMENTAL TECHNICAL BASIS FOR THE SEPARATIONS PROCESS

Since many of the concepts employed in the Hanford process are relatively new, there are given in this chapter brief summaries of the technical relationships of the process. These include basic nuclear physics, the pile reactions, radioactivity, shielding, basic chemistry of the materials employed, etc. A brief summary of the separations process is given also.

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FUNDAMENTAL NUCLEAR PHYSICS

Atomic Structure

According to the current conception of atomic structure, the atom consists of neutrons, protons, and electrons. The neutrons and protons form an assemblage called the nucleus around which the electrons move in orbits determined by electrical forces. Schematic drawings of the structure of several atoms, indicating the components of the nucleus and the orbital paths of the electrons are shown in Figure 1.

The weight of a neutron is very nearly equal to that of a proton, each having essentially unit weight in the atomic scale. In comparison, the weight of an electron is almost negligible, being only 1/1840 that of a proton or neutron. Each proton carries one positive charge and each electron one negative charge. Neutrons are electrically neutral. The electrical attraction between the positively charged protons of the nucleus and the negatively charged electrons of the orbits holds the atom together.

The forces which act between the orbital electrons and the nucleus are the familiar electrical forces of attraction between unlike charges and repulsion between like charges. This type of force does not, however, explain the cohesiveness of a nucleus which contains a number of positively charged massive protons, in a volume only 10^{-12} times as large as the volume of the entire atom. These nuclei are held together by forces which are peculiar to nuclei; strong attractive forces exist between similarly charged protons, between electrically neutral neutrons, and between neutrons and protons within the nuclear volume.

The diameter of an atom is approximately 10,000 times as great as the diameter of its nucleus. Because of this fact and the extreme smallness of the electrons, it is obvious that an atom is mostly empty space with minute electrons revolving about an extremely dense nucleus. It might be noted that the atomic diameter of the common isotope of uranium (atomic weight 238) is approximately 10^{-8} centimeters and the nuclear diameter approximately 10^{-12} centimeters.

The nucleus is characterized by the atomic number and mass number. The atomic number, Z, is a direct expression of the number of positive charges in the nucleus and is therefore equal to the number of protons. The mass number, A, is numerically equal to the total number of particles in the nucleus, that is, the sum of the neutrons plus protons. The absolute mass, M, of an atom is very nearly equal to the mass number.

The number of electrons in a neutral atom determines the chemical properties of the element. Because the number of protons is equal to the number of electrons, one may say also that the number of protons in the nucleus determines the chemical properties of the element. It is possible to have different numbers of associated neutrons and therefore different absolute masses, M, in atoms of the same element. These different species of the same

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FIGURE 1

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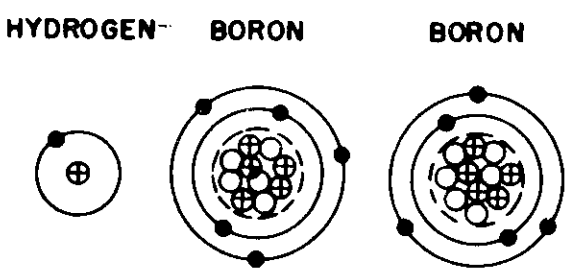
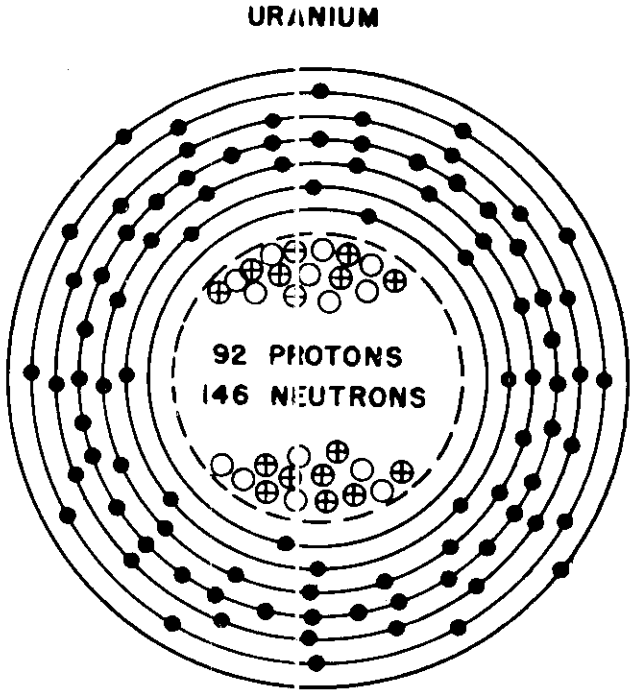
COMPONENTS OF ATOMS



(THE NUCLEUS IS SHOWN EXAGGERATED IN SIZE RELATIVE TO ELECTRON ORBITS)



SEVEN ORBITS CONTAINING A TOTAL OF 92 ELECTRONS

DESIGNATION	ELEMENT	HYDROGEN	BORON	BORON	URANIUM
Z	NUMBER OF PROTONS	1	5	5	92
A-Z	NUMBER OF NEUTRONS	0	5	6	146
A	MASS NUMBER	1	10	11	238
Z	ATOMIC NUMBER	1	5	5	92
	SYMBOL	$_{1}H^{1}$	$_{5}B^{10}$	$_{5}B^{11}$	$_{92}U^{238}$

[redacted]

element are call... The atomic weight, W, of a given element is dependent on the r... diance of its different isotopes. The mass numbers and relative abu... e known naturally-occurring isotopes are given in Table I. The at... of an element may be precisely calculated if the relative abundan... ute mass, M, or each isotope is known.

Since the n... tons in various isotopes of a given element are exactly the same ... al properties of isotopes are identical. Therefore, they cannot ... ated by ordinary chemical methods but only by procedures which take ... of the difference in physical properties due to difference in abs... of the atoms.

Artificial Transmutation

Nuclear reactions which change the atomic number, that is the number of protons in the nucleus, actually convert one element into another. Such changes can be accomplished by bombarding the nucleus with suitable projectiles such as neutrons, protons, deuterons and alpha particles. A deuteron is the nucleus of the heavy hydrogen atom containing one neutron and one proton. An alpha particle is the nucleus of a helium atom containing two neutrons and two protons. With the exception of neutrons, all of the aforementioned projectiles are positively charged, and tend to be repelled by the positively charged protons of the nucleus at which they are aimed. Therefore, it is difficult to introduce them into the nucleus unless they are moving at extremely high velocities and with great kinetic energy. However, most "atom smashers" such as the cyclotron are based on the principle of imparting extremely high velocities to these positively charged particles and using them as projectiles to enter the nucleus by impact.

Also, it has been found that neutrons are very effective in entering nuclei. This is due to the fact that they carry no free electric charge and consequently are not repelled by the positively charged protons of the nuclei with which they come in contact. As a result it is not necessary that the neutrons be at a high velocity. In fact, it is essential in the present application that they have an extremely low velocity.

Types of Nuclear Reactions

The most important types of nuclear reactions involving neutrons are the following:

a) Impact without capture of the incident neutron.

   1) Elastic impact, with no emission of particles or radiations.

   2) Inelastic impact accompanied by emission of a radiation called gamma rays. These rays are somewhat similar to X-rays and are emitted by the excited nucleus as a means of disposing of the energy acquired in the collision.

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TABLE I

MASS NUMBERS AND ABUNDANCE OF ISOTOPES OCCURRING IN NATURE

Z	Element	Sym.	A (percent abundance)	Atomic Weight
0	Neutron	n	1(---)	
1	Hydrogen	H	1(99.98), 2(0.02)	1.0080
2	Helium	He	$3(10^{-5})$, 4(~100)	4.003
3	Lithium	Li	6(7.5), 7(92.5)	6.940
4	Beryllium	Be	9(100)	9.02
5	Boron	B	10(18.4), 11(81.5)	10.82
6	Carbon	C	12(98.9), 13(1.1)	12.01
7	Nitrogen	N	14(99.62), 15(0.38)	14.008
8	Oxygen	O	16(99.757), 17(0.039), 18(0.204)	16.000
9	Fluorine	F	19(100)	19.000
10	Neon	Ne	20(90.00), 21(0.27), 22(9.73)	20.183
11	Sodium	Na	23(100)	22.997
12	Magnesium	Mg	24(77.4), 25(11.5), 26(11.1)	24.32
13	Aluminum	Al	27(100)	26.97
14	Silicon	Si	28(89.6), 29(6.2), 30(4.2)	28.06
15	Phosphorus	P	31(100)	30.98
16	Sulfur	S	32(95.1), 33(0.74), 34(4.2), 36(0.016)	32.06
17	Chlorine	Cl	35(75.4), 37(24.6)	35.457
18	Argon	A	36(0.307), 38(0.061), 40(99.632)	39.944
19	Potassium	K	39(93.38), $40(4 \times 10^{-8})$, 41(6.61)	39.096
20	Calcium	Ca	40(96.96), 42(0.64), 43(0.15), 44(2.06), 46(0.0033), 48(0.19)	40.08
21	Scandium	Sc	45(100)	45.10
22	Titanium	Ti	46(7.95), 47(7.75), 48(73.45), 49(5.51), 50(5.34)	47.90
23	Vanadium	V	51(100)	50.95
24	Chromium	Cr	50(4.49), 52(83.78), 53(9.43), 54(2.30)	52.01
25	Manganese	Mn	55(100)	54.93
26	Iron	Fe	54(6.04), 56(91.57), 57(2.11), 58(0.28)	55.85
27	Cobalt	Co	59(100)	58.94
28	Nickel	Ni	58(67.4), 60(26.7), 61(1.2), 62(3.8), 64(0.88)	58.69
29	Copper	Cu	63(70.13), 65(29.87)	63.57
30	Zinc	Zn	64(50.9), 66(27.3), 67(3.9), 68(17.4), 70(0.5)	65.38
31	Gallium	Ga	69(61.2), 71(38.8)	69.72
32	Germanium	Ge	70(21.2), 72(27.3), 73(7.9), 74(37.1), 76(6.5)	72.60
33	Arsenic	As	75(100)	74.91
34	Selenium	Se	74(0.9), 76(9.5), 77(8.3), 78(24.0), 80(48.0), 82(9.3)	78.96
35	Bromine	Br	79(50.6), 81(49.4)	79.916
36	Krypton	Kr	78(0.35), 80(2.01), 82(11.53), 84(57.11), 86(17.47)	83.7
37	Rubidium	Rb	85(72.8), 87(27.2)	85.48
38	Strontium	Sr	84(0.56), 86(9.86), 87(7.02), 88(82.56)	87.63
39	Yttrium	Y	89(100)	88.92
40	Zirconium	Zr	90(48), 91(11.5), 92(22), 94(17), 96(1.5)	91.22
41	Columbium	Cb	93(100)	92.91
42	Molybdenium	Mo	92(14.9),94(9.4),95(16.1),96(16.6),97(9.65),98(24.1),100(9.25)	95.95
43	Masurium	Ma		--
44	Ruthenium	Ru	96(5.68),98(2.22),99(12.81),100(12.70),101(16.98),102(31.34),104(18.27)	101.7
45	Rhodium	Rh	103(100)	102.91

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Z	Element	Sym.	A(Percent abundance)	Atomic Weight
46	Palladium	Pd	102(0.8), 104(9.3), 105(22.6), 106(27.2), 108(26.8), 110(13.5)	106.7
47	Silver	Ag	107(51.9), 109(48.1)	107.880
48	Cadmium	Cd	106(1.4),Cd(1.0),110(12.8),111(13.0),112(24.2),113(12.3),114(28.0),116(7.3)	112.41
49	Indium	In	113(4.5), 115(95.5)	114.76
50	Tin	Sn	112(1.1), 114(0.8), 115(0.4), 116(15.5), 117(9.1), 118(22.5), 119(9.8), 120(28.5), 122(5.5), 124(6.8)	118.70
51	Antimony	Sb	121(56), 123(44)	121.76
52	Tellurium	Te	120(< 1), 122(2.9),123(1.6),124(4.5),125(6.0),126(19.0),128(32.8),130(33.1)	127.61
53	Iodine	I	127(100)	
54	Xenon	Xe	124(0.094), 126(0.088), 128(1.90), 129(26.33), 130(4.07), 131(21.7), 132(26.96), 134(10.54), 136(8.95)	131.3
55	Caesium	Cs	133(100)	132.91
56	Barium	Ba	130(0.101),132(0.097),134(2.42),135(6.59),136(7.81),137(11.32),138(71.66)	137.36
57	Lanthanum	La	139(100)	138.92
58	Cerium	Ce	136(< 1), 139(< 1), 140(89), 142(11)	140.13
59	Praseodymium	Pr	141(100)	140.92
60	Neodymium	Nd	142(25.95),143(13.0),144(22.6),145(9.2),146(16.5),148(6.8),150(5.95)	144.27
61	Samarium	Sm	144(3),147(16.1),149(15.5),150(11.6),152(20.7),154(18.9)	150.43
62	Europium	Eu	151(49.1), 153(50.9)	152.0
64	Gadolinium	Gd	152(0.2),154(1.5),155(18.4),156(19.9),157(18.9),158(20.9),160(20.2)	156.9
65	Terbium	Tb	159(100)	159.2
66	Dysprosium	Dy	158(> 0.1),160(1),161(21.1),162(26.6),163(24.8),164(27.3)	162.46
67	Holmium	Ho	165(100)	164.94
68	Erbium	Er	162(0.1),164(1.5),166(32.9),167(24.4),168(26.9),170(14.2)	167.2
69	Thulium	Tm	169(100)	169.4
70	Ytterbium	Yb	168(0.06),170(4.21),171(14.26),172(21.49),173(17.02),174(29.58),176(13.38)	173.04
71	Lutecium	Lu	175(97.5), 176(2.5)	174.99
72	Hafnium	Hf	174(0.18),176(5.30),177(18.47),178(27.13),179(13.85),180(35.14)	178.6
73	Tantalum	Ta	181(100)	180.88
74	Tungsten	W	180(~0.2),182(22.6),183(17.3),184(30.1),186(29.8)	183.92
75	Rhenium	Re	185(38.2), 187(61.8)	186.31
76	Osmium	Os	184(0.018),186(1.59),187(1.64),188(13.3),189(16.1),190(26.4),192(41.0)	190.2
77	Iridium	Ir	191(38.5),193(61.5)	193.1
78	Platinum	Pt	192(0.8),194(30.2),195(35.3),196(26.6),198(7.2)	195.23
79	Gold	Au	197(100)	197.2
80	Mercury	Hg	196(0.15),198(10.1),199(17.0),200(23.3),201(13.2),202(29.6),204(6.7)	200.61
81	Thallum	Th	203(29.1), 205(70.9)	204.39
82	Lead	Pb	204(1.5), 206(23.6), 207(22.6), 208(52.3)	207.21
83	Bismuth	Bi	209(100)	209.00
84	Polonium	Po		(210)
85	Alabamine	Ab		(221)
86	Radon	Rn		222
87	Virginium	Vi		(224)
88	Radium	Ra		226.05
89	Actinium	Ac		(229)
90	Thorium	Th	232(100)	232.12
91	Protactinium	Pa		231
92	Uranium	U	234(0.006), 235(0.71), 238(99.28)	238.07

[redacted]

b) Capture of the incident neutron followed by emission of a nuclear particle (e.g. proton, alpha particle or neutron).

c) Simple capture of the incident neutron without emission of a nuclear particle but with emission of gamma rays. Where an unstable isotope is produced, the nucleus tends to stabilize itself by a process known as radioactive decay, in which an electron, called a beta particle or beta ray, is emitted. Gamma rays often times are emitted simultaneously with the beta particles.

d) Capture of the incident neutron followed by immediate fission or splitting of the nucleus into two new nuclei, accompanied by the emission of several free neutrons. Beta particles and gamma rays are also emitted due to the radioactive decay of newly created unstable nuclei.

Mass and Energy Relationships

The study of nuclear reactions is entirely parallel to the more familiar study of chemical reactions. One meets such fundamental concepts as the equation of the reaction, the process of balancing the equation with respect to quantities which are conserved in the reaction, and the rates of reaction. In nuclear physics the nucleus is expressed by a symbol which consists of the chemical abbreviations for the element with the atomic number as a pre-subscript and the mass number as the post-superscript, for example:

$$\overset{238}{\underset{92}{U}} \quad ; \quad \overset{10}{\underset{5}{B}} \quad ; \quad \overset{1}{\underset{1}{H}}$$

In this manual, the pre-subscript is usually omitted.

Two different notations are common in expressing the equation of a reaction. For example, the reaction between a neutron and a boron nucleus ($_5B^{10}$) can be written in the following ways:

$$\underset{\text{boron}}{{}_5B^{10}} + \underset{\text{neutron}}{{}_0n^1} \longrightarrow \underset{\text{lithium}}{{}_3Li^7} + \underset{\substack{\text{helium} \\ \text{(alpha particle)}}}{{}_2He^4} + Q \text{ energy}$$

or

$$_5B^{10}(n,\alpha)\ {}_3Li^7$$

In the latter method of writing the reaction, the first symbol within the parenthesis indicates the projectile and the last symbol indicates the emitted particle.

In every nuclear reaction the total number of particles, protons plus neutrons, is conserved. The total electrical charge is also conserved. For example, in the above reaction the balance is as follows:

	Left Side	Right Side
Total number of neutrons and protons	10 + 1	7 + 4
Total charge	5 + 0	3 + 2

As in the case of chemical reactions, the energy balance of a nuclear reaction provides an indication of the ease with which the reaction can be made to proceed. The reaction is exoergic or endergic, by analogy with chemical reactions, according to whether the energy, Q, of the reaction is positive or negative. In the energy balances of nuclear reactions there is a very striking equivalence between mass and energy which is not detected in chemical reactions because of the relatively small energies involved in chemical as compared with nuclear processes. The energy unit of nuclear physics is the electron-volt, abbreviated ev. The electron-volt is generally defined as the amount of energy which a singly charged particle, for example a proton or electron, has acquired after falling through a potential difference of one volt. The unit has acquired this form because of the convenience of expressing the energy of artificially accelerated atomic projectiles in terms of the voltage used to accelerate them. This unit of energy is now universally used in nuclear physics even when artificial acceleration is not involved. In many problems the unit electron-volt is inconveniently small. For this reason the million-electron-volt unit, abbreviated as Mev, and equal to 10^6 ev, is very often used. The comparative size of the electron-volt and the more familiar units of energy is presented in the following tabulation:

	per particle	per mol (6.02x10^{23} particles)
1 ev	1.6×10^{-12} erg	9.65×10^{11} ergs/mol
1 Mev	1.6×10^{-6} erg	9.65×10^{17} ergs/mol
1 Mev	1.18×10^{-13} ft.-lbs.	7.1×10^{10} ft.-lbs./mol
1 Mev	3.83×10^{-17} kg.-cal.	23.00×10^{6} kg.-cal./mol
1 Mev	4.45×10^{-20} KW-hrs.	2.68×10^{4} KW-hrs./mol

Energy of thermal agitation of molecules is approximately 1/40 ev.
Energy of alpha particles from naturally radioactive elements is about 5 Mev.
Energy of fastest alpha particles accelerated by cyclotron is about 40 Mev.
Energy released in the fission of a uranium nucleus is approximately 200 Mev.

The equivalence of mass and energy, referred to above, is a development of the theory of relativity. It is expressed by the relation:

$$E = mc^2$$

where E, is the energy in ergs; m, the mass in grams; and c, the velocity of light.

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Mass can be converted into energy and energy into mass. When mass disappears in a reaction, it appears as energy of the reaction. The equivalence between energy in electron-volts and mass in atomic units is set forth in the following relation:

One absolute mass unit (per mol) = 931 Mev

or

1 Mev = 0.00107 absolute mass units (per mol)

Energy equivalent of the mass of an electron (per mol) = 0.51 Mev

As an example of the use of the mass-energy equivalence relation, it is applied to predict the energy release of the neutron-boron reaction. The reaction is:

$$_5B^{10} + {}_0n^1(\text{slow}) \longrightarrow {}_3Li^7 + {}_2He^4 + \text{Energy}$$

Absolute mass of atoms (per mol)

	10.01605	1.00893	7.01804	4.00389
Total	11.02490	⟶	11.02193	

Difference 0.00305 units of atomic weight

Therefore, the predicted energy of reaction = $\frac{0.00305}{0.00107}$ = 2.85 Mev

The predicted energy release of 2.85 Mev agrees with the experimentally observed energies of the product nuclei.

Radioactivity

According to present theory, atomic nuclei are composed entirely of neutrons and protons. In stable nuclei the ratio of the number of neutrons to the number of protons falls in a fairly well defined and rather narrow range of values. If for some reason the ratio falls outside of this range, the nucleus is unstable. Nuclear reactions involve changing the composition of nuclei. Very frequently the product nucleus is unstable. An unstable nucleus is said to be "radioactive". It tends to adjust its neutron-proton ratio with energy emission to acquire stability by a process called "radioactive decay". The time consumed in the adjustment process may vary from fractions of seconds to millions of years.

In the process of acquiring stability most radioactive materials emit either electrons or positrons (positively charged particles of mass similar to the electrons). However, many of the naturally radioactive, and some of

the artificially radioactive, elements emit alpha particles. The rate at which these particles are emitted is known as the activity of the material; when the rate is high, the activity is said to be high and vice versa. Each radioactive species has a characteristic rate at which its activity decreases. The rate of decay is usually expressed in terms of the time required for the activity to decrease by one-half. This time is called the half-life of the species. Half-lives of artificially radioactive species range from seconds to years. A chart of the three series of natural radioactivities and important related isotopes artificially produced is shown as Figure 2.

The activity of a radioactive material is expressed in terms of the number of disintegrations of its atoms which occur each second. When the number of disintegrations per second is equal to 3.7×10^{10}, which is the number of disintegrations produced per second by one gram of radium, the sample is said to have one curie of activity.

NUCLEAR REACTIONS IN URANIUM

Nuclear Fission

A reaction which results in the splitting of a nucleus into two approximately equal parts is called fission. One of the most important fission reactions occurs in uranium nuclei. In the most prominent reaction leading to fission in normal uranium, isotope 235 captures a neutron to form an excited compound nucleus U^{236}. This nucleus decays predominantly by fission but also by gamma emission, with the result that a small amount of rather stable (i.e. long-lived) U^{236} is formed. In the fission act the excited U^{236} nucleus behaves like a liquid drop of matter in vibration. If, during these vibrations, there is a sufficient separation of the two nuclear masses so that the electrostatic forces of repulsion between them exceed the cohesive forces of surface tension for the whole nuclear mass, then fission occurs. However, if the excess energy given to the nucleus upon capture of the neutron can be released as gamma radiation from the vibrating compound nucleus, then a condition of stability may be attained without the occurrence of fission. The sequence of events occurring in fission are illustrated in Figure 3.

The new nuclei produced by the fission reaction belong to elements in the middle of the atomic series. The importance and great value of this reaction results from two of its features. First, each fission is accompanied by the largest energy release yet obtained from a nuclear reaction. Second, the fission reaction releases neutrons in amounts sufficient to make the fission reaction self-sustaining when the conditions are properly controlled. The reactants and products of fission can be presented in the form:

Uranium nucleus + single neutron $\longrightarrow$ a) two new nuclei
b) several free neutrons
c) large amount of energy

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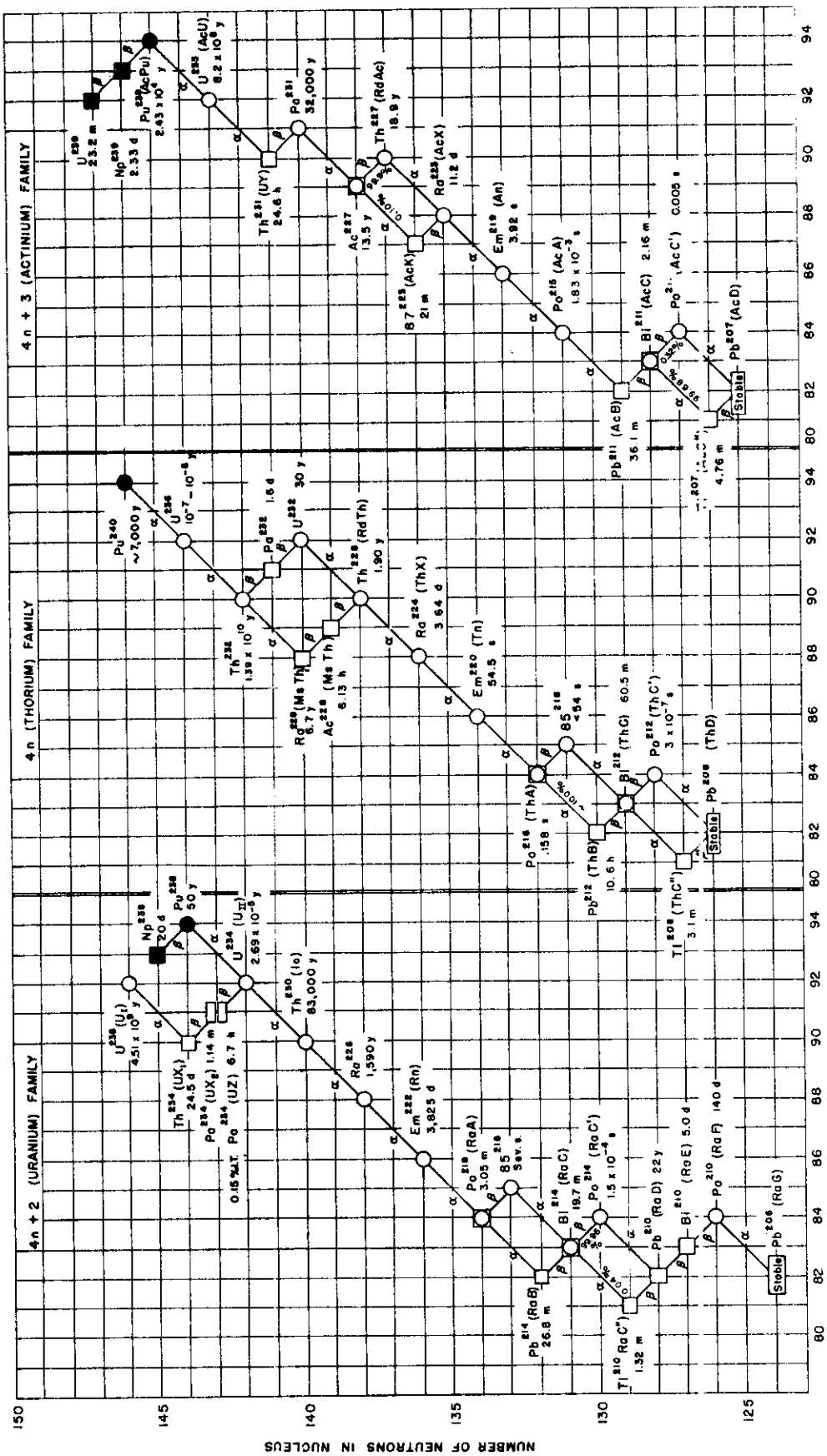
FIGURE 2

THE THREE SERIES OF NATURAL RADIOACTIVITIES
INCLUDING RELATED ISOTOPES ARTIFICIALLY PRODUCED



In the above scheme, an alpha disintegration is shown by the symbol α; and beta decay by the symbol β. The following abbreviations are used for denoting half-life values of the radio-isotopes: s-seconds, m-minutes, h-hours, d-days, and y-years. An isomeric transition is indicated by the abbreviation I.T. The classical or historical symbol for each radio-isotope is given in parenthesis as well as the modern designation, using the element symbol and mass number.

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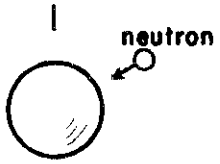
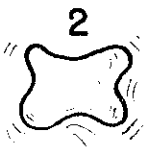
FIGURE 3

FISSION OF URANIUM



Uranium nucleus in normal spherical shape captures a neutron.

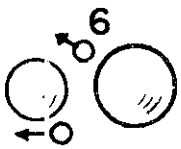


The energy imparted to the nucleus appears as motion of the entire droplet.

Violent oscillations may draw the droplet into a dumbell shape.

Variations in oscillations result in unequal distribution of mass. Electrical repulsion acts to push ends farther apart.

Fracture occurs resulting in two fission fragments.



The fragments attempt to acquire stability by emission of neutrons.

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At the instant of fracture the highly excited nuclear fragments have sufficient energy to eject neutrons. A typical reaction might be:

$$_{92}U^{235} + {}_{0}n^{1}\ (\text{slow}) \longrightarrow (_{92}U^{236}) \longrightarrow {}_{36}Kr^{94} + {}_{56}Ba^{142}$$

highly excited — highly excited

On the average, between two and three fast neutrons are emitted by the excited nuclei immediately on fission. The emission of these several neutrons for the single neutron captured makes self-sustained fission possible. This gives:

$$_{36}Kr^{94} + {}_{56}Ba^{142} \longrightarrow {}_{36}Kr^{92} + {}_{56}Ba^{141} + 3\ {}_{0}n^{1} + \text{Energy}$$

Even following neutron release, the products are unstable, due to the unsatisfactorily high neutron to proton ratios of these nuclei. The unstable krypton and barium nuclei decay by the emission of beta particles, resulted in conversion of neutrons to protons, in a so-called "fission decay chain". The decay continues from element to element until a stable isotope is reached. For example, the above unstable nuclei decay with beta and gamma emission in the following possible fission chains:

$$_{36}Kr^{92} \xrightarrow{\beta^-} \text{Rb} \longrightarrow \text{Sr} \xrightarrow{\beta^-} \text{Y} \xrightarrow{\beta^-} {}_{40}Zr^{92}\ (\text{stable})$$

$$_{56}Ba^{141} \xrightarrow{\beta^-} \text{La} \xrightarrow{\beta^-} \text{Ce} \xrightarrow{\beta^-} {}_{59}Pr^{141}\ (\text{stable})$$

The tremendous energy released in the fission process make the reaction important as a source of energy for special applications. The energy which is released arises from the excess mass of the original uranium nucleus and neutron over that of the final stable products, in accordance with the mass-energy equivalence. An estimate of the magnitude of the energy released per fission can be obtained by considering the reaction already referred to:

$$_{92}U^{235} + {}_{0}n^{1} \longrightarrow \left(\begin{array}{l}\text{fission and neutron}\\ \text{emission followed by}\\ \text{decay to stable products}\end{array}\right) \longrightarrow {}_{40}Zr^{92} + {}_{59}Pr^{141} + 3\,{}_{0}n^{1}$$

To compute the amount of mass which is converted to energy, the difference in the sums of the atomic weights is obtained.

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Atomic Weight of Original Particles		Atomic Weight of Final Particles	
$_{92}U^{235}$	235.1240	$_{40}Zr^{92}$	91.9420
$_{0}n^{1}$	1.0089	$_{54}Pr^{141}$	140.9590
		$_{0}n^{1}(3)$	3.0267
	236.1329		235.9277

Mass difference = 0.205 units

Energy released per fission = $\frac{0.205}{0.00107}$ = 190 Mev

(0.00107 Mass Unit = 1 Mev)

The estimated energy release is close to the accepted average release of 200 Mev per fission. Most of the energy appears as energy of motion of the large fission fragments. This energy is dissipated by heating of the stopping materials. The remainder of the energy appears as energy of the gamma rays, beta particles, and neutrons which accompany the process. The distribution of the energy among the various particles is as follows:

Kinetic energy of fission fragments	159 Mev
Gamma radiation from fission products	2[illegible] Mev
Beta radiation from fission products	1[illegible] Mev
Kinetic energy of neutrons	[illegible] Mev
Total	200 Mev

An interesting and important feature in the decay of the fission products is that, in the case of several of these products, the decay proceeds with emission of neutrons. Bromine and iodine have been identified as the source of two of the neutron activities. At least two other activities are present but their sources have not been identified. Neutron emission arises in those cases in which the excess energy of a nucleus is so great that the excess cannot be dissipated quickly enough by beta or gamma emission alone. The neutrons which are emitted in this fashion are called "delayed neutrons". The growth and decay of delayed neutron activity is very similar to that of beta activity.

Formation of Plutonium

Plutonium is formed from uranium by nuclear reactions involving isotope U^{238}. Thus, slow neutrons and resonance neutrons may be captured by this nucleus to give U^{239}, which decays by beta particle emission to element 93,

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or neptunium. The beta-unstable neptunium isotope, Np^{239}, then decays to element 94, which is called plutonium. The series of reactions leading to the formation of plutonium is given as follows:

$$_{92}U^{238} + {}_{0}n^{1} \longrightarrow {}_{92}U^{239} + \gamma$$

$$_{92}U^{239} \xrightarrow[t_{\frac{1}{2}}\ 23.5\ \text{min.}]{} {}_{93}Np^{239} + {}_{-1}\beta$$

$$_{93}Np^{239} \xrightarrow[t_{\frac{1}{2}}\ 2.33\ \text{days}]{} {}_{94}Pu^{239} + {}_{-1}\beta$$

Plutonium itself is radioactive and decays by alpha particle emission to uranium, according to the reaction

$$_{94}Pu^{239} \xrightarrow[t_{\frac{1}{2}}\ 24{,}300\ \text{years}]{} {}_{92}U^{235} + {}_{2}He^{4}$$

Since the half-life of Pu^{239} is 24,300 years, it is relatively stable and can be manufactured in quantity. However, to produce the material, an intense source of neutrons must be available, since 2.52×10^{23} neutrons are required for each gram of plutonium. Such a source of neutrons is provided by the excess neutrons from the fission of isotope U^{235}. Thus, the process for producing plutonium consists of providing the conditions necessary for slow neutrons to start the fission of isotope U^{235} and then to slow down the resultant high neutrons so that some of them enter the U^{238} nucleus while maintaining at least one new neutron each time to continue the fission of more U^{235} and so maintain the chain reaction. The structure in which the chain reaction is carried out is called a pile.

THE FISSION PRODUCTS

Formation and Fission Yield

The two nuclear fragments which are formed during the fission of a heavy isotope, such as U^{235}, are called fission products. The masses (and mass numbers) of these fragments vary considerably because of variations in asymmetry of the uranium nucleus at the instant of fission. In all cases, however, the sum of the mass numbers of the two fragments plus the liberated neutrons must total to 236, the mass number of the excited compound uranium nucleus. Thus, if two neutrons are liberated in a given fission reaction, one may find a large number of possible combinations of mass numbers, such as 160-76,

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159-75, 158-76 ·····140-94, 139-95, 138-96 ·····118-116, 117-117. The probability that any given combination of mass numbers will occur during fission is called the fission yield. Since the elemental composition of the fission products changes with time because of beta decay, the fission yield must be based on mass number rather than atomic number. The fission yield versus mass number curve, as obtained from the best experimental data at present (Aug. 1945), is shown in Figure 4. The center of symmetry in the curve is found at mass number 117, since it is assumed that, on the average, two neutrons are liberated in each fission. Thus, the mass numbers of the fission fragments from a given fission must total 234, while their atomic numbers must total 92.

In Figure 5 are listed all of the fission product activities which have been identified thus far in pile fission material. The decay of a given chain proceeds diagonally upward and to the left. The approximately 160 fission product activities occur in more than 60 decay chains, an average of 2.5+ members per chain. Theoretically, there should be, on the average, between 3 and 4 members per decay chain. The fact that the average chain length is only about 2.5 indicates that there are other radio-isotopes which have not yet been identified. Most of these activities, however, are very short-lived or very long-lived so that it is difficult to conduct experimental studies to identify them.

Activities Important in Plant Operations

In the nominally normal operation of the plant, the uranium metal slugs are irradiated in the piles for between 100 and 200 days and then are stored for a period of 40 to 60 days before being processed to extract plutonium. The variations in irradiation time are necessary at a given pile power level (normally, 250 megawatts) to take into account differences in neutron flux at different points in the pile. A cooling time of 40 to 60 days is normally used so that the gamma radiations from the principal activity, 2.33-day Np^{239}, is only a minor radiation hazard as compared to the longer-lived activities. Also, other short-lived activities decay to negligible levels.

Of the longer-lived activities which remain after the cooling period, only those listed in Table II are considered important because of half-life and/or fission yield.

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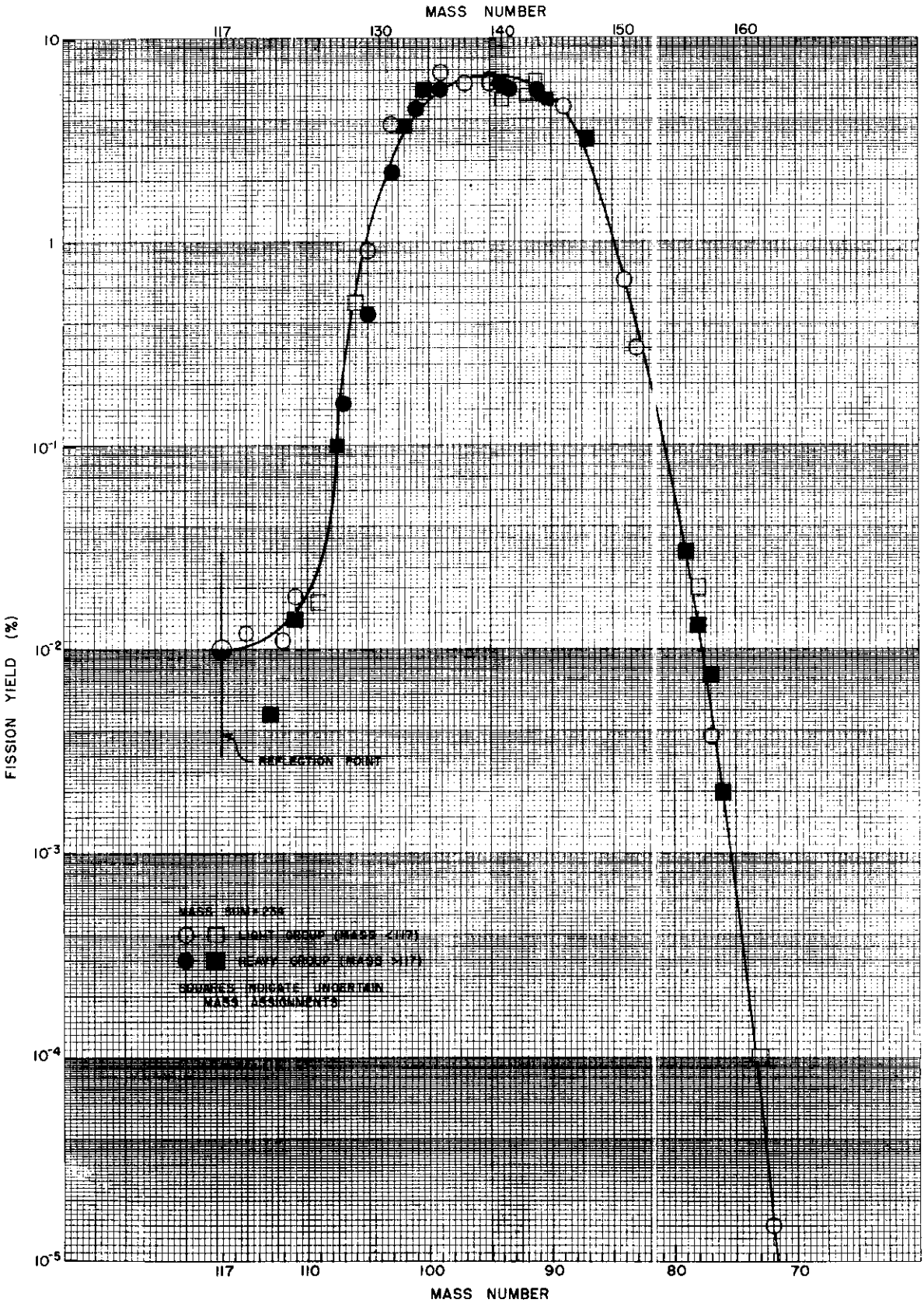
FIGURE 4

U^{235} FISSION YIELD – MASS CURVE



A CHART OF THE RADIO-ISOTOPES PRODUCED IN PILE FISSION.

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TABLE II

Fission Product Activities Important in Plant Operations After a 40 to 60 Day Cooling Period

Element	Isotope	Half-life	Fission Yield	Beta Ray Energy (Mev)	Gamma Ray Energy (Mev)
Strontium	Sr^{89}	55 days	4.6%	1.48	None
Strontium	Sr^{90}	>30 years	~5%	0.4	None
Yttrium	Y^{90}*	60 hours	~5%	2.16	None
Yttrium	Y^{91}	57 days	5.9%	1.4	None
Zirconium	Zr^{95}	65 days	~6%	1.0(97%)0.3(3%)	0.23, 0.75, 0.92
Columbium	Cb^{95}	35 days	~6%	0.154	0.775
Ruthenium	Ru^{103}	42 days	~3.7%	0.3(95%)0.8(5%)	0.56
Ruthenium	Ru^{106}	1 year	0.5%	very soft	None
Rhodium	Rh^{106}*	30 sec.	0.5%	4.0	None
Tellurium	Te^{129}	32 days	0.24%	---	0.086
Tellurium	Te^{129}*	72 min.	0.24%	1.9	---
Iodine	I^{131}	8 days	~1.9%	0.595	0.08,0.367
Xenon	Xe^{133}	5.5 days	~4.6%	0.42	~0.085
Cesium	Cs^{137}	~33 years	~5%	0.5(50%),0.84(50%)	0.5
Barium	Ba^{140}	12.5 days	6.1%	1.0(75%),0.4(25%)	0.5(25%)
Lanthanum	La^{140}*	40 hours	6.1%	1.8	0.3,1.69(>97%)2.5(<3%)
Cerium	Ce^{141}	28 days	5.7%	0.6	0.22
Praseodymium	Pr^{143}	13.8 days	5.4%	0.95	No. 8
Cerium	Ce^{144}	275 days	~5.0%	0.35	
Praseodymium	Pr^{144}*	17.5 min.	~5.0%	3.07	0.22, 1.25(~5%)

*Amount of activity due to radio-isotope is governed by decay of long-lived parent.

Activity and Heating Effects of Fission Products

During the operation of the pile there are 3×10^{10} fissions/second occurring for each watt of pile power. Thus, for a pile operating at 250 megawatts, there are $250 \times 10^{6} \times 3 \times 10^{10} = 7.5 \times 10^{18}$ fissions/second, 6.5×10^{23} fissions/day or a total of 1.3×10^{24} fission fragments/day. Since all of these fission products do not decay immediately to stable isotopes, radioactive isotopes accumulate in the pile metal. The rate of accumulation is governed by the pile power, and the total activity is a function of the time of operation. At any given moment of operation, however, the activity of the metal in the pile, as measured in curies, is not a simple function of the number of fission fragments which have been produced since: 1) the half life of each of the many radio isotopes governs its disintegration rate, as shown by the relation $dN/dt = 0.693N/T_{\frac{1}{2}}$, where dN/dt = disintegration rate per second, N = the number of atoms of fission fragments having a given mass number, A, and atomic number, Z, and $T_{\frac{1}{2}}$ = half-life of the fission fragment as specified above; 2) many radio-isotopes decay to daughter activities which contribute to the overall

[redacted]

disintegration rate. For example, the short-lived daughter activities of long-lived radio-isotopes listed in Table II have disintegration rates approximately equal to their parents at equilibrium and hence the long-lived parent activities appear to have about twice as much activity as radio-isotopes decaying in a simple manner.

The overall changes in radioactivity associated with metal which has been irradiated in a pile for various times and then removed is shown in Figure 6. The curves are useful in determining the amount of activity in metal to be processed. For example, 1 ton of average metal, in a 100-ton pile operated for 100 days at 250 megawatts and cooled for 50 days, should have $\frac{0.128 \times 250 \times 10^6}{100} = 3.2 \times 10^5$ curies of activity. The activity due to any one important fission product, as given in Table II, may be calculated from curves such as those shown in Figures 7 and 8. For example, in the case cited above, it was found that 320,000 curies of activity were present in 1 ton of metal after 50 days cooling. The amount of 65-day Zr present at this time, as shown by Figure 7, is 8.1% of the total number of curies present. Thus, there are $3.2 \times 10^5 \times 0.08 = 25{,}600$ curies of Zr^{95}. Figure 8 gives the limiting values for the intensity of the principal fission product activities following infinite irradiation since, at the end of such an irradiation, all fission product activities have an intensity proportionate to their fission yields.

Calculations of the heating effects due to the fission product activities are made from data concerning the energies and absorption characteristics of the beta and gamma radiations associated with them. The power developed by the fission product radiations, without considering their absorption in surrounding matter, may be obtained from the curves shown in Figures 9, 10, 11, 12, and 13. The heating effects may be calculated, knowing the power, absorption coefficients for beta and gamma radiations, specific heat and transfer coefficients for the surrounding materials, etc.

Weights of Fission Product Elements

The amount of any stable isotope formed directly in fission is proportional to the total number of fissions and the fission yield for that isotope. With radioactive isotopes, growth and decay of the activities is important in calculating their weights. For metal exposed at a power level of 2500 kilowatts/ton for 100 days and then cooled for 60 days, the weights of both active and inactive fission product isotopes are given in Table III.

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FIGURE 6

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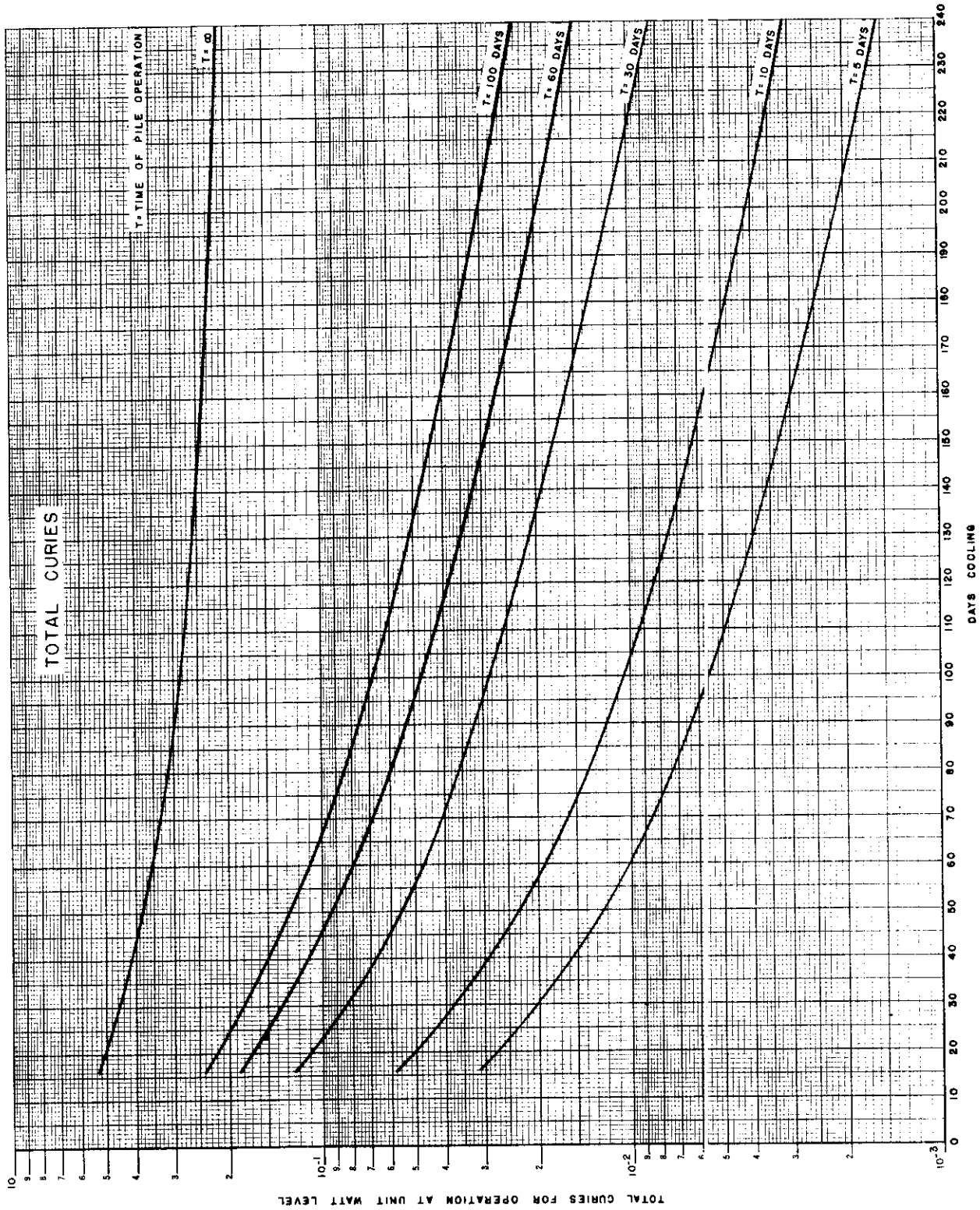

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FIGURE 7

FRACTIONS OF TOTAL CURIES DUE TO INDIVIDUAL RADIOACTIVE ISOTOPES FOLLOWING 100 DAYS OF PILE OPERATION

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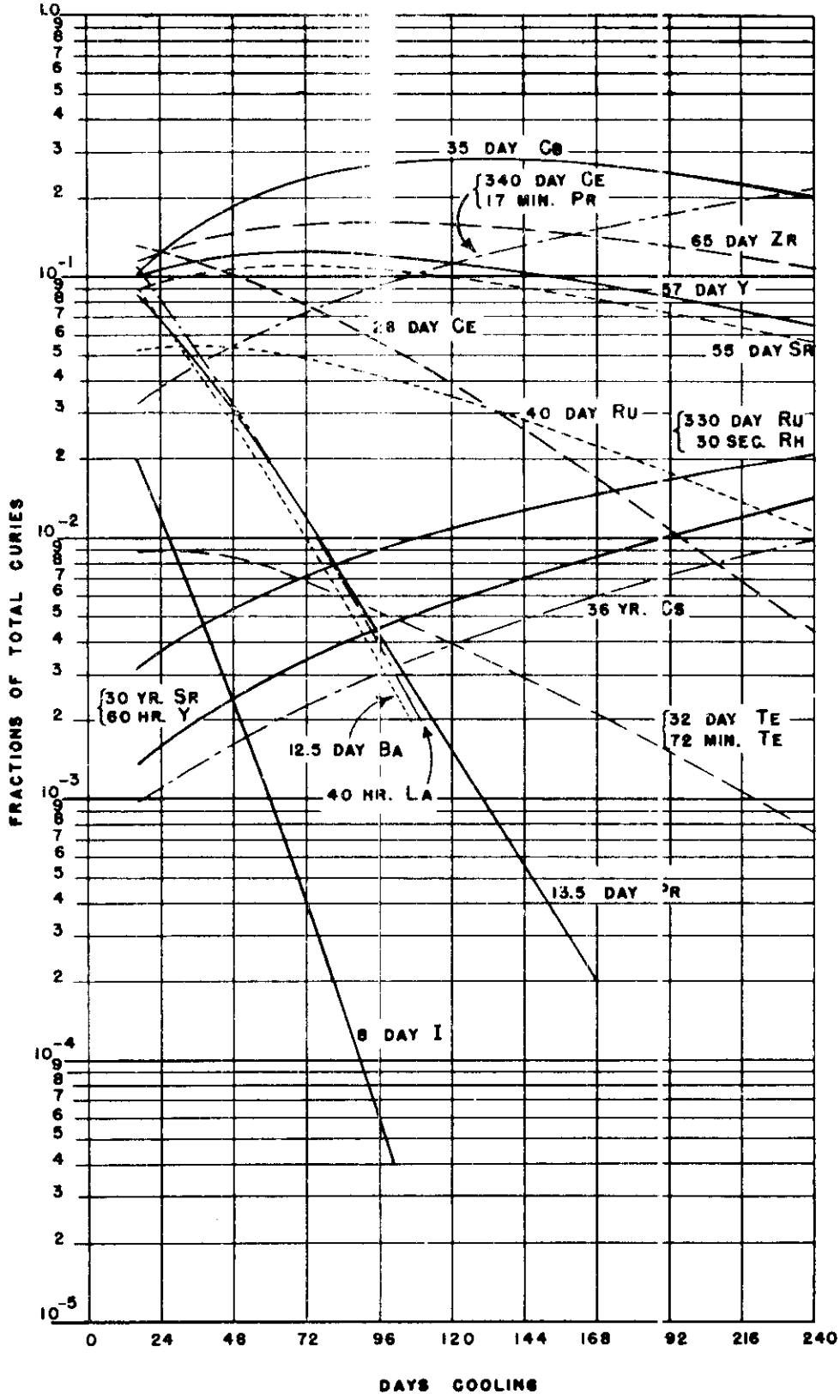

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FIGURE 8

FRACTIONS OF TOTAL CURIES DUE TO INDIVIDUAL RADIOACTIVE ISOTOPES FOLLOWING INFINITE TIME OF PILE OPERATION

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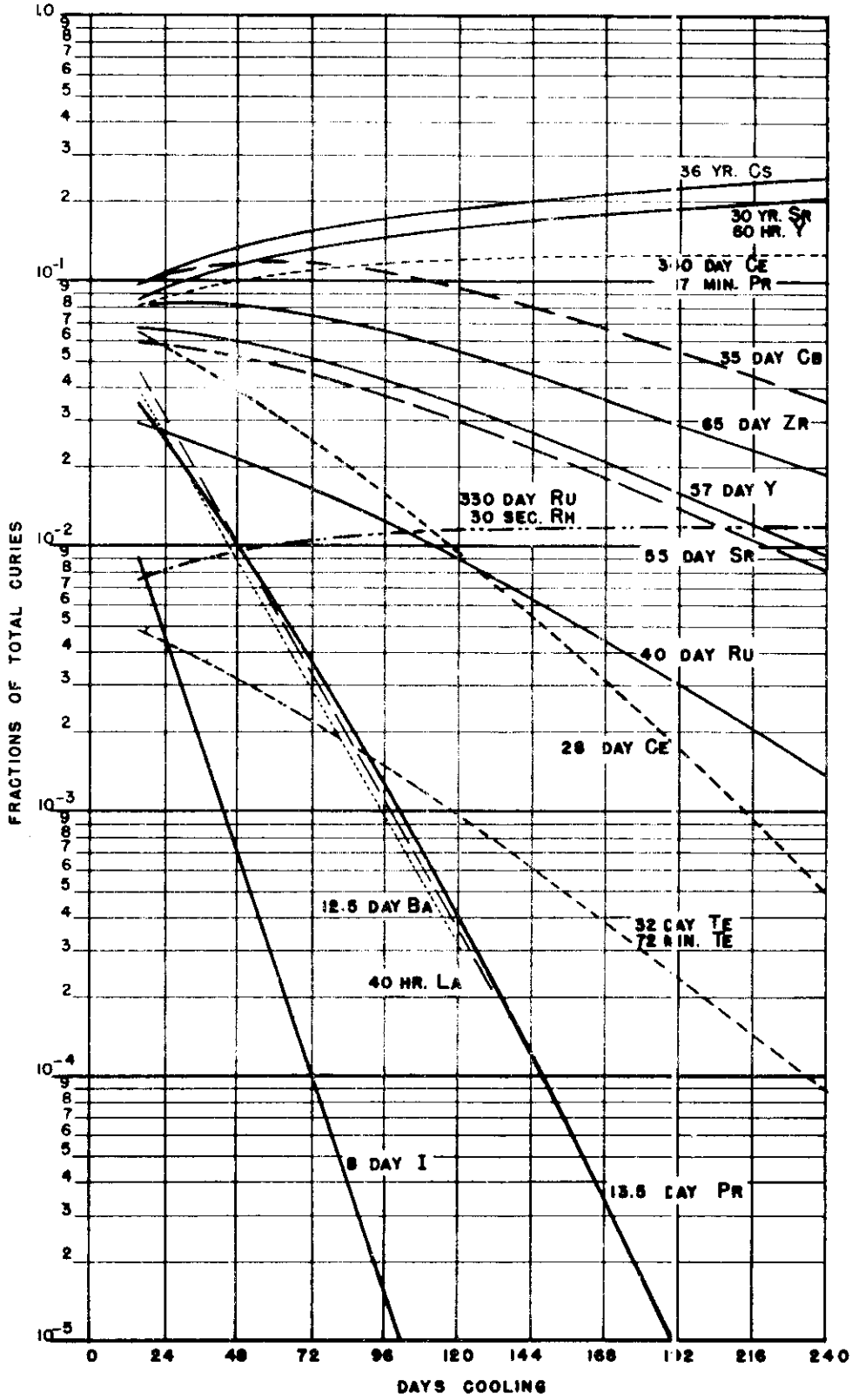

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FIGURE 9

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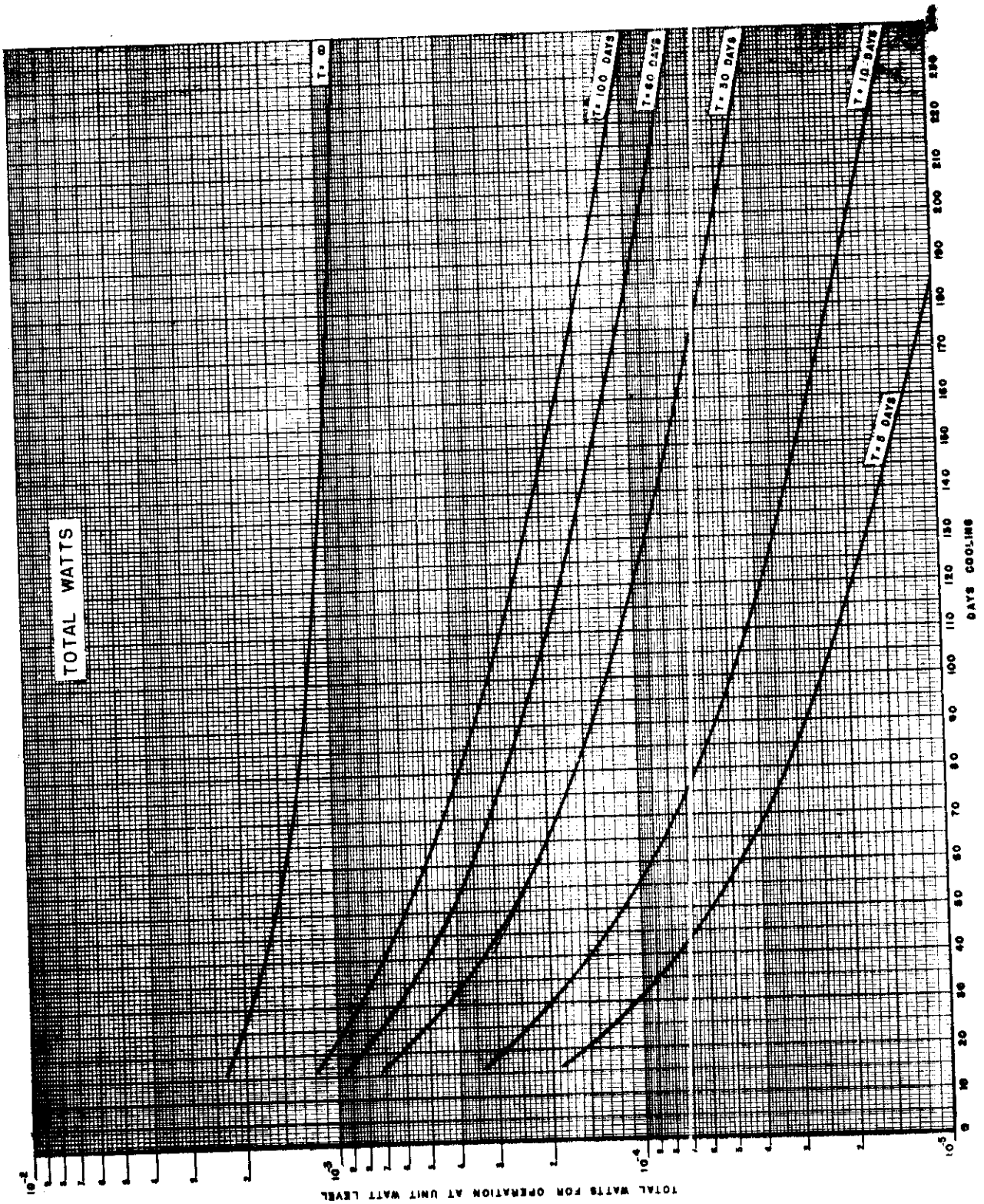

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FIGURE 10

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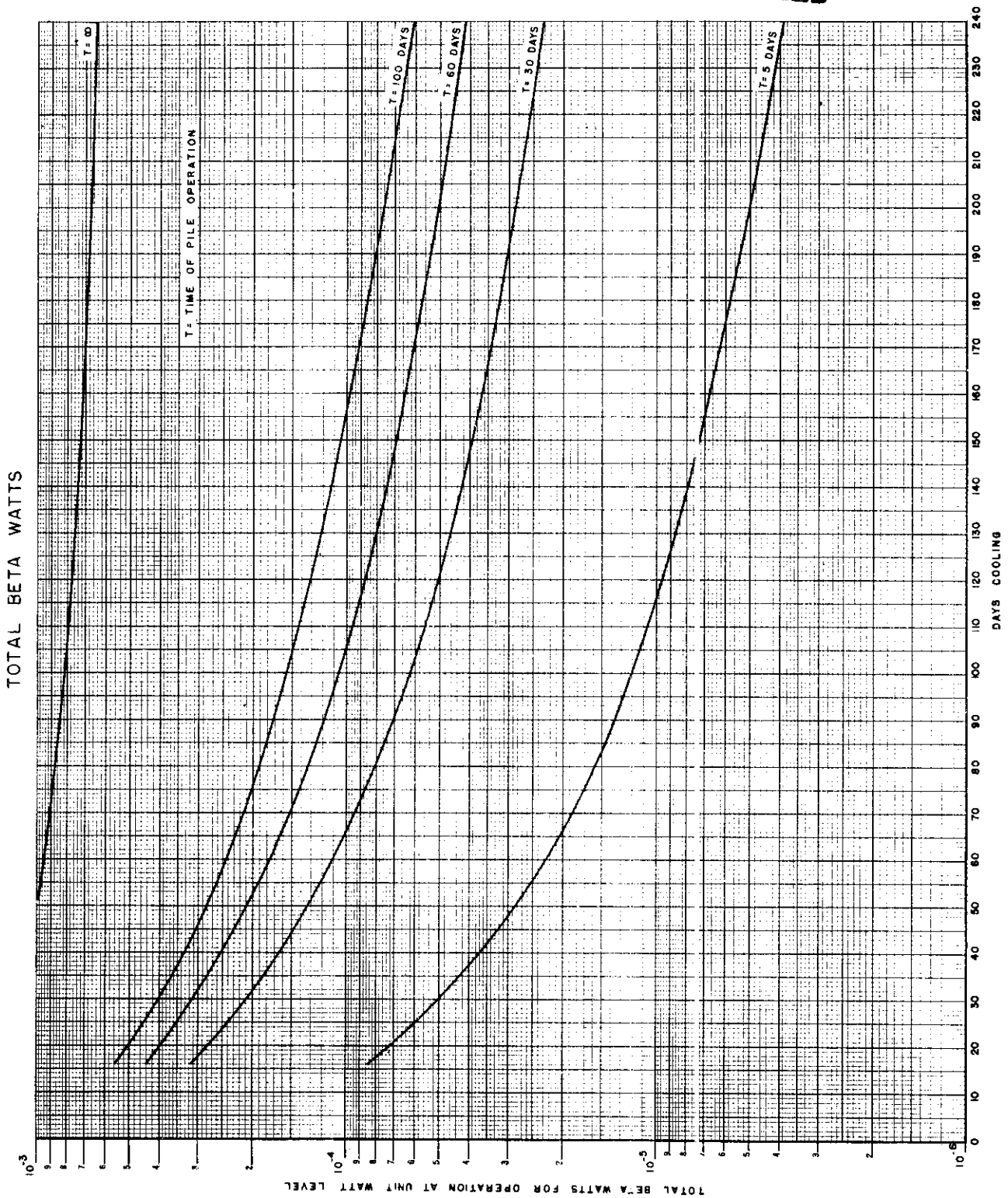

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FIGURE 11

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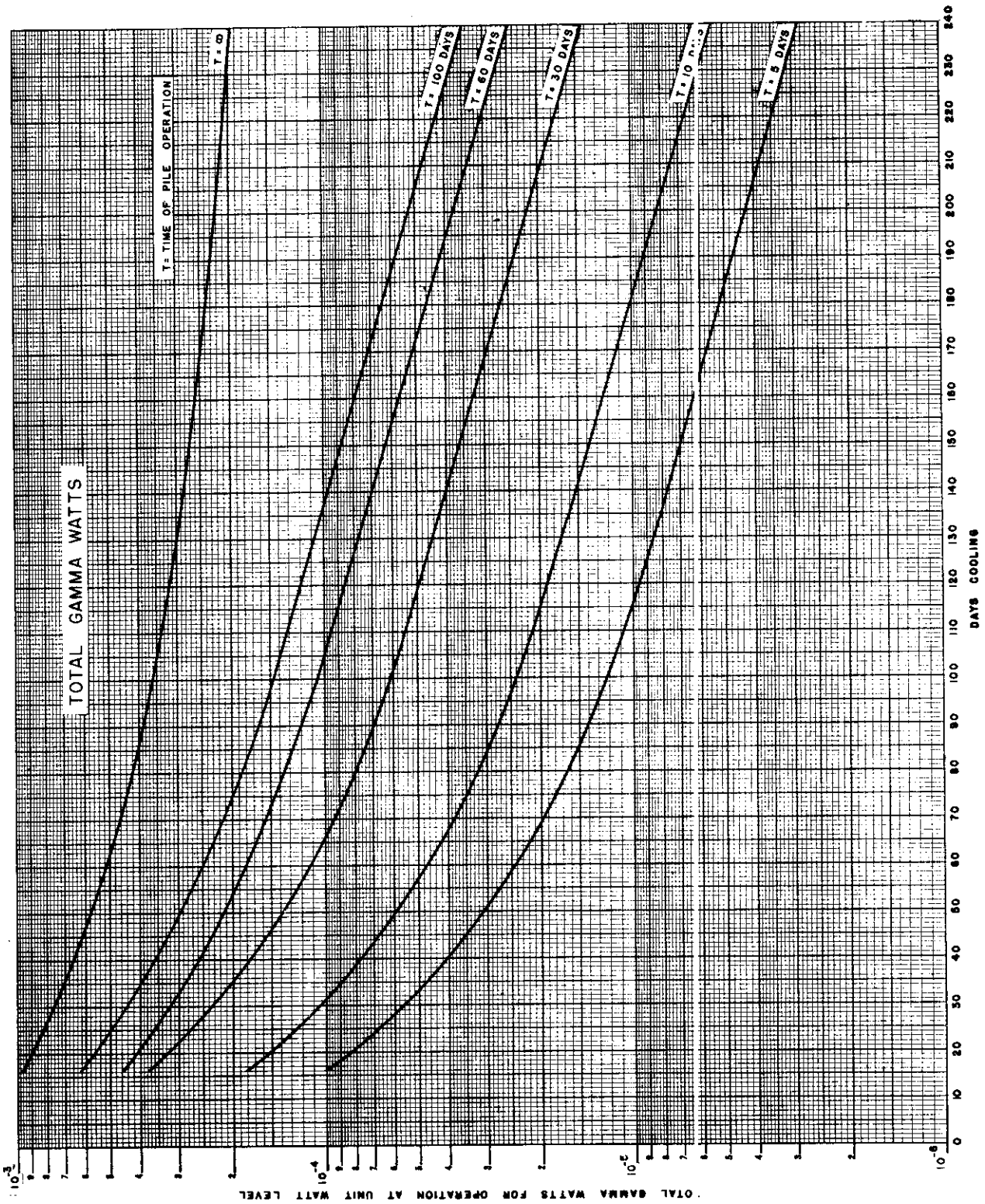

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FIGURE 12

FRACTIONS OF TOTAL BETA WATTS DUE TO INDIVIDUAL RADIOACTIVE ISOTOPES FOLLOWING 100 DAYS PILE OPERATION

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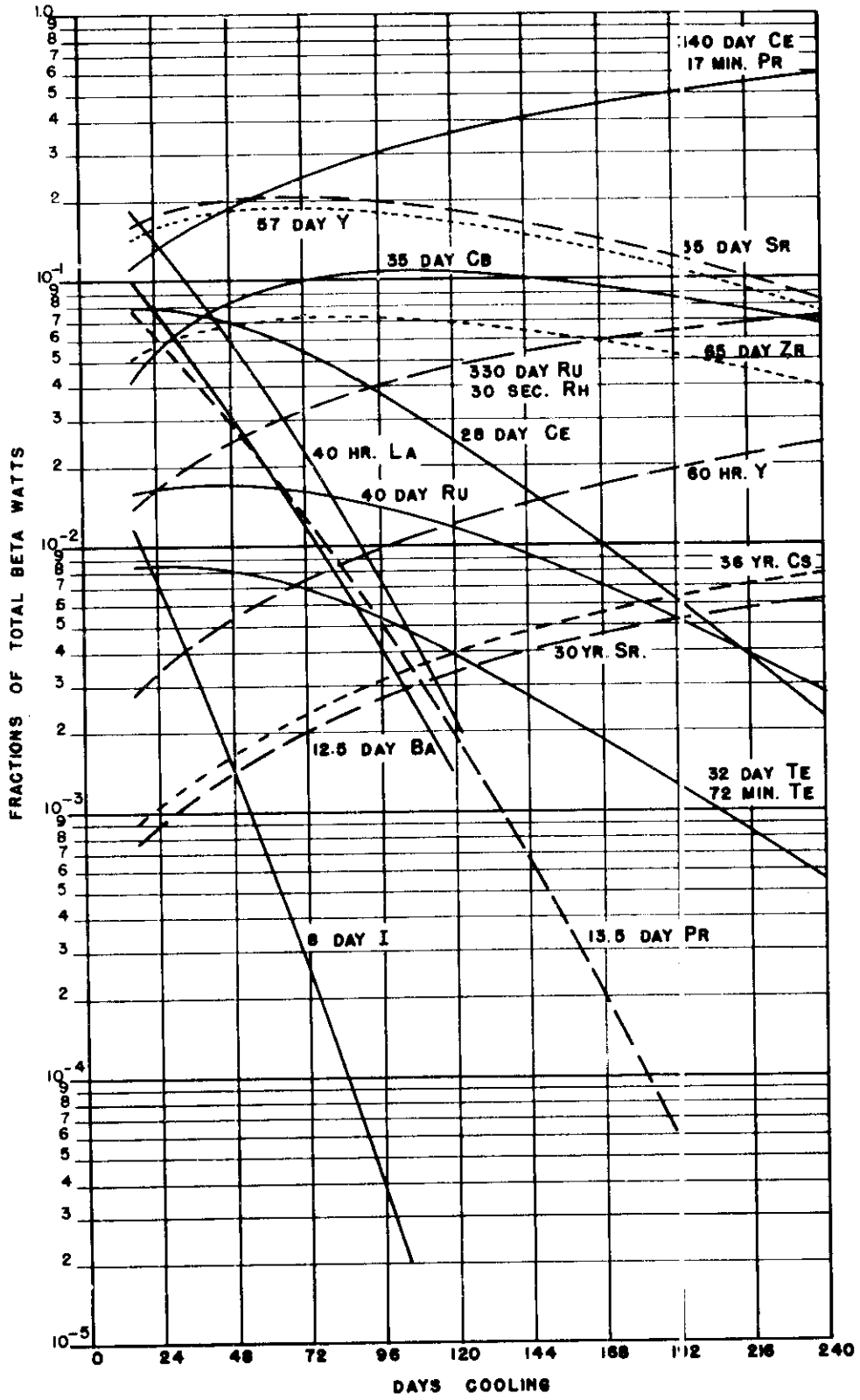

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FIGURE 13

FRACTIONS OF TOTAL GAMMA WATTS DUE TO INDIVIDUAL RADIOACTIVE ISOTOPES FOLLOWING 100 DAYS PILE OPERATION

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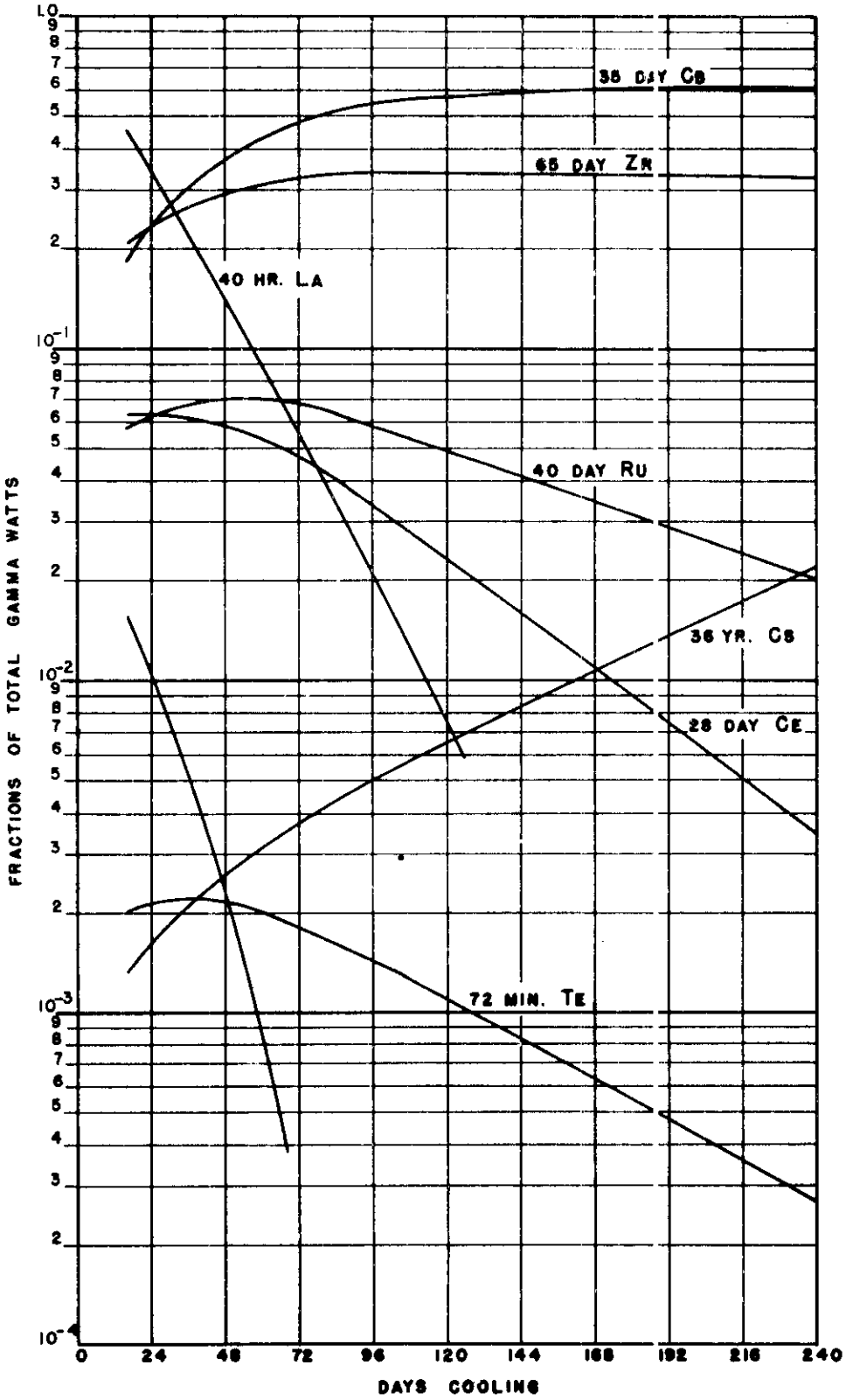

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TABLE III

Weights of Active and Inactive Fission Product Isotopes in Uranium Metal Exposed at 2500 KW/ton for 100 Days and with 50 Days Cooling

Active Isotopes		Inactive Isotopes	
Isotope	g./ton U	Element	g./ton U
55d Sr^{89}	1.32	Sr	10.7
57d Y^{91}	1.3	Y	7.5
65d Zr^{95}	1.35	Zr	26.5
35d Cb^{95}	1.01	Cb	12.2
42d Ru^{103}	0.84	Ru	14
1y $Ru^{(106)}$	0.45		
8d $I^{(131)}$	0.002	I	1.75
12.5d $Ba^{(140)}$	0.055	Ba	11
40h $La^{(140)}$	0.0086	La	11
28d Ce^{141}	0.605	Ce	29.2
275d $Ce^{(144)}$	6.4		
17.5m Pr^{143}	0.00022	Pr	13
Total	13	Total	137

Fission Products with No Active Isotope*

Element	g./ton U	Element	g./ton U
Se	0.25	Sb	0.05
Br	0.15	Te	3.25
Kr	0.75	Xe	29.8
Rb	4.5	Cs	29
Mo	21.7	Nd	28.2
43	7.25	Sm	8.25
Rh	2.5	Eu	1.25
Pd	1.5	Gd	0.05
Ag	0.225		

*The very long-lived radio-isotopes are considered to be essentially stable.

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SHIELDING AGAINST RADIOACTIVE RADIATION

Radioactive radiations of the types encountered in the 200 Areas have been known for half a century, and their action upon matter and upon living tissue is reasonably well understood. The intensity of the radiations from process materials is, however, enormously greater than that of any source which has previously been employed on an industrial scale. The largest radium sources that are handled routinely weigh about 1 gram, and can be handled safely encased in about 5 inches of lead. This thickness of lead reduces the gamma intensity about a thousand-fold. Half-ton buckets of processed metal are brought into the 200 Area in casks shielded by 12 inches of lead, which reduces the hard gamma intensity by a factor of about 10 million.

The use of massive shields is not in itself an absolute guarantee of safety. Apertures or cracks in the shield may permit the escape of dangerous amounts of radiation. Therefore, good practice demands that holes through shields be staggered so that there is no direct path along which radiation may escape. The scattering of radiation from surrounding objects may also be significant. A shield which protects personnel from the direct rays of the source may be inadequate if the radiation can reach them indirectly by reflection from the walls or ceiling. Hazards may also be created by the transport of active materials through a shield, for instance by accidental suckbacks of process materials into pipe galleries or by the discharge of radioactive materials into stack gases. Measures must also be taken to prevent the introduction of active materials into the body by inhalation, swallowing, or puncture wounds.

The following discussion covers the general principles of protection against radioactive radiations.

Nature and Penetrating Power of Radiations

The radiations emitted by 200 Area process materials are alpha particles, beta rays, and gamma rays. These radiations can be detected only by instruments, such as ionization chambers, Geiger counters, photographic films, and, at relatively high intensities, by fluorescent screens. Since the physiological effects of the radiations are delayed, it is possible for a man to receive a fatal dose without feeling it in any way at the time when he gets it. It is therefore imperative that adequate shielding be maintained and that it be monitored constantly at critical points.

Alpha Particles

Alpha particles are helium nuclei of mass four and positive charge two. They are ejected spontaneously by the nuclei of uranium and the product, but not by the fission elements. Because of the large mass and charge of the alpha particle, its penetrating power is very low. For example, the alpha particles from uranium are stopped completely by about 1 inch of air, 0.001 inch of aluminum, or 0.0001 inch of lead. Alpha particles present an important hazard only when their parent substance is taken into the body.

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Beta Rays

Beta rays are fast electrons of very small mass, roughly 1/2000 that of a proton or 1/8000 that of an alpha particle. They carry a negative charge of one unit and are emitted, with energies ranging up to 3 or 4 Mev, by radioactive nuclei. Their penetrating power is rather low (2 Mev betas are completely stopped by 23 feet of air or about 1/8 inch of aluminum). Shields of ordinary structural thicknesses are usually sufficient for beta rays; and the beta radiation hazard assumes importance only when the observer is directly exposed to the source.

Gamma Rays

Gamma rays, also called photons or quanta, are electromagnetic waves or particles whose wave length is roughly one millionth the wave length of visible light. The "hardest", most penetrating radiation emitted by the fission products is the 2.1-Mev gamma of lanthanum. Unlike alpha and beta particles, gamma radiation does not have a definite range in matter but is absorbed exponentially. The intensity of a beam of 2-Mev gamma radiation is reduced ten-fold by passage through 1300 feet of air, 20 inches of water, 8.5 inches of concrete, 3 inches of aluminum, or 1.7 inches of lead. Reduction of the intensity of this radiation by a factor of 100 would be obtained, for example, by using a shield of 3.4 inches of lead. The massive lead and concrete shielding used in the Canyon and Concentration Buildings of the 200 Area is intended primarily to reduce this penetrating radiation to a harmless intensity.

Safe Radiation Limits

The unit of X-ray or gamma radiation dosage is the Roentgen, usually designated as r, which represents that dosage which will produce 1 electrostatic unit of ions in 1 cubic centimeter of atmospheric air. This corresponds to an energy dissipation of 0.1 erg/cc. of air or 83 ergs/cc of body tissue. The Roentgen does not apply strictly to radiations other than gamma or X-rays; hence, beta radiation dosage is expressed in a unit which produces the same energy dissipation as the Roentgen and is called the "Roentgen equivalent, physical", abbreviated rep. This unit is also used to express the dosage produced by mixed beta and gamma radiation.

Dosage rate is most frequently expressed in milliroentgens/hour (mr/hr) or milliroentgen equivalents/hour (mrep/hr), although the term Roentgens per 8-hour period (r/8-hr) is also used.

Experience with radium and X-rays has shown that the human body can be given a dosage not exceeding 0.1 r in any 24-hour period, and repeated indefinitely, without any perceptible effect. Since personnel are on the job 8 hours each day, a maximum permissible dosage rate of 0.1 r/8-hrs. or 12.5 mr/hr has been established for total body irradiation. An ion chamber having an effective volume of 1 liter passes a current of approximately 10^{-12} amperes when exposed at this intensity. The shielding of the 200 Area is designed so that the dosage rate in any work area does not exceed 12.5 mr/hr, and in the majority

of cases is less than this by a factor of ten or more. The tolerance dosage of 0.1 r means that a 150-pound man can safely absorb only about 16 microwatt-hours of gamma energy per day. In contrast, a man stretched full length in the noonday sun receives solar energy at a rate on the order of 100 watts.

Single overdoses of gamma radiation react on the individual somewhat as follows:

A dose of 10 r produces a temporary decrease in blood count. The decrease appears several days after exposure. A normal count may be regained within a few weeks.

A dose of 100 r produces nausea, a mild "sunburn", and a serious reduction in the blood count. Recovery may be very slow, and the susceptibility of the individual to subsequent overexposure is increased.

A dose of 1000 r is lethal, with death occurring something like a week after exposure.

These figures are very rough, and take no account of very large individual differences in ability to tolerate radiation.

A more localized type of exposure results when the body is exposed to beta rays or to narrow beams of gamma radiation. Beta rays penetrate only the first centimeter or so of tissue, so that deeper layers are untouched. Narrow beams irradiate only part of the body at any one time, and so are less damaging than total body irradiation at the same intensity. These favorable effects are not considered great enough to permit an upward revision of the tolerance figure, which remains at 12.5 mrep/hr for beta rays and at 12.5 mr/hr for the most intense part of a gamma beam.

When radioactive materials are taken into the body, the situation is considerably more complicated. An important factor is the tendency of certain elements to concentrate in specific body tissues. For instance, iodine concentrates in the thyroid whether it is inhaled or ingested. Mixed fission products remain to a great extent in the lungs if they are inhaled, but deposit in the bones if swallowed. Plutonium is most dangerous when inhaled or introduced through a wound, and is somewhat less dangerous when swallowed. It also deposits in the bones.

The amounts of active materials which the body can tolerate are of the following orders of magnitude: 10^{-2} to 10^{-3} curies of mixed fission products, or 10^{-3} curies of iodine, may be swallowed as a single dose and never repeated. Or, approximately 10^{-6} curies of either mixed fission products or iodine may be taken daily for an indefinite period. Air containing 10^{-10} curies/cc. of mixed fission products or iodine may be breathed for a single day, but for continued exposure the tolerance limit is dropped to 10^{-13} curies/cc. Xenon, which does not deposit in specific locations, is tolerable for continued exposure at a concentration of 10^{-11} curies/cc. The maximum amount of product which can be tolerated by the skeleton, e.e., the so-called lifetime tolerance dose, is about 10^{-7} grams, and the tolerable concentration in the air is set

at 5×10^{-16} gm/cc. This is the concentration which is believed safe to breathe 8 hours/day for 2 years.

The significance of these tolerance figures may be illustrated in terms of the amounts of process materials which are handled routinely. Consider a 1-ton batch of metal containing 100 grams of product. The total fission product activity associated with this batch, as it enters the first processing building, is roughly 10^5 curies, of which about 10^3 curies is iodine. This ton of metal is made up into about 3000 gallons or 10^7 cc. of 20% uranyl nitrate hexahydrate (UNH) solution. The solution therefore contains about 10^{-2} curies/cc. of mixed fission activity and 10^{-5} gm/cc. of product. The mixed fission products in 0.1 to 1.0 cc. of this solution could be tolerated as a single dose, and no more than the fission products in 10^{-4} cc. could be tolerated as an indefinitely repeated daily dose. The amount of product in 10^{-2} cc. of the 20% UNH solution provides a lifetime dose. If all the iodine in the batch were discharged into the atmosphere, it would have to be diluted in approximately one cubic mile of air before it would be safe for continued breathing; similarly, the amount of product in the batch would require dilution in a cube of air about four miles on a side.

Contamination of working areas, clothing, or the skin may result in transfer of active materials into the body, and is therefore an important hazard. In addition, contamination of a small area of skin may result in a severe local overexposure. The procedures by which hazards of this type are minimized in the 200 Areas, are described in Chapter XIII.

Principles of Shielding

The amount of radiation which an observer receives from an external source depends on the strength and geometry of the source, on its distance from the observer, on the type and energy of the radiation, and on the geometrical arrangement and composition of the shielding. A detailed discussion of these factors is beyond the scope of this chapter, and may be found in the references. A number of rough generalizations are given in the following sections.

Strength of Sources

The intensity of radiation from a point source of gammas, like the intensity from a point source of light, varies inversely with the square of the distance between the source and the point of measurement. A point source is one whose dimensions are small in comparison with the distance between it and the point of measurement. If such a source produces unit intensity at a distance of 1 foot, then the intensity at 2 feet is 1/4; at 3 feet 1/9, and so on.

The approximate strength of a number of typical sources may be illustrated as follows: A single slug which has been processed at the center of a pile for 100 days and brought into the 200 Area after a 40-day cooling period produces a gamma dosage rate of approximately 5×10^5 mr/hr at a

distance of 1 foot. Approximately equal numbers of beta particles and gamma photons are generated in the slug, and if all particles could escape from the slug, e.e., if there were no self-shielding, the beta dosage rate would be roughly one hundred times greater than the gamma dosage rate because the betas are very much more readily absorbed in air or tissue. Because of their limited range, however, the beta particles have a relatively small chance of escaping from the slug. Consequently, the beta and gamma dosage rates at short distances from a slug are approximately equal. The average activity of the slugs in the central flattened zone of the pile is about 60% of that cited above for the most radioactive slug.

A single half-ton bucket of metal from the central zone of the pile produces a gamma radiation intensity of the order of 107 mr/hr at a distance of 1 foot. The stainless steel walls of the bucket stop all of the beta radiation from the slugs but have only an insignificant effect on the gamma radiation.

One cubic centimeter of 20% UNH solution prepared from this bucket of slugs, if spread out so that self-absorption is negligible, produces a gamma radiation intensity of about 50 mr/hr at 1 foot, and a beta radiation intensity of 2500-5000 mrep/hr at the same distance. Where the beta ray contribution predominates, as in the present case, the inverse square law is not followed for the total radiation, because more and more beta particles are absorbed in air as the distance is increased. It has been found that at distances from 1 to 10 feet from a source of mixed fission products, the radiation intensity falls off very nearly as the -2.5 power. At distances greater than about 25 feet the beta particles are completely stopped by air.

At the surface of a large tank containing this 20% UNH solution, the gamma radiation intensity is of the order of 6500 mr/hr. The beta radiation intensity at the liquid surface is roughly the same because of the self-shielding effect mentioned previously, but these beta particles are completely stopped by the steel walls.

Estimation of the Radioactive Radiation from Processed Metal

The amount of radiation emitted by processed metal may be estimated if the history of the metal is known. In certain cases it is necessary to take into account the distribution of energy among the various fission products, since these emit radiations of different penetrating power. The detailed required for such a calculation is available in the Project Handbook (CL-697), Chapters III, V, and XI, and in Reports CC-829 and TNX-7. Frequently, however, a sufficiently close estimate may be based on the Borst-Wheeler Curves (Figure 14) which relate radioactive power generation to time of irradiation and time of decay.

The general procedure for estimating the dosage rate produced by processed metal may be illustrated by an example. Suppose that a mass of metal has been irradiated for 100 days in the central columns of the pile, where the average power generation is 7550 watts/slug or 2.1 watts/gram of metal. After 40 days' decay, the Borst-Wheeler curves indicate that the

FIGURE 14

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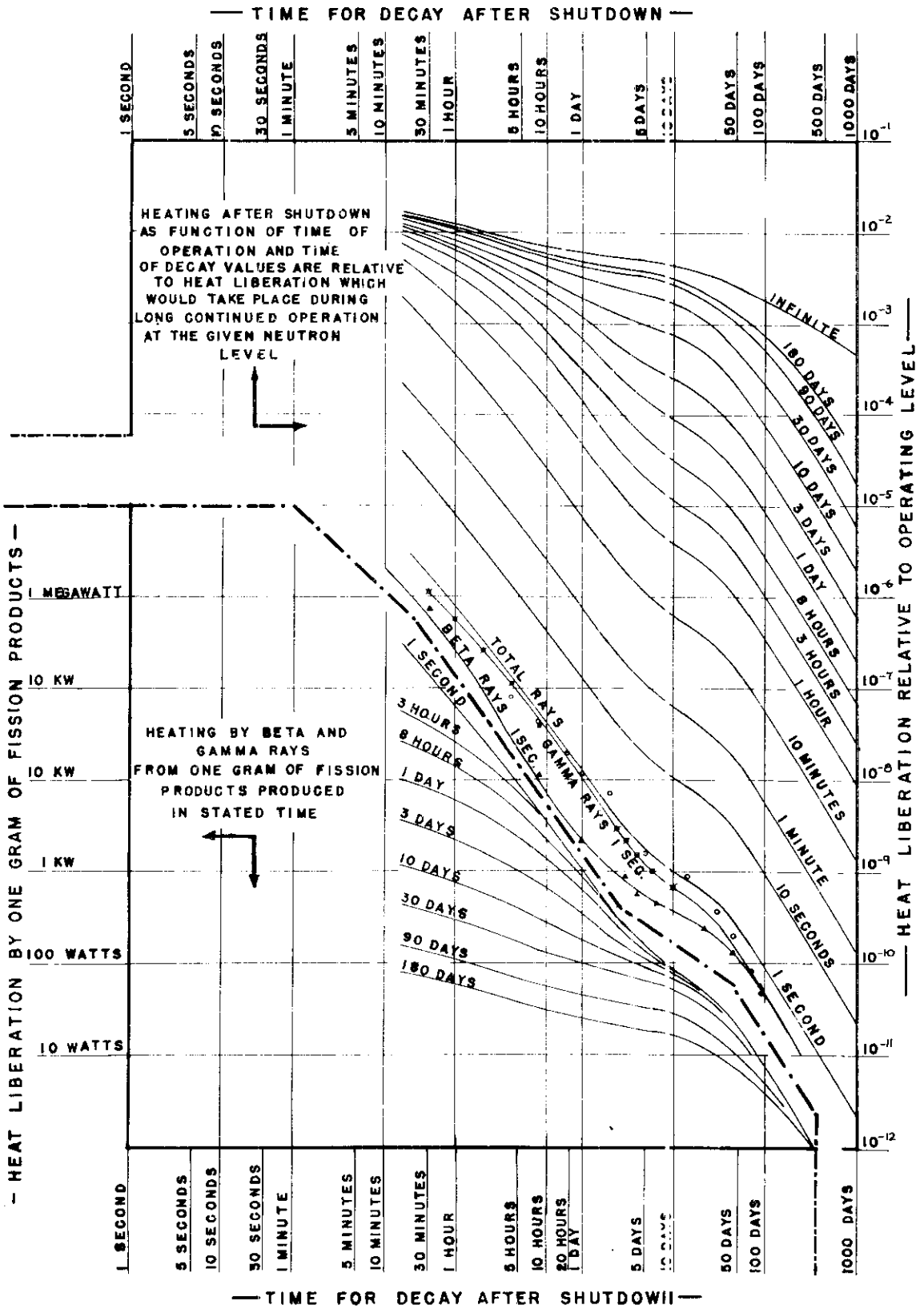
RATE OF ACTIVITY DECAY



power generation is reduced by a factor of 1000 and the total radioactive heating effect is therefore 0.0021 watts/gram of metal. One cubic centimeter of 20% UNH solution prepared from this metal contains 0.114 grams of metal and generates 0.00024 watts, of which one-half, or 0.00012 watts, is due to gamma radiation. A 1-watt gamma source produces a radiation intensity of very nearly 10^6 mr/hr at a distance of 1 foot; hence 1 cc. of the solution is expected on this basis to produce 120 mr/hr at a distance of 1 foot. This value is about twice as high as the more exact figure of 50 mr/hr indicated above; however, it is generally accepted that the Borst-Wheeler curves, at decay periods of interest to 200 Area operations, give values which are high by a factor of approximately two. Correction by this factor brings the rough estimate into fairly good agreement with the more precise value.

The beta power generation is approximately equal to the gamma power generation, but beta radiation is absorbed by air or tissue about 100 times as strongly as gamma radiation a short distance from a small volume such as 1 cc. of solution, the beta ray dosage rate is consequently about 100 times as great as the gamma dosage rate.

Geometry of Sources

The geometry of the source governs the rate at which the gamma dosage rate falls off with increasing distance from the source. It has already been indicated that radiation from a point source follows an inverse square law. The dosage rate produced by a line source, such as a pipe filled with a radioactive solution and observed from a point on a perpendicular to the axis of the pipe, depends on whether the pipe is long or short in comparison to the distance at which it is observed. When the pipe is long and the distance is short, the dosage rate varies inversely as the first power of the distance; when the distance is equal to or greater than the length of the pipe, the inverse square law is followed approximately. The intensity produced by a disc or plane source, such as a cylindrical tank of liquid viewed from a point on the axis of the cylinder, decreases at a rate slower than a first power law at distances short in comparison to the diameter of the tank. At large distances the inverse square law is followed.

For example, suppose that a long pipe of 2 inches inside diameter is filled with 20% UNH solution and an estimate is desired of the gamma dosage rate at a point 5 feet to one side of this pipe. The dosage rate is $I = \pi I_o'/a$, where I_o' is the dosage rate at unit distance from the activity in unit length of pipe, and a is the distance from pipe to observer. Taking the unit of length as a foot, one finds that each foot of pipe contains 600 cc of solution. Each cubic centimeter of solution produces a dosage rate of 50 mr/hr at 1 foot; hence I_o' is 30,000 mr/hr at 1 foot from a 1-foot length of pipe. The distance a is 5 feet; and the dosage rate at the required point is 19000 mr/hr.

As another example, suppose that a puddle of the same solution, 1 cm. thick and 2 feet in diameter, has been spilled on the floor. The gamma

[redacted]

dosage rate at a point a feet above the center of the puddle is $I = I_o'' \ln (R^2 + a^2)/a^2$, where I_o'' is the dosage rate at unit distance from the activity in unit area of the source. Therefore, 50 mr/hr at 1 foot from 1 cc x 930 cc/sq.ft. = 46500 mr/hr at 1 foot from 1 sq.ft. of puddle. If a is taken as 2 feet, the dosage rate I turns out to be 32600 mr/hr. On the other hand, if the puddle were considered as a point source and the inverse square law were applied, the result would be 36400 mr/hr. This demonstrates that at distances comparable to the dimensions of an extended source, the inverse square law provides a fair approximation.

Self-absorption in the source becomes important when the dimensions of the source are large in comparison to the mean free path of the radiation in the source. The mean free path is the thickness of shield required to reduce the intensity of a parallel beam of gamma rays by a factor of e = 2.718. For example, the most penetrating gamma radiation generated in a slug has an energy of 2 Mev and a mean free path in uranium (see Fig. 5) of 1 cm., while the slug has a radius of 1.5 cm. In such case only 27% of the gamma rays are able to escape. The remaining 73% are absorbed before they can get out of the slug. Softer gamma rays are even more strongly absorbed. A similar situation exists in the case of large tanks filled with radioactive solutions. Effectively, the radiation can escape from a depth of liquid equal to one man free path, which for 2 Mev gamma rays is of the order of 20 cm. An observer viewing a large tank at a distance receives radiation only from that layer of solution, one mean free path in thickness, which is represented by [illegible].

Absorption

Alpha and beta radiations are completely absorbed by comparatively thin shields, and the details of their absorption are of minor interest in separation plant design. Under exceptional circumstances the stopping of beta rays gives rise to significant quantities of penetrating radiation, the so-called "Bremsstrahlung" or slowing-down radiation which is identical with the continuous X-ray spectrum. The stopping of beta radiation from a 100-curie source produces an amount of Bremsstrahlung equivalent roughly to 1 curie of gamma activity. In 200 Area processing materials, 100 curies of beta activity are usually accompanied by 10 to 100 curies of gamma activity, and the contribution of the Bremsstrahlung is negligible.

Shielding requirements are determined in most cases by the gamma radiation, whose penetrating power depends on the energy of the photons and the atomic number of the absorber. The absorption takes place exponentially, in such a way that if a shield of absorption coefficient μ and thickness t is placed in a beam of intensity I_o, the intensity in the emerging beam is

$$I = I_o e^{-\mu t}$$

Frequently it is convenient to express the absorbing power of a shield in terms of the number of inches k required to reduce the intensity by a factor of ten. In this case,

$$I = I_o 10^{-t/k} \quad \text{or,} \quad \log_{10}(I_o/I) = t/k$$

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[redacted]

FIGURE 15

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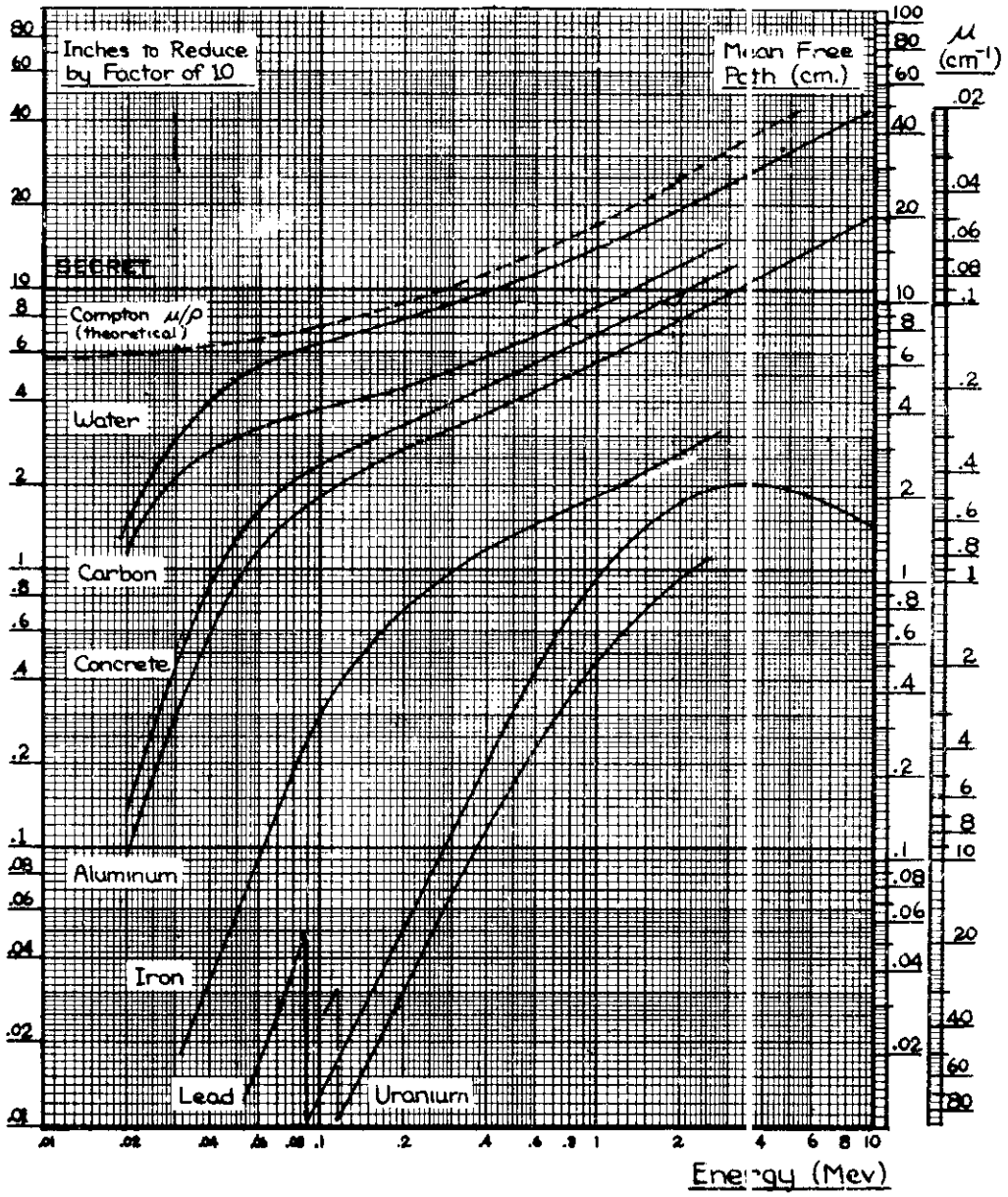
Absorption of Gamma Radiation



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The absorption coefficients of a number of common shielding materials are given as functions of gamma energy in Fig. 15. The curves have various shapes because three mechanisms are involved in the absorption process: photoelectric absorption, Compton scattering, and pair production.

Photoelectric absorption occurs when the active energy of the photon is used to eject an electron from one of the orbits of an atom. Absorption by this mechanism predominates at low gamma energies and increases with the atomic number of the absorber. In light elements such as aluminum, the photoelectric effect accounts for most of the absorption up to about 0.1 Mev. In heavy elements such as lead, it is effective up to about 1.0 Mev. The sharp break in the lead curve at 0.09 Mev is called the K absorption edge. Photons having energies less than this value are unable to pry loose the most tightly bound electrons of the K shell; therefore a photon of 0.08 Mev is less strongly absorbed by lead than a photon of 0.10 Mev, in spite of the fact that it is less energetic.

Compton scattering occurs when part of the photon's energy is used to eject an electron while the remainder appears as a scattered photon of lower energy. This process accounts for most of the absorption in the intermediate energy range (0.1 to 10 Mev in aluminum; 1.0 to 4.0 Mev in lead). Compton absorption varies with the number of electrons per cubic centimeter of absorber and therefore is proportional to the density of the absorber. In the energy range where this mechanism predominates, the effectiveness of a shield is roughly proportional to its sectional density, expressed as grams/square centimeter, and is nearly independent of the composition of the shield. Compton absorption decreases with increasing gamma energy.

Pair production occurs when a photon enters the field of a nucleus and is converted into a positron and an electron, plus a certain amount of translational energy of these particles. The electron is absorbed like a beta ray; the positron collides with another electron and the mass of the two particles is converted into two 0.5-Mev photons. This phenomenon occurs to a significant extent only at very high energies (above 10 Mev in aluminum; above 4 Mev in lead), and increases rapidly with the energy of the photons and the atomic number of the absorber.

As a result of these three processes, every substance shows a minimum in its curve of absorption coefficient vs. energy somewhere in the region where Compton scattering predominates. This minimum appears at 2.5 Mev in lead and at 20 Mev in aluminum.

The penetrating gamma rays of the fission products are absorbed mainly by the Compton process; therefore the material of which the shield is made is unimportant as long as the shield is sufficiently massive, and the choice of shielding materials depends primarily on considerations of cost and available space. The cheapest shielding materials, water and concrete, are used where space and total mass are unimportant, as in the storage areas and the separations buildings. Lead is used where compactness and minimum total mass are desired, as in the transfer casks. Steel is used where structural stability is important, as in the crane cab. Stainless steel is used where ease of decontamination is necessary, as in the sample carriers.

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The geometry of source and shield have some influence on the required thicknesses. When a point source is shielded by a slab perpendicular to the line joining source and observer, all rays effectively enter the shield at normal incidence and the intensity is reduced by the factor e^{-ut}. When a line source is shielded by a slab, many of the rays which reach the observer must travel an oblique path through the shield. The effectiveness of a given thickness of shield is thereby increased, and the intensity is reduced by a function of the form $(ut)^{-\frac{1}{2}}e^{-ut}$. When a large tank is shielded by a slab, this effect of obliquity is even more pronounced and the function takes the approximate form $(ut+2)^{-1}e^{-ut}$. In other words, the effectiveness of the shield is improved by a square root factor when a line source is used, and by a first power factor when a tank source is used.

It was mentioned earlier that a single bucket of metal produces a dosage rate of about 10^7 mr/hr at 1 foot. About 67% of this is contributed by 2 Mev gamma radiation, about 28% by 0.8 Mev gamma radiation, and the rest by softer components. The 12 inches of lead shielding provided by the transfer cask give a reduction of 10^7 for 2 Mev gammas (one factor of ten for each 1.7 inches of thickness) so that, provided there are no blowholes in the lead casting, the dosage rate outside the cask is less than 1 mr/hr.

Similarly, Hanford concrete with a density of 2.5 reduces the intensity of 2 Mev gamma radiation by a factor of ten for every 8.5 inches of thickness. (Figure 15, which shows that 10 inches are required, is based on a concrete density of 2.2). Therefore a 5-foot concrete wall provides a reduction of about 10^7 in addition to the factor of 25 introduced by the greater distance.

Scattering

It has been noted that the Compton scattering process produces photons of reduced energy. These scattered photons are absorbed with comparative ease and therefore make no very large contribution to the radiation which penetrates the shield. Under certain conditions, however, those photons which are diffusely reflected from a shield or from other matter may be of concern.

The energy of the scattered photons depends entirely on the energy of the primary photons and on the angle at which they are scattered. For example, 2 Mev primary photons which are deviated only a few degrees from their initial path retain nearly all of their energy. At the other extreme, if they are scattered 180°, i.e., reflected back along the same path, their energy is reduced to 0.2 Mev. Primary photons of low energy are not so greatly affected. The energy of a 0.05 Mev photon is reduced only to 0.04 Mev by scattering through 180°.

The intensity of the scattered radiation depends in part on the energy of the primary photons. For example, if a beam of 2 Mev photons strikes a concrete slab at normal incidence, about 0.1% of the total primary energy is scattered back into unit solid angle in the direction of the beam. If the primary beam consists of 0.5 Mev photons, about 0.7% of the primary energy is similarly scattered. The scattered intensity also depends on the atomic number of the scatterer. The less energetic scattered photons are more susceptible to further scattering and photo-electric absorption in the scatterer, and hence have a re-

[redacted]

duced chance of escaping through the surface of the scatterer. For this reason the radiation scattered by a lead slab is less than that scattered by a concrete slab. At distance close to the lead slab, however, the effective dosage rate is increased by the photoelectrons which are knocked out of the lead.

Estimates of the order of magnitude of the intensity of the radiation scattered by a thick slab may be made by use of the formula

$$I = (0.01\ I_o\ A)/(d_1 d_2)^2$$

where I is the dosage rate at the location of the receptor, I_o is the dosage rate 1 foot from the point source, A is the area in square feet of the slab which scatters the radiation, d_1 is the distance from source to scatterer in feet, and d_2 is the distance from scatterer to receptor. For example, suppose that a bucket of metal ($I = 10^7$ mr/hr at 1 ft.) located on one side of the head-end barricade of T Concentration Building irradiates a patch of ceiling 50 feet square ($A = 2500$ ft.^{2}), $d_1 = d_2 = 50$ ft. The dosage rate of the scattered radiation at the receptor then turns out to be about 40 mr/hr. Similar considerations apply in the design of labyrinths, where several reflections may be necessary to bring the dosage rate down to the desired level. An example of the behavior of radiation in a labyrinth is given in Figure 16.

Scattering from the air surrounding a source may become important when the source is sufficiently strong. For example, if a single bucket of metal were placed outdoors and shielded so that radiation could escape upward in a cone of unit solid angle (apical angle 68°), the tolerable intensity of 12.5 mr/hr would not be reached until the observer retreated about 40 feet from the bucket.

Apertures

Apertures in shields may permit the escape of dangerous beams of radiation unless they are properly designed. Such apertures include the clearances around cell covers, pipes between cells and operating areas, and the like.

Frequently the aperture is small in comparison to the dimensions of the source, so that only a small fraction of the source is able to send radiation directly through the aperture. For example, if a straight length of pipe runs through a heavy concrete wall and abuts on a large tank of active solution, a man located on the safe side of the wall receives radiation only from that part of the tank which he can see through the pipe. If the pipe were to be plugged with concrete, the necessary length of the concrete plug would be somewhat less than the thickness of the surrounding wall. It is therefore apparent that if the pipe is brought through the wall along a path which is curved or staggered so that scattering is unimportant, the radiation leakage through the pipe may be made comparable to the leakage through the solid wall. In general, the ends of the pipe should be out of line by at least four to six diameters to reduce scattering through the pipe, and the pipe should be bent so that the least possible length of pipe lies along any line drawn between source and observer.

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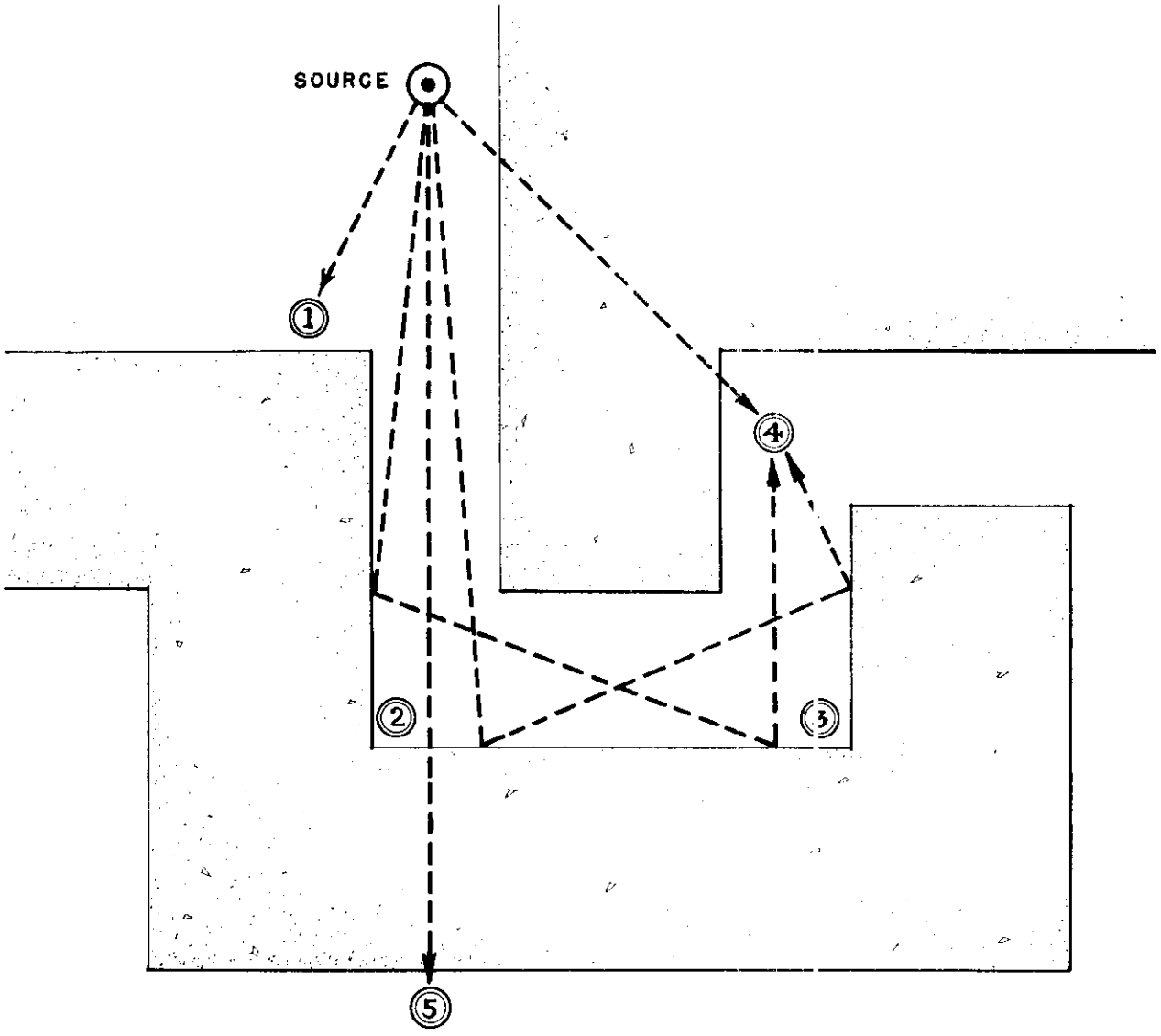
FIGURE 16

PRINCIPLE OF A [illegible]RINTH



LEGEND

SOURCE : 1/2 TON OF FRESHLY DISCHARGED METAL

① 150,000 r/8HR. OF 2 Mev GAMMAS 5FT. FROM SOURCE

② 17,000 r/8HR. OF 2 Mev PRIMARY GAMMAS AT FIRST BEND OF LABYRINTH

③ 10 r/8HR. OF 0.4 Mev SCATTERED GAMMAS AT SECOND BEND

④ 0.3 r/8HR. OF 0.2 Mev SCATTERED GAMMAS PLUS 0.0004 r/8HR. OF 2 Mev GAMMAS TRANSMITTED THRU CONCRETE

⑤ 0.04 r/8HR. OF 2 Mev GAMMAS TRANSMITTED THRU CONCRETE

[redacted]

Similar considerations apply in the design of stepped shield plugs, such as the concrete cell covers of the Canyon Buildings. Here the clearances are kept as close as is structurally feasible, and the three steps of the plugs prevent any crack from running more than a third of the way through the shield.

PROPERTIES OF URANIUM OF PROCESS IMPORTANCE

Properties of Uranium Metal

Uranium is a silver-colored metal, resembling nickel in appearance and capable of taking a high polish. The metal turns a golden color after a few hours' exposure to the atmosphere, however, and after several days tarnishes to a dark brown color. The metal melts at about 1100 °C (2010 °F) and the boiling point is estimated to be about 4300 °C (7770 °F). It is one of the heaviest metals, having a density of 19 gms/cc. (1185 lbs./cu.ft.). Its density is nearly twice that of lead (about 11 gm/cc.), and about the same as that of gold and tungsten. One metric ton (2200 lbs.) of uranium metal in one piece would occupy a volume equivalent to a cube only 1 foot 2-3/4 inches on a side. (Additional physical properties are given in Section A of this manual).

Uranium metal is fairly reactive chemically. As mentioned above, it tarnishes easily on exposure to air and burns briskly when heated to about 170 °C; a vacuum or inert atmosphere is needed for melting the metal. The finely-divided metal (and the dioxide and hydride) burns spontaneously in air, and therefore "sparks" when machined, sawed, filed, etc. Finely-divided uranium also slowly decomposes cold water, and the reaction becomes quite vigorous at the boiling point.

Metallic uranium (and the dioxide) is soluble in HNO_3 to form solutions of $UO_2(NO_3)_2$. A fast rate of dissolution is favored by high concentrations of HNO_3 and high temperatures.

Properties of Uranium in Solution

The salts of uranium consist chiefly of two classes: (1) the uranous, U(IV), green in color and a strong reducing agent; and (2) the uranyl, UO_2^{+2}, yellow in color with a strong greenish fluorescence. The compounds of hexavalent uranium (UO_2^{+2}) are more important than those of tetravalent uranium. $UO_2(NO_3)_2$, the product of uranium metal dissolution in HNO_3, is very soluble in H_2O_2 or dilute HNO_3. In Figure 17 the density of aqueous solution of $UO_2(NO_3)_2 \cdot 6H_2O$ (commonly abbreviated "UNH") and the grams of UNH per liter are plotted against the weight per cent of UNH in the solution. Additional data on densities of UNH solutions containing HNO_3 and/or H_2SO_4 and on the crystallization temperatures of UNH - HNO_3 solutions are given in Table IV, V and VI.

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FIGURE 17

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CONCENTRATION AND DENSITY OF AQUEOUS UNH SOLUTIONS
AS A FUNCTION OF THE PERCENT UNH BY WEIGHT

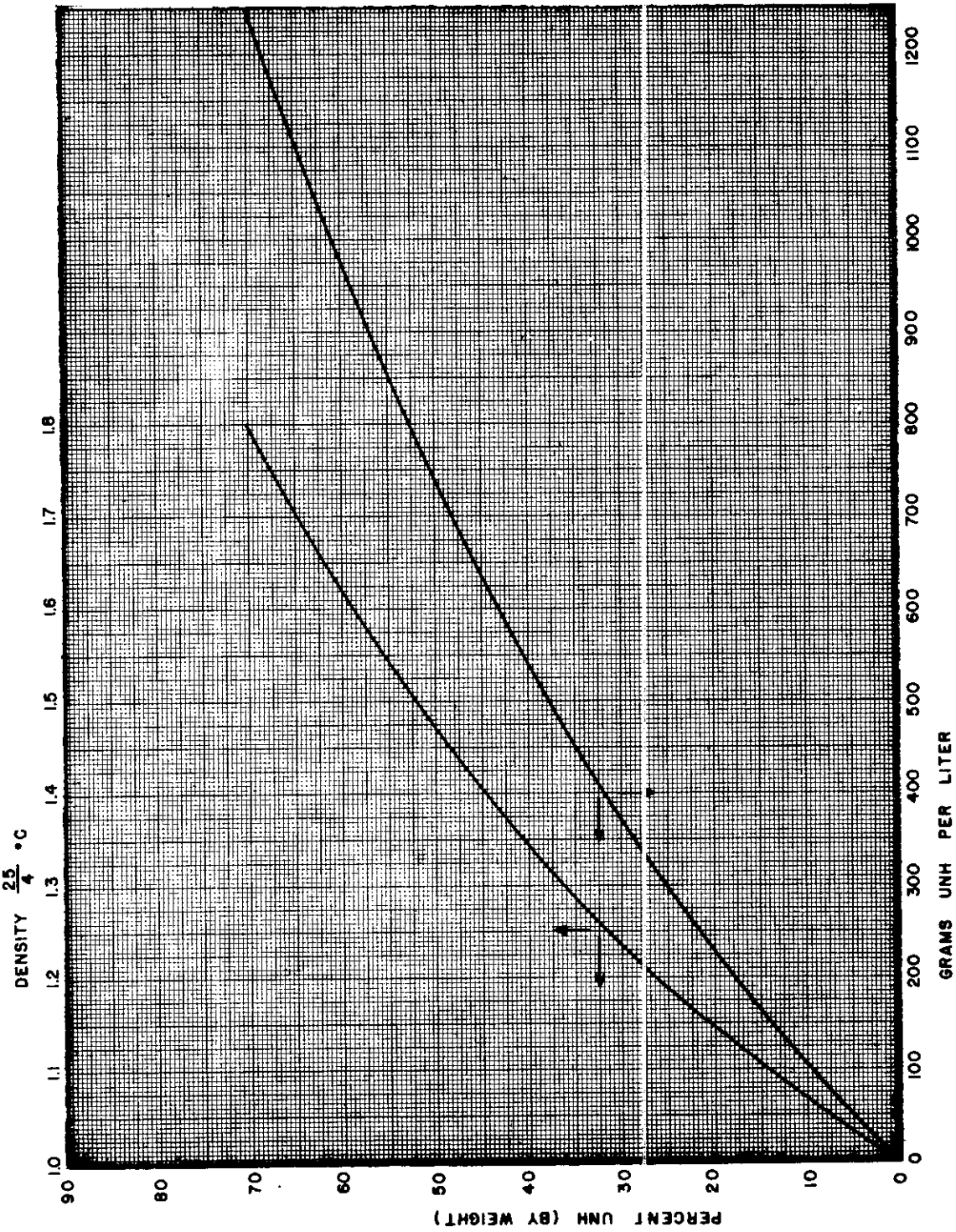

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TABLE IV

Densities of UNH Solutions Containing HNO_3 (at 40 °C)

% HNO_3	40% UNH	5[illegible]% UNH
0	1.338	1.462
2	1.354	1.482
4	1.372	1.501

TABLE V

Densities of UNH Solutions Containing HNO_3 and H_2SO_4

% UNH	% H_2SO_4	% HNO_3	Density (gm/cc.) 20 °C	35 °C	50 °C
16.5	3.0	0.8	1.144	1.138	1.130
21.8	4.0	1.1	1.204	1.197	1.188
27.2	5.1	1.3	1.268	1.260	1.250
35.0	7.1	1.9	1.374	1.364	1.354
37.7	7.1	1.9	1.423	1.418	1.406
40.0	8.1	2.1	1.447	1.436	1.424
43.0	8.1	2.1	1.523	1.511	1.498
48.3	9.0	2.4	1.630	1.630	1.602

TABLE VI

Crystallization Temperatures of UNH-HNO_3 Solutions

% UNH	% HNO_3	Crystallization Temperature (°C)
80	0	46 - 48
	6	49.5 - 51
	12	49
70	0	29 - 30
	5.2	39.
	10.5	42.5 - 43
60	0	< 7
	4.5	20 - 21
	9.0	29 - 30
50	7.5	< 9

UO_2^{+2} forms a number of compounds insoluble in aqueous or alkaline solutions: $Na_4UO_2(CO_3)_3$, $BaUO_2(CO_3)_3$, $NaNO_2(C_2H_3O_2)_3$ (at pH 3-4), $UO_2NH_4PO_4 \cdot XH_2O$, etc. Comparatively few acid-insoluble uranyl compounds are known. Uranyl phosphate is relative insoluble in acid solution, and precipitates slowly on addition of H_3PO_4 to a 1N HNO_3 solution of UNH. Uranyl and sulfate ions form an undissociated complexion, and uranyl phosphate therefore does not precipitate from an acid UNH solution containing sufficient SO_4^{-2} to complex the UO_2^{+2}. This complexing action is applied in the extraction step of the separation process.

As mentioned above, U(IV) is a strong reducing agent; it therefore follows that it is difficult to reduce UO_2^{+2} to U(IV). Uranium exhibits other valence states, but only the tetravalent and hexavalent states are comparatively stable in aqueous solutions.

PROPERTIES OF PLUTONIUM OF PROCESS IMPORTANCE

Introduction

Plutonium is a member of the seventh series of the periodic table, together with such elements as radium, actinium, thorium, uranium and neptunium. It also might be "eka-osmium", falling below osmium in the first column of the eighth group. In its properties it shows little similarity to the platinum metals, (osmium, ruthenium, etc.), however, it resembles uranium and neptunium closely in a number of respects. The U-Np-Pu similarities give emphasis to consideration of the possible existence of a second series analogous to the rare earths. Such a series has been predicted a number of times on the basis of electronic considerations, but uncertainty in the predictions still exists regarding the starting point of the series. The principal choices appear to be among an "actinide", a "thoride" and a "Uranide" series.

Elementary Pu is a bright silvery metallic material, which tarnishes in air with the formation of a black film. Hydrochloric acid (6N) dissolves both the film and the metal, with evolution of H_2. The melting point of the metal is about 630 °C. Two forms are known, with the transition point at about 137-140 °C. The form stable at the lower temperature has a density of about 19.5 gm/cc., and the high-temperature form, about 16.3 gm/cc.

Oxidation States in Solution

Solutions of the tri-(blue-violet), tetra-(brown-green), penta-(colorless), and hexavalent (pink-orange) Pu have been produced. Pu(V) is unstable, and soon disappears by disproportionation to the other valence states. Pu(III) is more stable than Pu(V) but in the presence of air or other oxidants is rather easily converted to Pu(IV). The tetra- and hexavalent forms are the most important from process considerations.

The usual form of the tetravalent ion is one showing a brown color in

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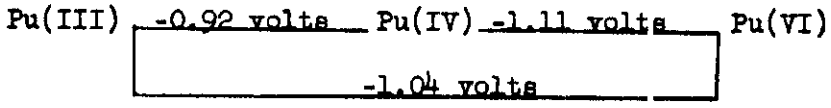
solution. Under certain conditions, in dilute acid (about 0.1-0.5N HCl or HNO_3), a bright green form appears; it is oxidized and reduced more slowly than the brown Pu(IV) and may be precipitated by the addition of HNO_3, 1-2% of the stoichiometric amount of oxalic acid, or ammonia. The green form is usually designated as "abnormal" or "polymeric" Pu(IV) and it is believed to be polymer of variable molecular weight.

Hexavalent plutonium is customarily considered to exist as "plutonyl" ion, PuO_2^{+2}. This viewpoint is suggested by the similarity between compounds of hexavalent plutonium and hexavalent uranium (uranyl), and is supported by the oxidation-reduction behavior of the Pu(IV) - Pu(V) couple.

Pu(IV) can be converted to PuO_2^{+2} by a number of common oxidants including BiO_3^{-1}, $Cr_2O_7^{-2}$, MnO_4^{-1}, Ce^{+4}, and Pb_3O_4; under certain conditions, HNO_3 is also capable of slowly oxidizing Pu(IV) to PuO_2^{+2}. PuO_2^{+2} can in turn be reduced to Pu(IV) (or Pu(III)) by the use of Fe^{+2}, SO_3^-, hydroxylamine, H_2O_2, $C_2O_4^{-2}$; etc. The oxidation-reduction potentials (referred to the normal hydrogen electrode) linking Pu(III), Pu(IV) and Pu(VI) in 1M HNO_3 at 30 °C can be summarized as follows:



The close agreement of the Pu(III) - Pu(IV) and the Pu(IV) - Pu(VI) potential suggests that under the proper conditions all three ions may coexist. In other words, a solution of Pu(IV) may disproportionate; such behavior has actually been observed in dilute HNO_3 solutions. The amount of disproportionation is very small at 1M HNO_3, but increases as the acid concentration is lowered to 0.1M.

Solubility of Plutonium Compounds

Tetravalent plutonium forms a number of relatively insoluble compounds. Some of these, of special interest to the separations process, are listed in Table VII.

Other relatively insoluble compounds of Pu(IV) include the alkali fluorides (K_2PuF_6, etc.), the iodate, chromates, ferrocyanide, arsenate, and several organic compounds.

By comparison to Pu(IV), Pu(VI) forms very few insoluble compounds. The phosphate, fluoride, arsenate and iodate of Pu(VI) are all quite soluble. Insoluble Pu(VI) compounds include $NaPuO_2Ac_3$ and various "diplutonates", (postulated to be $BaPu_2O_7$, $CaPu_2O_7$, $K_2Pu_2O_7$, etc.) formed in basic solutions. The difference between the solubilities of analogous Pu(IV) and Pu(VI) compounds forms the basis for the extraction and decontamination process now in use.

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TABLE VII

Solubilities of Some Pu^{+4} Compounds

Compound	Conditions	Solubility (mg. Pu/liter*)
Phosphate	20% UNH, 1N H_2SO_4, 0.6M H_3PO_4	about 600
"	1M HNO_3, 0.5M H_3PO_2	17
"	" " " , 0.03M Fe(III)	100
Oxalate	1M HNO_3, 0.05M $H_2C_2O_4$, 0.5M HF	about 30
2 LaF_3 PuF_4	" " " " "	20-70
Hydroxide	Excess base	about 2
Peroxide	1M HNO_3, 7.5% H_2O_2	about 20

*Roughly equivalent to parts per million

CARRIERS FOR PLUTONIUM AND BY PRODUCTS

General Considerations

If an element in solution is not present in sufficient concentration to precipitate directly (i.e., at a "trace" concentration) it can be co-precipitated, or carried, from solution by the formation of a precipitate of an element present in larger amounts. The most efficient carriers are those which have the same crystal lattice type and approximate closely the same lattice parameters (unit cell dimensions) as the compound to be carried - i.e., they form isomorphous crystals. In addition, the element to be carried must form an insoluble (or only very slightly soluble) compound with the precipitating anion. The most widely known application of a process of this sort is the concentration and isolation of radium with the aid of barium sulfate. Radium and barium form isomorphous sulfates, both of which are very insoluble. As a result, even the most minute trace of radium in a solution can be carried on barium sulfate precipitated from that solution.

A carrier may operate, with greater or lesser efficiency, in other ways. If the carrier cation and the trace cation form insoluble compounds with the same anion, good carrying may result even though the compounds are not isomorphous. The mechanism may be one designated as "internal adsorption", in which the trace element is adsorbed on the surface of the carrier as the carrier crystal is growing; in this case, two of the three unit cell dimensions of the carrier and compound being carried may be approximately the same. A certain amount of carrying may be due to more or less strictly electrostatic adsorption; this type of carrying is more efficient at lower concentrations of the ion carried. An element may also be carried if it forms an insoluble double salt with the carrier.

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Carriers for Product

Although a great many compounds are known which will carry Pu(IV) from solution, only two are of prime importance in the separation process; bismuth phosphate ($BiPO_4$) and lanthanum fluoride (LaF_3). Both $BiPO_4$ and LaF_3 carry Pu(IV) from acid solutions when the product is present at any concentration from very minute traces to several times the maximum expected amount in the separations process. $BiPO_4$ is thought to carry Pu(IV) by the "internal adsorption" mechanism discussed above, while LaF_3 may carry Pu(IV) by virtue of double salt ($2LaF_3 \cdot PuF_4$) formation. The efficiency of carrying of Pu(IV) by either of these compounds, but especially by $BiPO_4$ is dependent on the conditions of carrier precipitation; the effect of the various conditions on the efficiency of carrying is discussed in more detail in a later section.

Tetravalent product is also carried by a number of other compounds, including zirconium phosphate, insoluble salts of U(IV) and Th^{+4} (iodate, hypophosphate, oxalate, etc.), metallic hydroxides, and Ce^{+3} and other rare earth fluorides.

Trivalent product is in general carried to a greater or lesser extent by the same compounds which carry Pu(IV), but not necessarily with the same efficiency under similar conditions. As might be expected from a knowledge of the relatively small number of insoluble Pu(VI) (or PuO_2^{+2}) compounds, very few carriers for Pu(VI) are known. The most thoroughly investigated Pu(VI) carrier is sodium uranyl acetate ($NaUO_2(C_2H_3O_2)_3$), which is isomorphous with $NaPuO_2(C_2H_3O_2)_3$. $BiPO_4$ carries Pu(VI) to only a very minor extent (about 1% or less, which may be due to surface adsorption or traces of Pu(IV).)

The same can be said of LaF_3, except that under quite narrowly defined conditions (La/Pu ratio, presence of Fe(III), etc.) as much as 25% of the Pu(VI) present may be carried.

Carriers for Fission Products

Because of the large number of individual species of fission products, it is customary to classify them according to their solubility in the presence of certain anions, especially phosphate and fluoride. A classification of the most important by-products according to phosphate-solubility follows:

(a) Phosphate-soluble

Ba, Sr, Pr, Y, La

(b) Phosphate-insoluble

Zr, Cb

(c) Those possessing two oxidation states with different phosphate solubilities

Ce, Ru

In general, the elements in Class (b) (and oxidized Ce and Ru) are carried quite well by $BiPO_4$ or other insoluble phosphates ($Ce_2(PO_4)_4$ or $ZrO(H_2PO_4)_2$), while those in Class (a) (and reduced Ce and Ru) do not precipitate very well with $BiPO_4$. This basis of classification is of course arbitrary, but the above classification is used because of its applicability to the $BiPO_4$ separations process. A classification according to fluoride-solubilities is also of interest; fluoride-insoluble fission products, carried by LaF_3, include the rare-earths (La, Ce(III), Y, etc.) and also Zr.

The number of carriers for the fission elements are not limited to phosphates or fluorides; a wide variety of carriers for individual or groups of fission products is possible. In general, a fission product A may be removed from solution by addition of a larger amount of non-radioactive A and then addition of an anion which forms an insoluble compound with A. For instance, a trace of radioactive Ba may be removed from solution by addition of inactive Ba and then precipitation of $BaSO_4$.

While not exactly a "carrying" procedure, some fission products can also be removed from solution on silica gel, ion-exchange resins, etc.

Holdback Carriers and Solubilization Agents

Although these agents are not carriers in the sense that the term is used in the above paragraphs, a brief discussion of them is included here since they are of importance in the separations process. Holdback carriers and solubilization agents, rather than removing a trace element (fission products or Pu) by precipitation with a carrier, act to keep the trace element in solution during a precipitation.

A holdback carrier can be best defined by consideration of an actual case. It may be assumed that under certain conditions radiolanthanum will carry to the extent of about 3% with $BiPO_4$. If the radiolanthanum in solution be "diluted" by addition of a relatively large amount of inactive La, $BiPO_4$ then precipitated under otherwise similar conditions may carry less than 1% of the radiolanthanum (although the total amount of lanthanum carried may be 3% or more of the total of radioactive plus inactive La present).

A solubilization agent achieves the same end result, but by a different mechanism than by the dilution effect of a holdback carrier. Specific examples again are used to illustrate what is meant by a solubilization agent. $BiPO_4$, under certain conditions, carries about 95% of the Zr in solution; in the presence of added fluosilicate (SiF_6^{-2}), however, only about 15-20% of the Zr is carried. The mechanism is undoubtedly the known ability of SiF_6^{-2} ions to make Zr phosphate more soluble. Similarly, the presence of Fe^{+3} reduces the carrying of rare earths by $BiPO_4$, but in this case the exact mechanism is not known. Fe^{+3} in sufficient concentration also interferes with the carrying of Pu(IV) by $BiPO_4$ and LaF_3.

BASIC CHEMISTRY OF THE 200 AREA PROCESS

A flow sheet of the entire separations process is given in the accompanying diagrams (Figures 18 and 19). The underlying principle of the various steps of the process are presented below, but first the overall aims of the process are briefly discussed.

The purpose of the 200 Area process is to separate the product, plutonium, from the uranium in which it is formed; to remove practically completely from the Pu the fission activities which are present as by-products of the pile reaction; and to isolate the Pu, free of carrier, in a relatively pure state. At full production levels there is about 250 g. of Pu in a ton of uranium metal, or about 250 parts per million. After dissolution of the uranium metal, and throughout most of the process, the Pu is present in a concentration of about 25 mg/liter, or about 25 parts per million. The amount of fission product radioactivity associated with 1 ton of uranium metal and the accompanying Pu must be reduced by a factor of 10^7 (0.00001% of the original) before the isolation step of the process is reached, so that the isolation process can be safely performed without the large amount of concrete shielding and remote control operations necessary in the previous processing. This 10^7 reduction factor for radioactivity is an example of an expression commonly known as the "decontamination factor" (D.F.), which can be expressed mathematically as follows:

$$\text{D.F.} = \frac{\text{Fission activity initially present}}{\text{Fission activity present at step in question}}$$

The gamma radiations are usually used as an index of the fission activity present, and the decontamination factor thus determined is called the "γ D.F.". A logarithmic method of expressing decontamination factors is also used, and is related to the D.F. by the following expression:

$$dF = \log_{10} \text{D.F.}$$

Therefore, a D.F. of 10^5 is equivalent to a dF of 5, D.F. of 20 equals dF of 1.3, etc.

Metal Solution and Extraction - (Canyon Building)

The uranium slugs as received from the 100 Areas are encased in aluminum jackets, and the first step in the 200 Area process is to remove these jackets by preferentially dissolving them with a solution of NaOH - $NaNO_3$, which does not dissolve the uranium. The uranium metal and accompanying Pu and fission products are then dissolved in hot HNO_3. Some of the fission products (I_2, Xe, Kr, etc.) are evolved during this metal solution step.

The Pu is then removed from the bulk of the uranium by carrying it on a $BiPO_4$ precipitate, with the solution being first treated with $NaNO_2$ to insure

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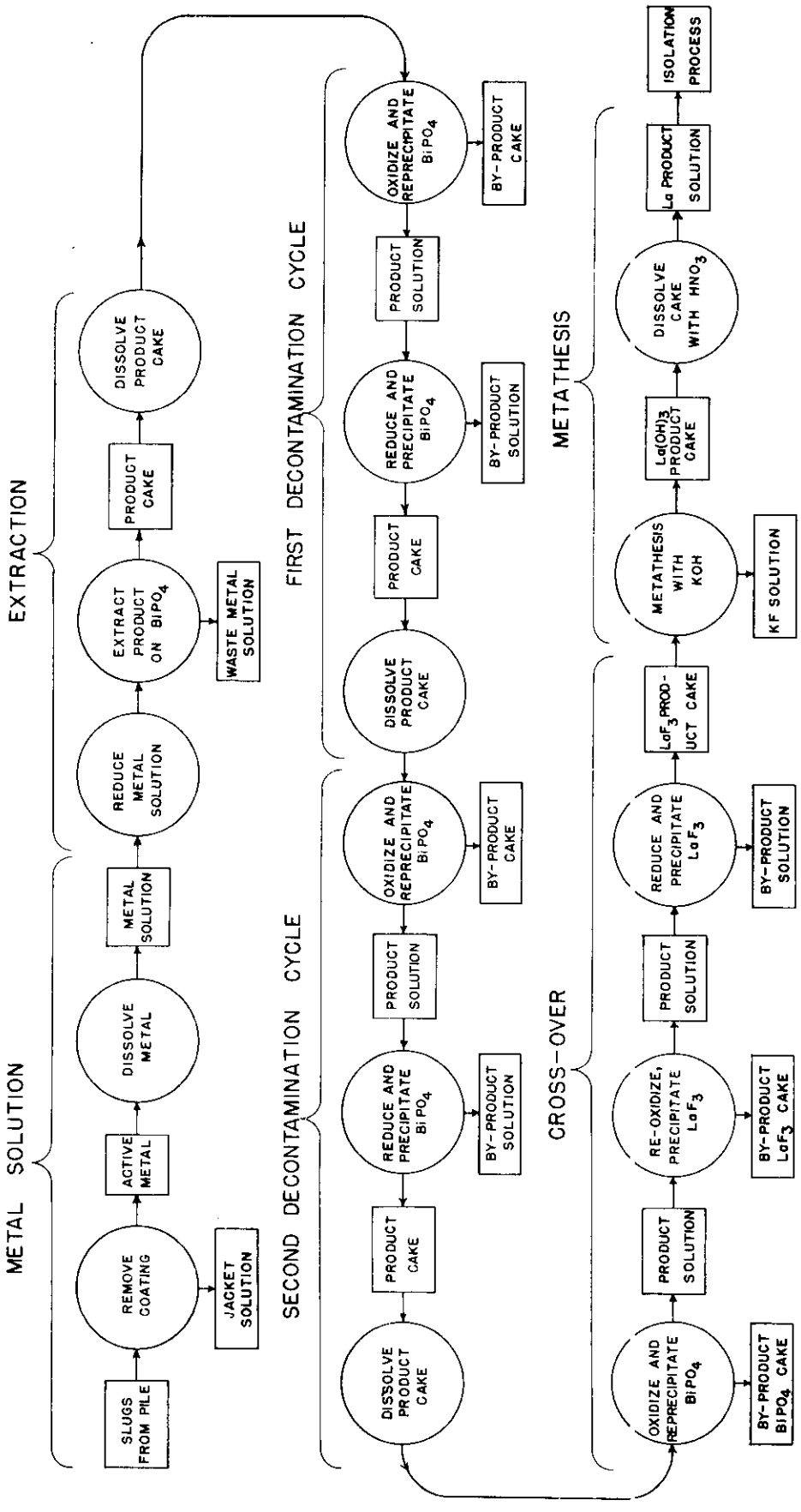
FIGURE 18



SEPARATION PROCESS

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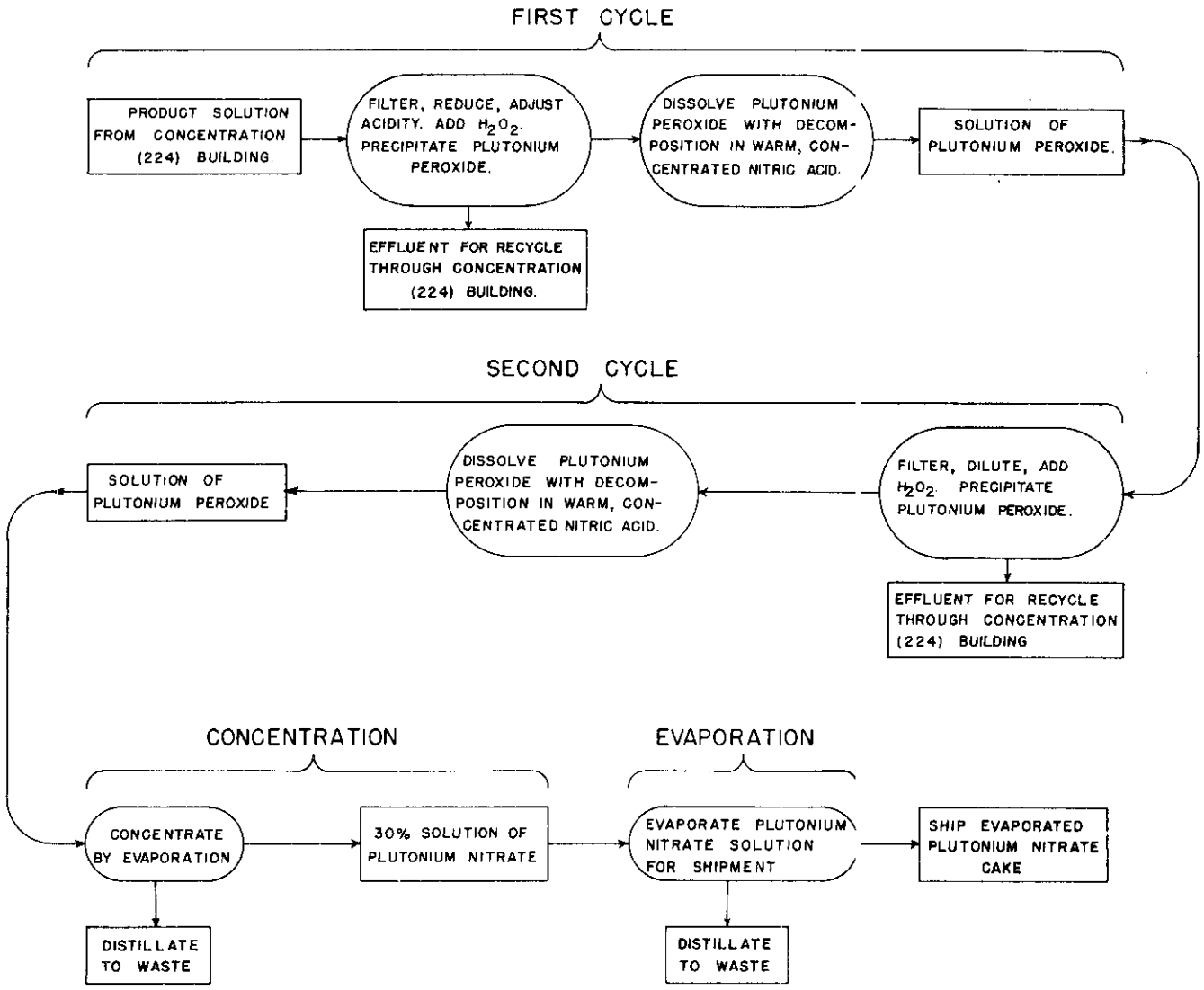
FIGURE 19



ISOLATION PROCESS.

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FLOWSHEET OF PLUTONIUM PROCESS

FIGURE 20

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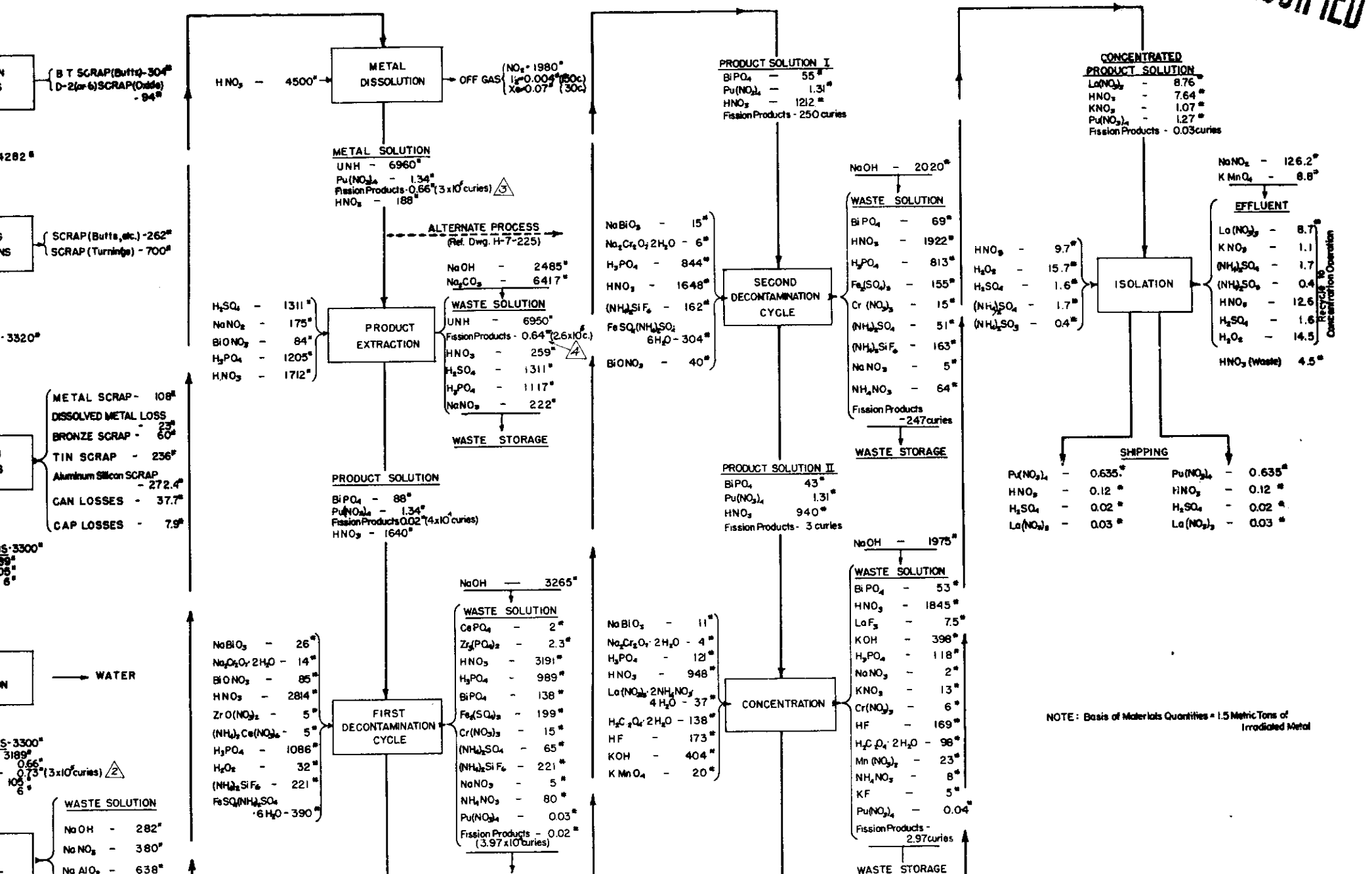

the absence of hexavalent Pu. The $BiPO_4$ is precipitated from the uranium solution in the presence of H_2SO_4, which serves to complex the UO_2^{+2} and thereby avoiding uranyl phosphate precipitation. The $BiPO_4$ extraction precipitate carries, in addition to about 99% of the Pu, about 10% of the fission activity present in the metal solution. The $BiPO_4$ is separated from the waste metal solution by centrifugation and then dissolved in 60% HNO_3.

Decontamination - (Canyon Building)

The fission activity still associated with the product is further reduced by processing the Pu through two "decontamination cycles". Each cycle consists of two $BiPO_4$ precipitations; the first from a PuO_2^{+2} solution, so that the product remains in the solution, and the second from a Pu(IV) solution, so that the Pu is carried on the $BiPO_4$. The precipitation from the oxidized product solution (usually called the "by-product precipitation") removes the major share of the fission products, since those present at this point have already been carried by $BiPO_4$ and are therefore mainly at the phosphate-insoluble class. This by-product precipitation leaves mainly phosphate-soluble fission products in solution with the Pu, so that the following $BiPO_4$ precipitation from a reduced product solution (the "product precipitation") carries only a minor portion of the remaining fission products. The efficiency of fission product removal on the by-product precipitate can be increased by precipitating zirconium and ceric phosphates scavengers in addition to the $BiPO_4$. Such a procedure is used in the first decontamination cycle. The extent of fission product carrying by the product precipitate is decreased by throwing down the $BiPO_4$ in the presence of SiF_6^{-2}, which serves to solubilize the phosphate-insoluble fission products (especially Zr and Cb) which have leaked through to this point.

In the decontamination cycles, the product is oxidized by $NaBiO_3$ in a 7.5M HNO_3 solution of $BiPO_4$ from the previous product precipitation. The PuO_2^{+2} is reduced to Pu(IV) after the by-product precipitation by heating the solution after adding ferrous ammonium sulfate. The Fe^{+3} thus introduced also aside in decontamination by reducing the carrying of rare-earth fission products on the $BiPO_4$ precipitate.

Concentration - (Concentration Building)

At the end of the processing in the Canyon Building, the product has been decontaminated by a factor of about 3×10^5. Before proceeding to the isolation process, an additional factor of at least 33 is needed. This decontamination is obtained in the Concentration Building. Before proceeding with the isolation, it is also necessary to concentrate the product with respect both to the volume of the solution in which it is contained and to the amount of carrier with which it is associated. This is most conveniently done by switching from $BiPO_4$ carrier to a LaF_3 carrier. For this reason, the operations performed in the Concentration Building are commonly referred to as the "cross-over cycle".

The first step consists of oxidation and $BiPO_4$ by-product removal as in the previous decontamination cycles with no scavengers being used. Before reduction,

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LaF_3 is also precipitated from the solution; since most of the fission activity present at this point is fluoride-insoluble (La and other rare earths), an appreciable decontamination factor is obtained by the LaF_3 by-product precipitation. The PuO_2^{+2} in solution is then reduced by oxalic acid, catalyzed by M^{+2} to Pu(IV), which is coprecipitated with LaF_3. The total amount of La(III) used in one charge for this product precipitation is about 2 lbs., which represents a considerable reduction in amount of carrier compared to the amount of Bi^{+3} (about 55 lbs.) used in the extraction precipitation.

The LaF_3 is then converted (metathesized) to the hydroxide by digesting in KOH solution. The $La(OH)_3$ - $Pu(OH)_4$ is dissolved in HNO_3 to give a solution having a volume of about 8 gallons, which is sent to the Isolation Building for the final processing. About 90% of the original product has been carried through to this point, and the fission product gamma activity associated with it has been reduced by a factor of at least 10^7.

Isolation - (Isolation Building)

The $La(NO_3)_3$ - $Pu(NO_3)_4$ solution received from the Concentration Building contains about four times as much La as Pu (at full production levels) and also smaller amounts of other impurities (K, Ca, Bi, Fe, Cr, etc.). The isolation process must free the product of the La and other impurities and result in a final product nitrate at least 95% pure (with respect to non-volatile impurities). The process used to accomplish the isolation consists of two plutonium peroxide precipitations. Pu peroxide is used as the compound for isolation for a number of reasons including the following:

a) It has a low solubility (20-50 mg. Pu/l.), leading to small losses or small amounts of Pu requiring recovery.

b) Under proper conditions (presence of SO_4^{-2}) it settles well, thereby avoiding the necessity for centrifugation or filtration procedures for separation.

c) Only a few other compounds (U, Np, Th) form insoluble peroxides under the conditions used, and these are present only in very small amounts. The purification obtained through plutonium peroxide precipitation is therefore limited mainly by mechanical leak-through of impurities in heels. An exception exists in the case of sulfate ion, however, which probably forms a part of the Pu peroxide molecule and thus is present in the final isolated product at a mol ratio of Pu/SO_4 = of about 2.

The product solution received from the Concentration Building is first filtered and then treated with ammonium sulfite to reduce any hexavalent plutonium which might be present, and then sulfate is added before the precipitation in order to provide conditions favorable to rapid settling of the Pu peroxide. The precipitate is washed and dissolved in HNO_3, the solution is filtered and the Pu then reprecipitated as the peroxide. This second precipitate is washed, dissolved in HNO_3, with decomposition of the peroxide, and the

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$Pu(NO_3)_4$ solution concentrated in a still. This concentrated solution is transferred to a sample can, where it is further concentrated by evaporation to a thick paste.

The final isolated product is usually about 98% pure; the major soluble impurities are La, Fe and Cr. Insoluble impurities are also often associated in variable amounts with the final product. They consist mainly of SiO_2 (from the filter media and filter aid used) and meta-stannic acid (due to the use of Sn stabilized H_2O_2 reagent).

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SECTION C - SEPARATIONS

CHAPTER II - GENERAL DESCRIPTION OF THE SEPARATION PLANT

The fundamental technical basis for the separation process is outlined in the foregoing chapter. From this description it can be seen that the unit operations involved are in themselves quite simple, involving chiefly the precipitation of solids under conditions of adequate agitation and the separation of these precipitated solids from their mother liquors. The operations are conducted essentially at atmospheric pressure and within the temperature limitations of ordinary process steam. By the use of suitable stainless steel alloys, the corrosion problems due to the acid media employed are relatively minor. Hence the design and operation of the commercial equipment to carry out this process would, under normal circumstances, be relatively simple and straightforward. However, the presence of radioactive radiation at the high levels encountered appreciably complicates the design, operation, and maintenance of the plant, and has necessitated a completely new approach which is described in the present chapter.

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CHAPTER II - GENERAL DESCRIPTION OF THE SEPARATION PLANT

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AREA LAYOUT

100 Areas

The production areas of the Hanford Engineer Works, as shown in Figures 1 and 2, are set up in independent, self-contained units located in widely separated districts, because of the hazardous nature of all of the operations involved. With this plant arrangement, damage to any unit would not shut down the plant, since other identical units could be operated and the wide separation of the individual units would prevent accidents in one unit from affecting the operation of the other units. The producing units of the plant are located in two separate operating areas, known as the 100 and 200 Areas. In the 100 Areas are the piles which produce the product. There are three separate and identical plants in these areas known as 100-B, 100-D, and 100-F. Each unit has approximately one-third of the total production capacity of the entire plant. The product of the 100 Area units is the raw material for the 200 Areas.

200 Areas

The 200 Areas are located approximately 6 to 10 miles from the 100 Areas and contain all of the operating facilities required for the storage, separation, isolation and shipment of the product. The facilities are contained in three main areas designated as the 200 North, 200 East, and 200 West Areas.

The 200 North Area (See Figures 3 and 4) contains facilities for the underwater storage of metal after its discharge from the pile. In storage the metal is allowed to "decay" prior to its use in the separation plant. Three separate units known as Metal Storage Basins (212) are provided in the 200 North Area, known as N, P, and R, each handling the production from one 100 Area unit.

The 200 East and 200 West Areas are each separated approximately 2 to 3 miles from the 200 North Area, and, in turn, are separated approximately 4 miles from each other (See Figure 1). The 200 East and 200 West Areas contain all of the facilities for the separation, isolation and shipment of the product, and each contains its own separate power and service facilities as shown in Figures 5 to 18. The facilities for the separation of the product are comprised in three separate and identical main units, known as T, U and B. The T and U units are located in the 200 West Area, and the B unit in the 200 East Area. Originally, a fourth unit designated as C was projected to be located in the 200 East Area, but was subsequently abandoned as further progress in process development indicated it to be unnecessary.

Each separation unit consists of the following buildings arranged with respect to each other as shown in the outside piping diagram, Figure 31:

Canyon Building (221)

The operations of coating removal, metal dissolving, extraction, and

[redacted]

[redacted]

FIGURE 1

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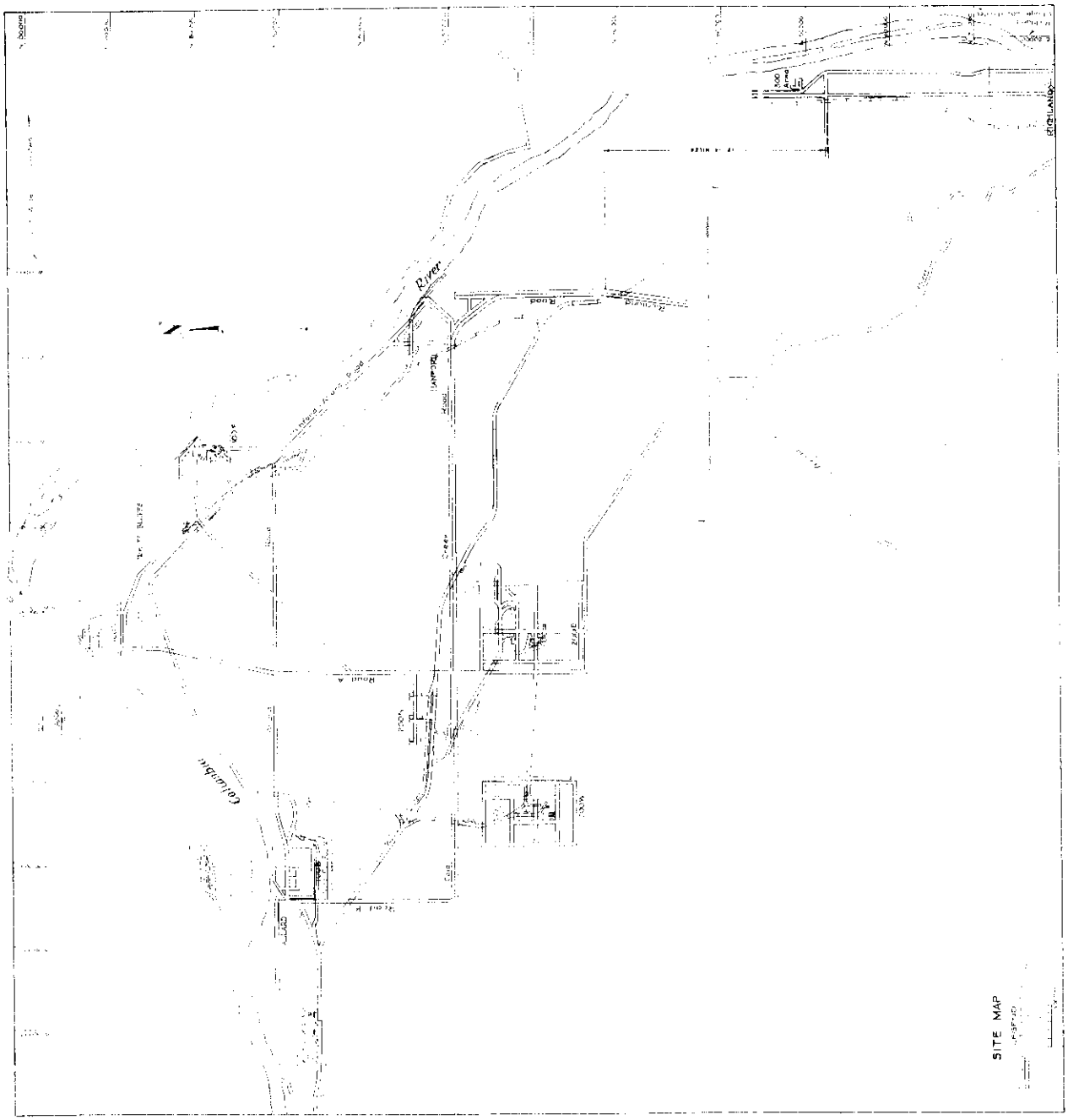

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FIGURE 2

PLOT PLAN SHOWING REFERENCE POINTS FOR HANFORD ENGINEER WORKS

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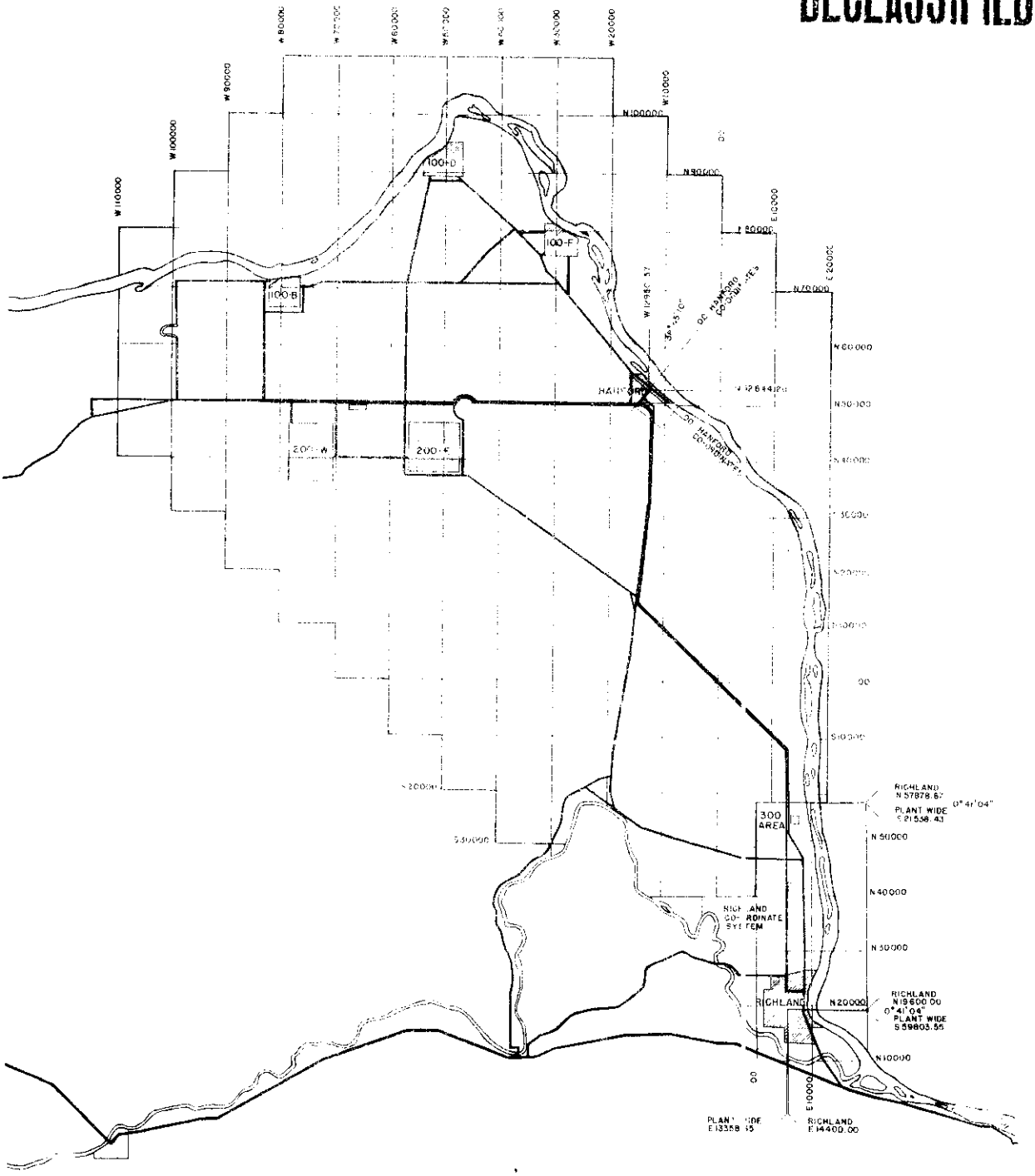

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FIGURE 3

200 NORTH AREA PLOT PLAN

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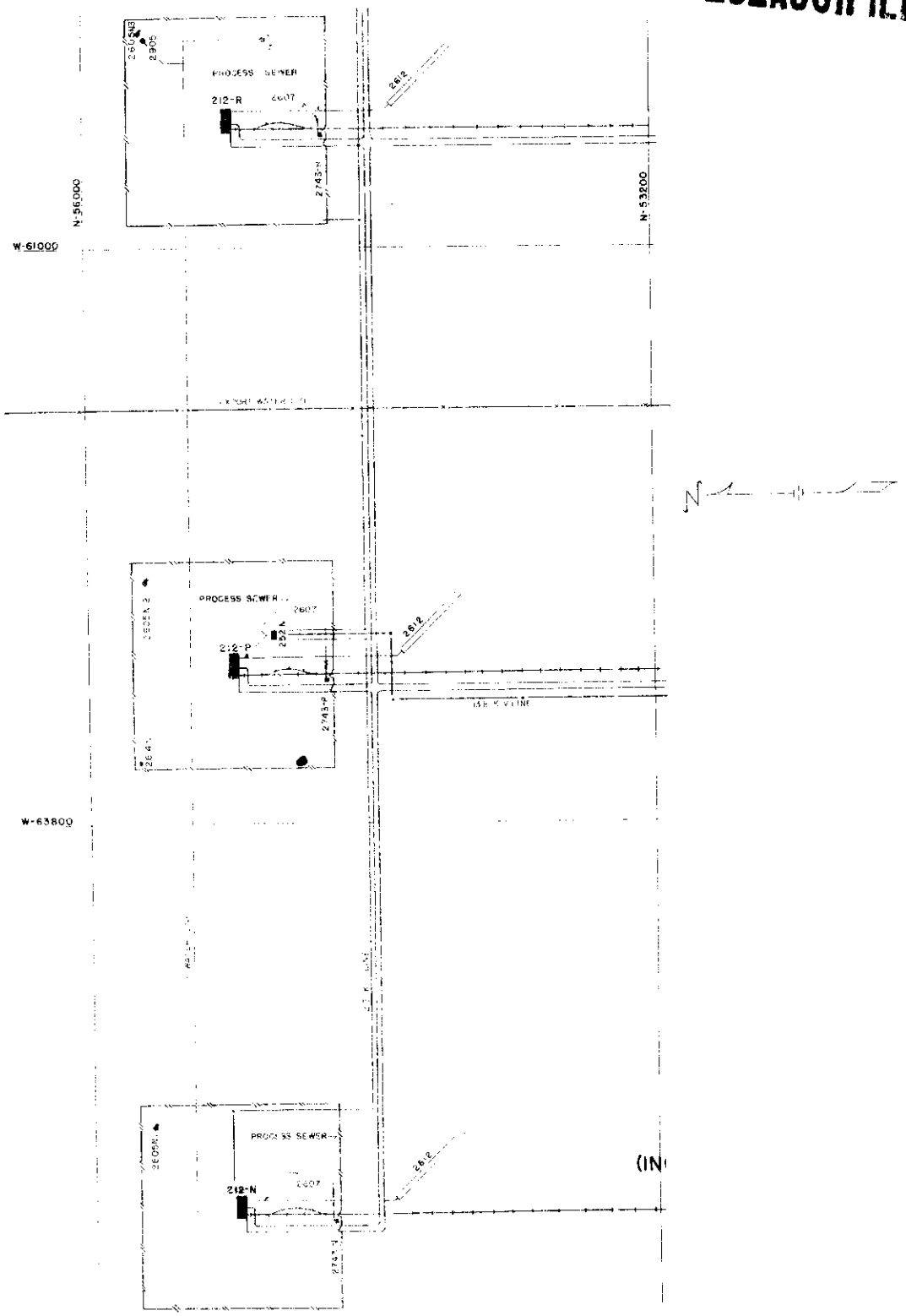

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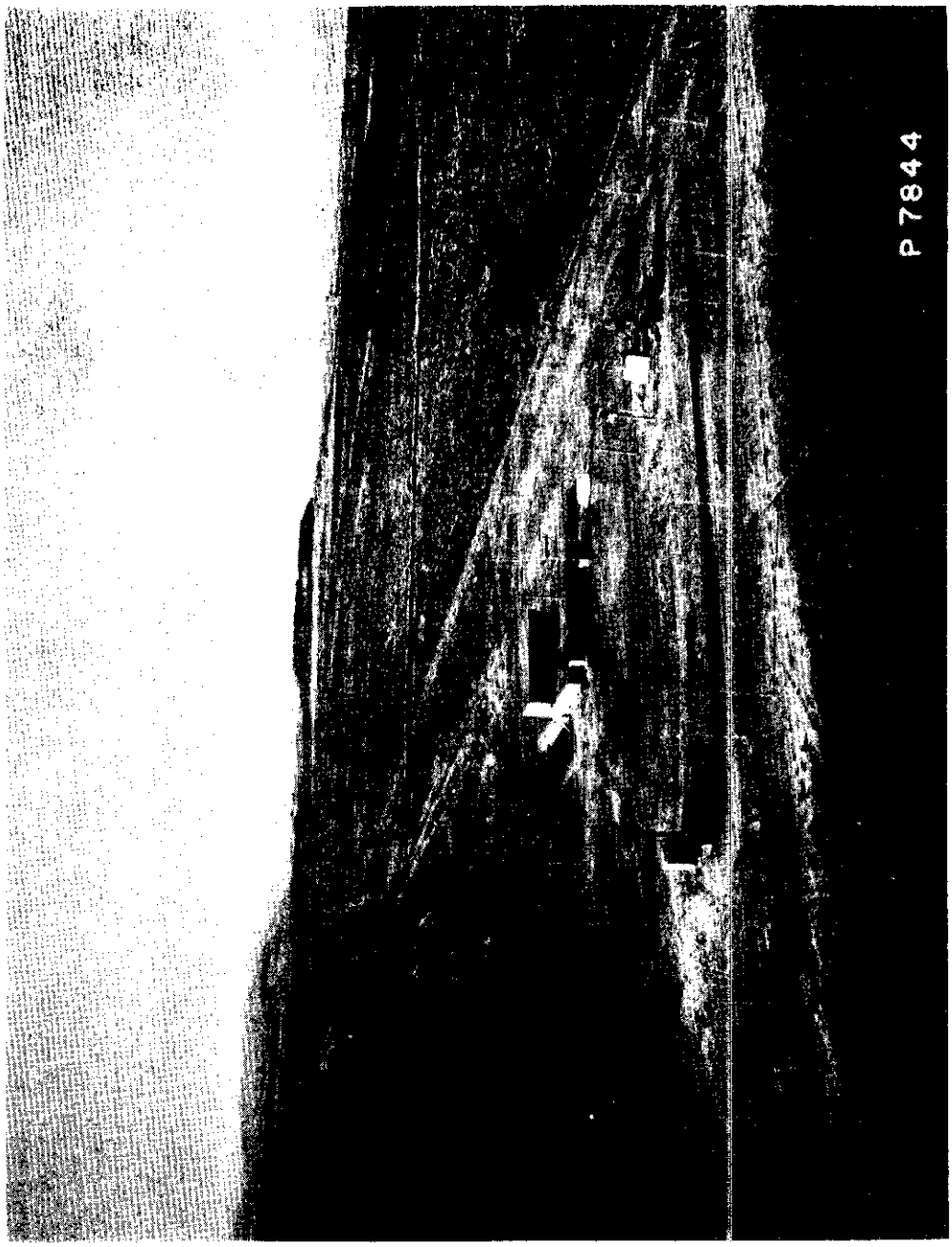
FIGURE 4

200 NORTH AREA



[redacted]

FIGURE 5

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200 EAST AREA PLOT PLAN

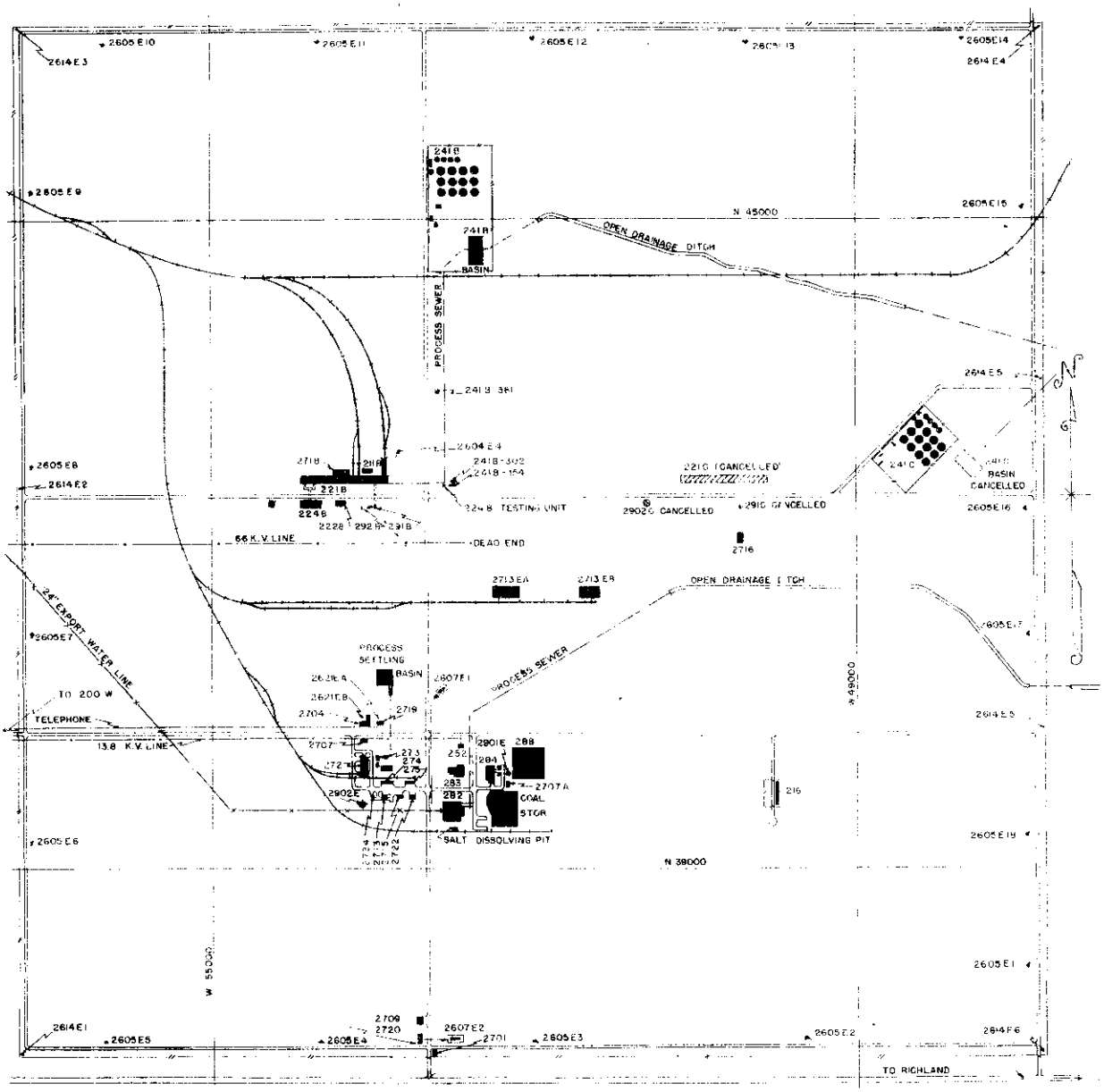

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FIGURE 6

200 EAST AREA (LOOKING NORTHEAST)

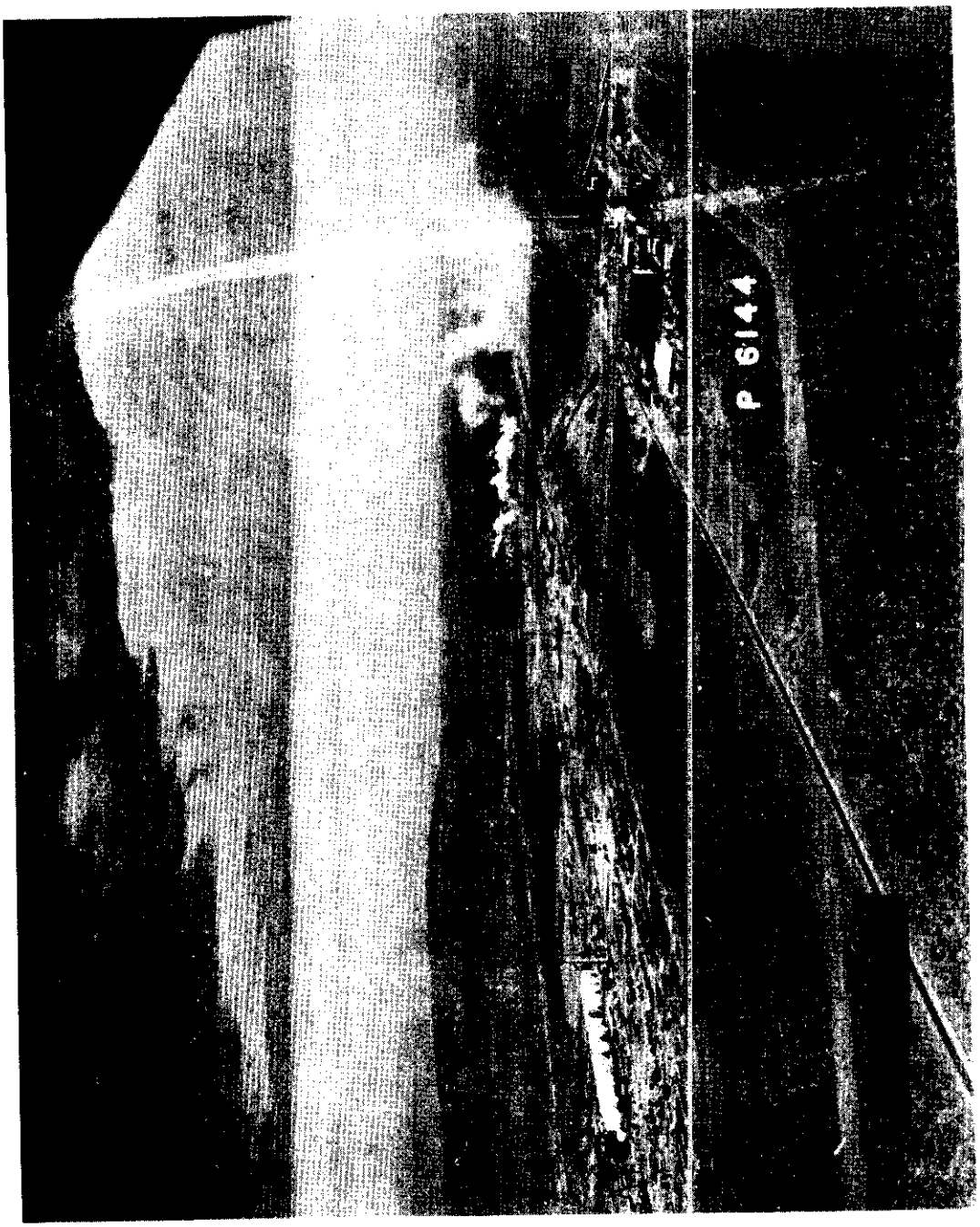
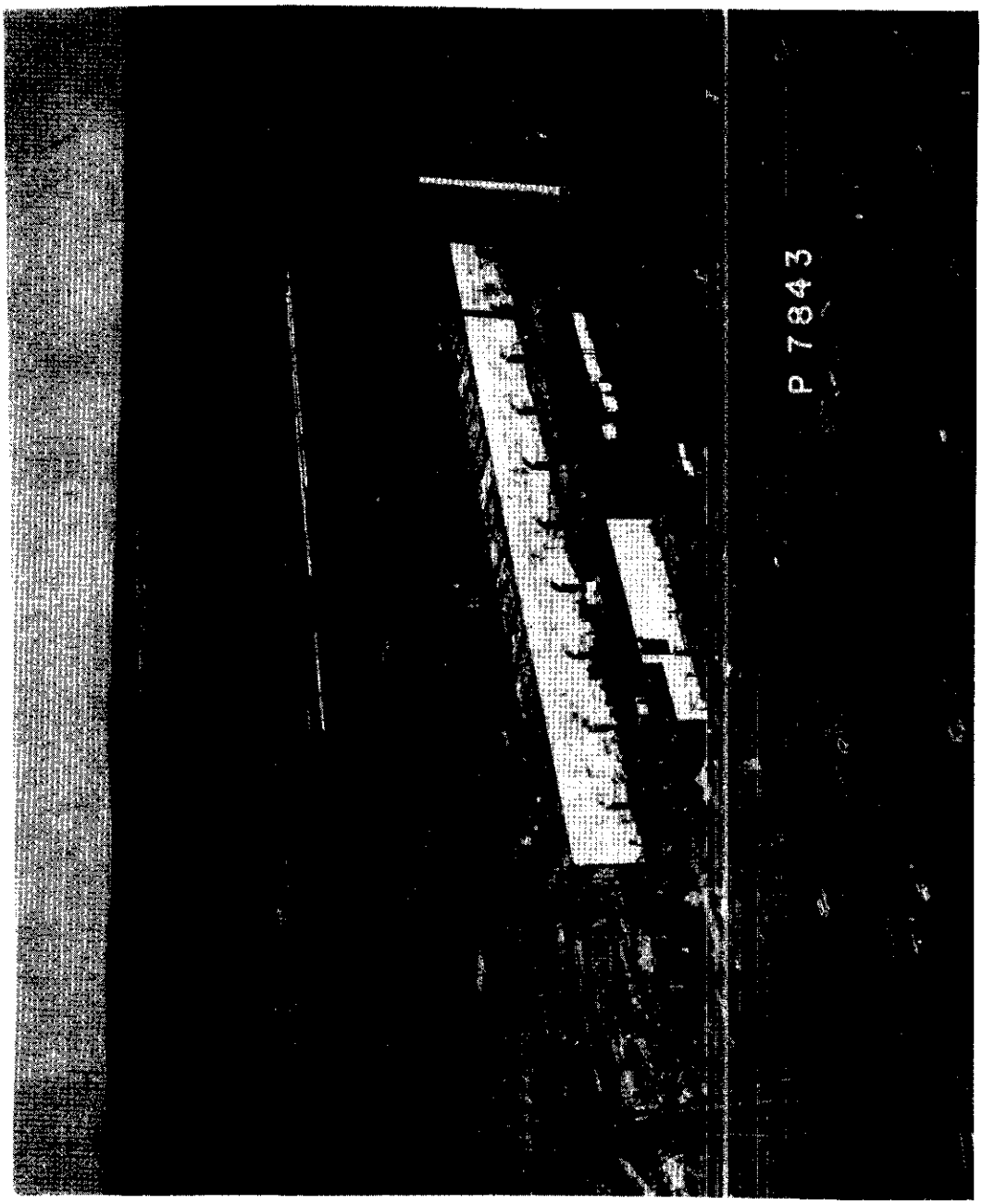


FIGURE 7



200 B AREA PROCESS GROUP (LOOKING NORTHEAST)

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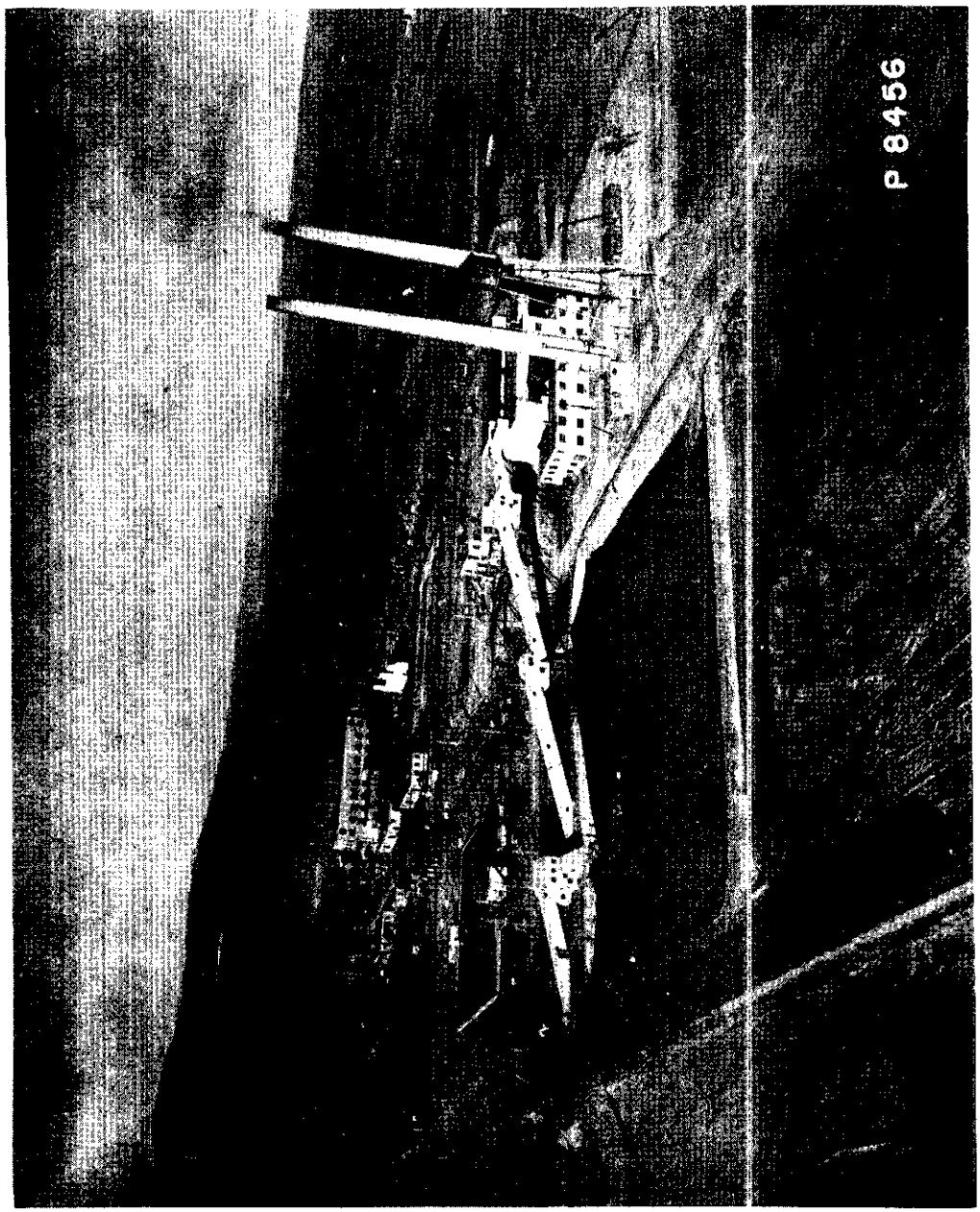
FIGURE 8



200 EAST AREA POWER AND SERVICE GROUP (LOOKING NORTHEAST)

[redacted]

FIGURE 9

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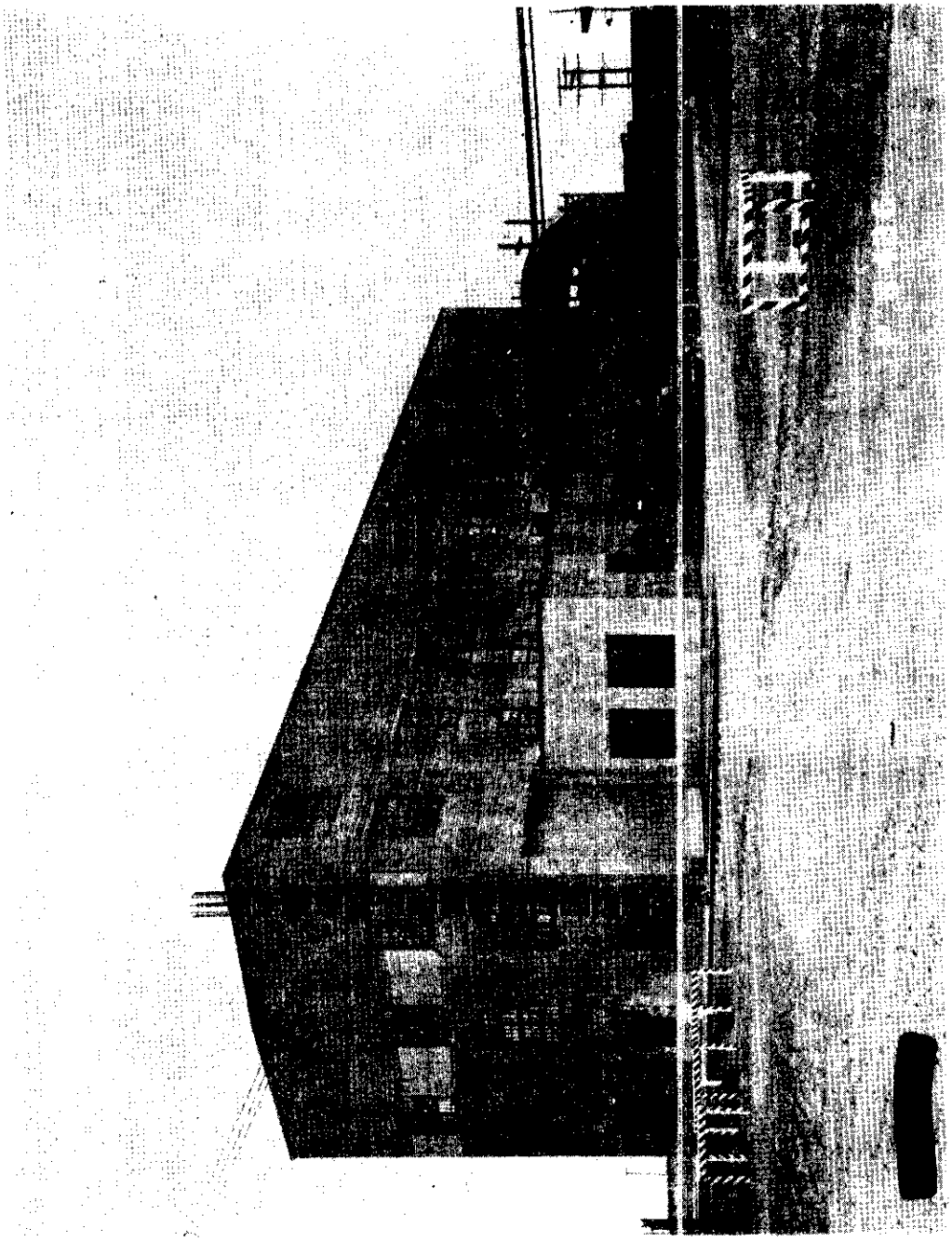

272 EAST AREA SHOP (LOOKING NORTHWEST)

[redacted]

[redacted]

FIGURE 10

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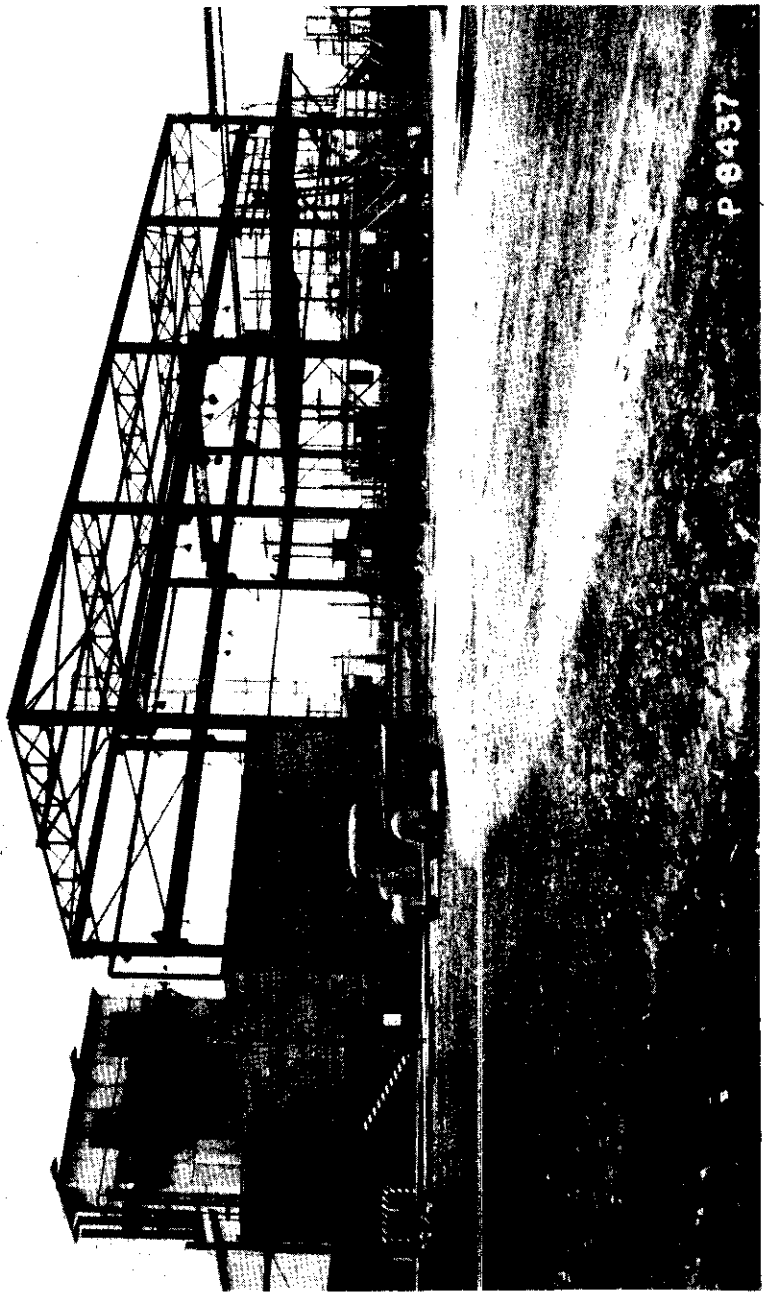

272 EAST HEAT TREATMENT BUILDING (LOOKING WEST)

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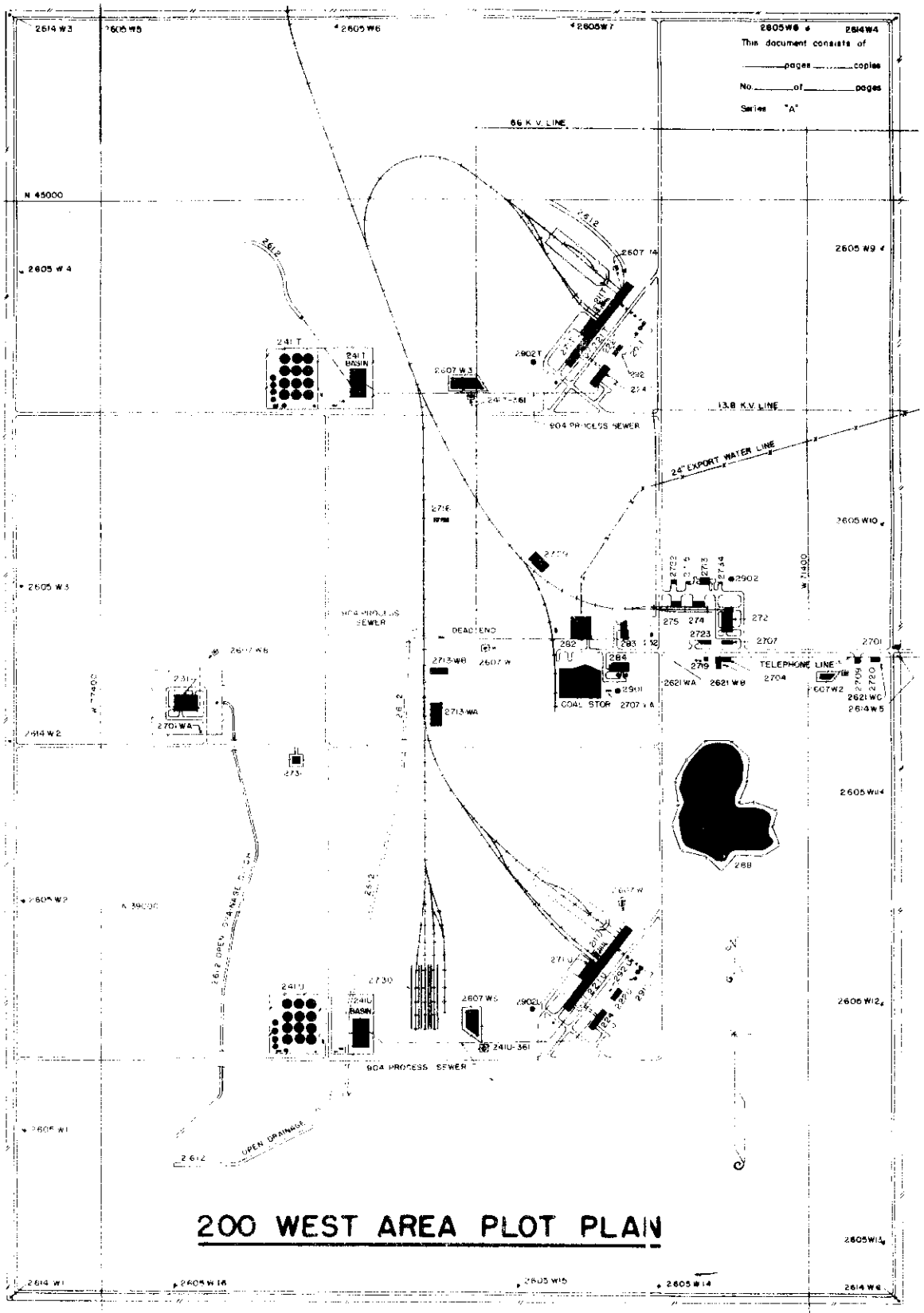
FIGURE 11



200 WEST AREA PLOT PLAN

[redacted]

FIGURE 12

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200 WEST AREA

FIGURE 13

200-T AREA PROCESS GROUP

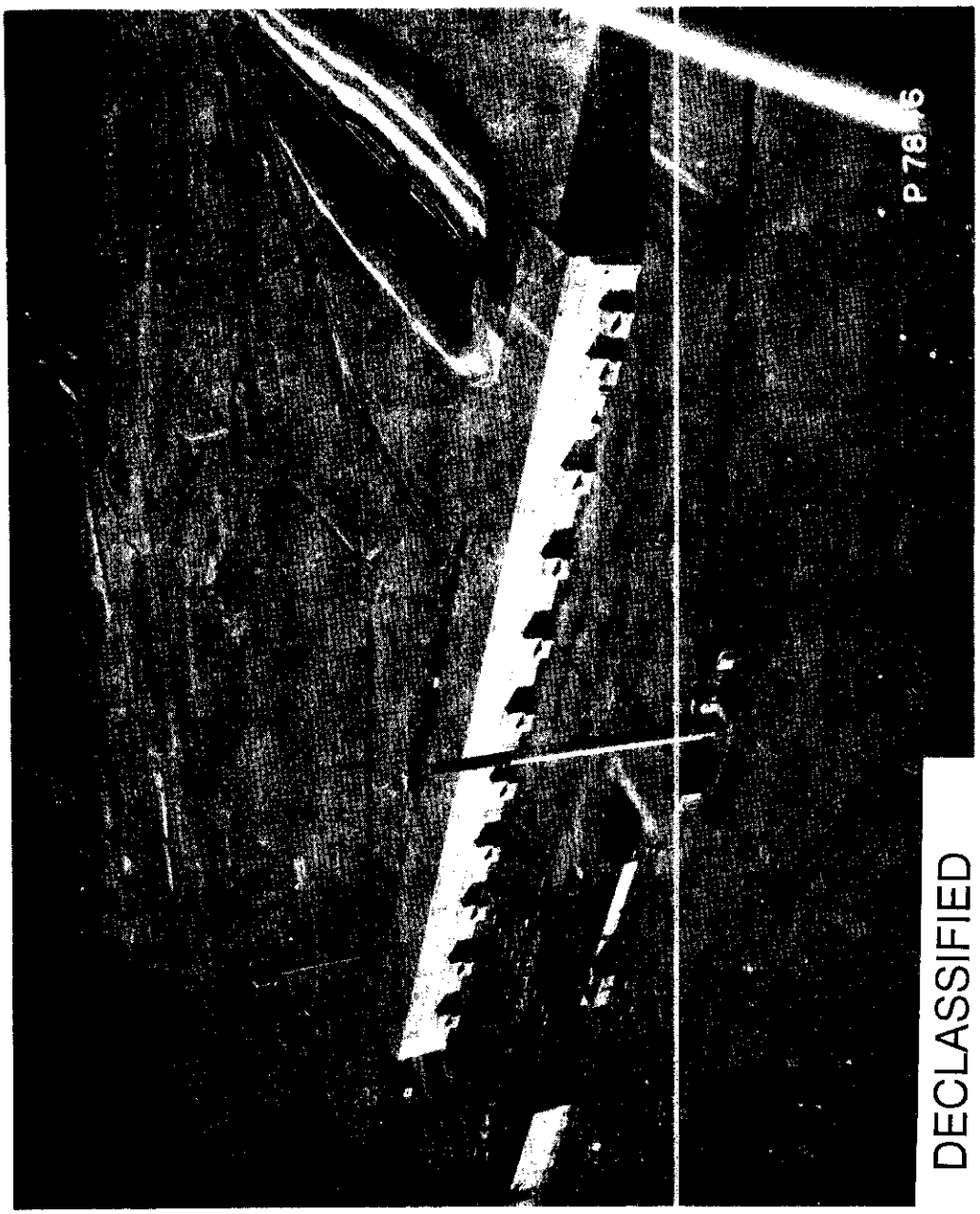

FIGURE 14

200-U AREA PROCESS GROUP

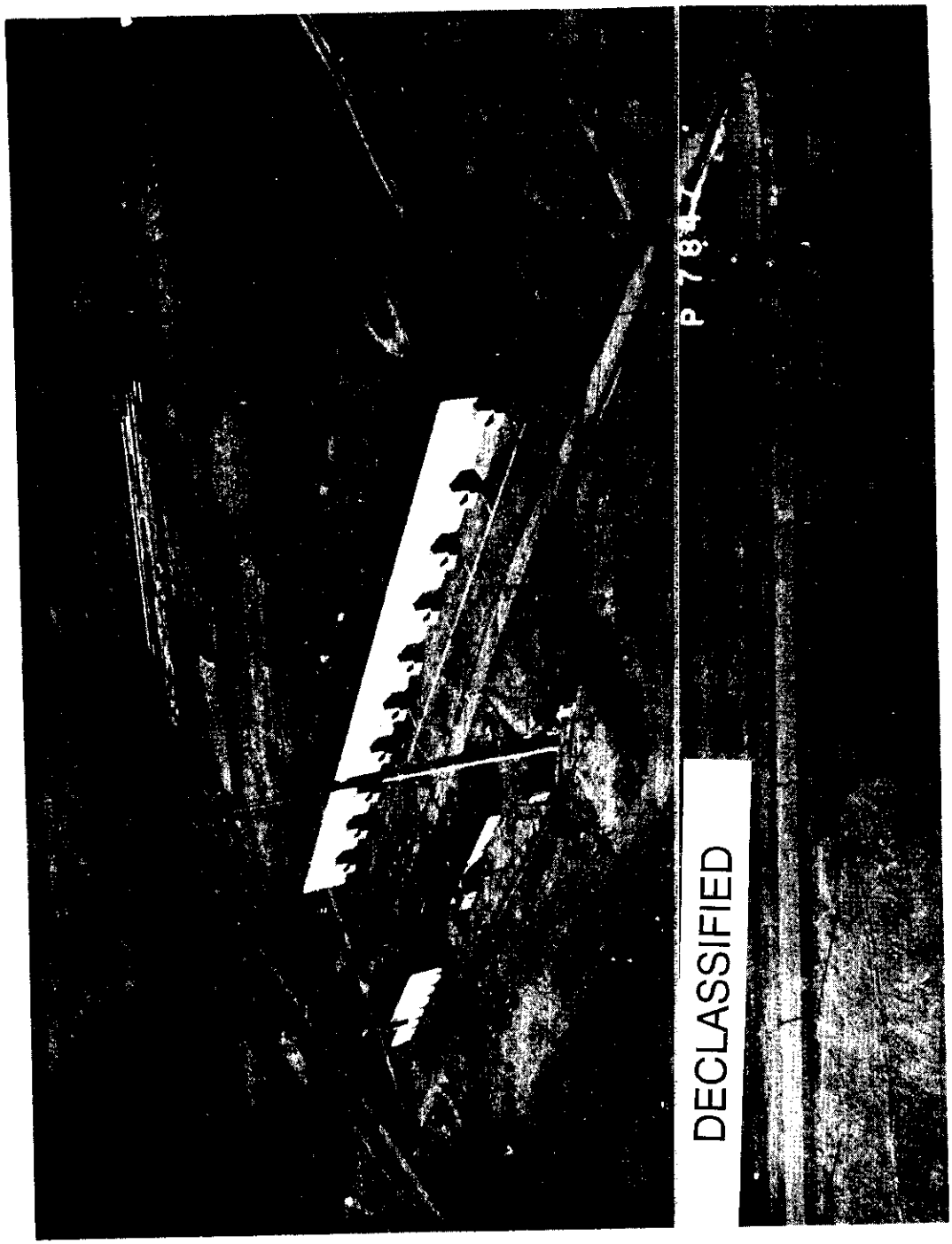

FIGURE 15

200 WEST AREA POWER AND SERVICE GROUP

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FIGURE 16

282-W RESERVOIR AND PUMP HOUSE

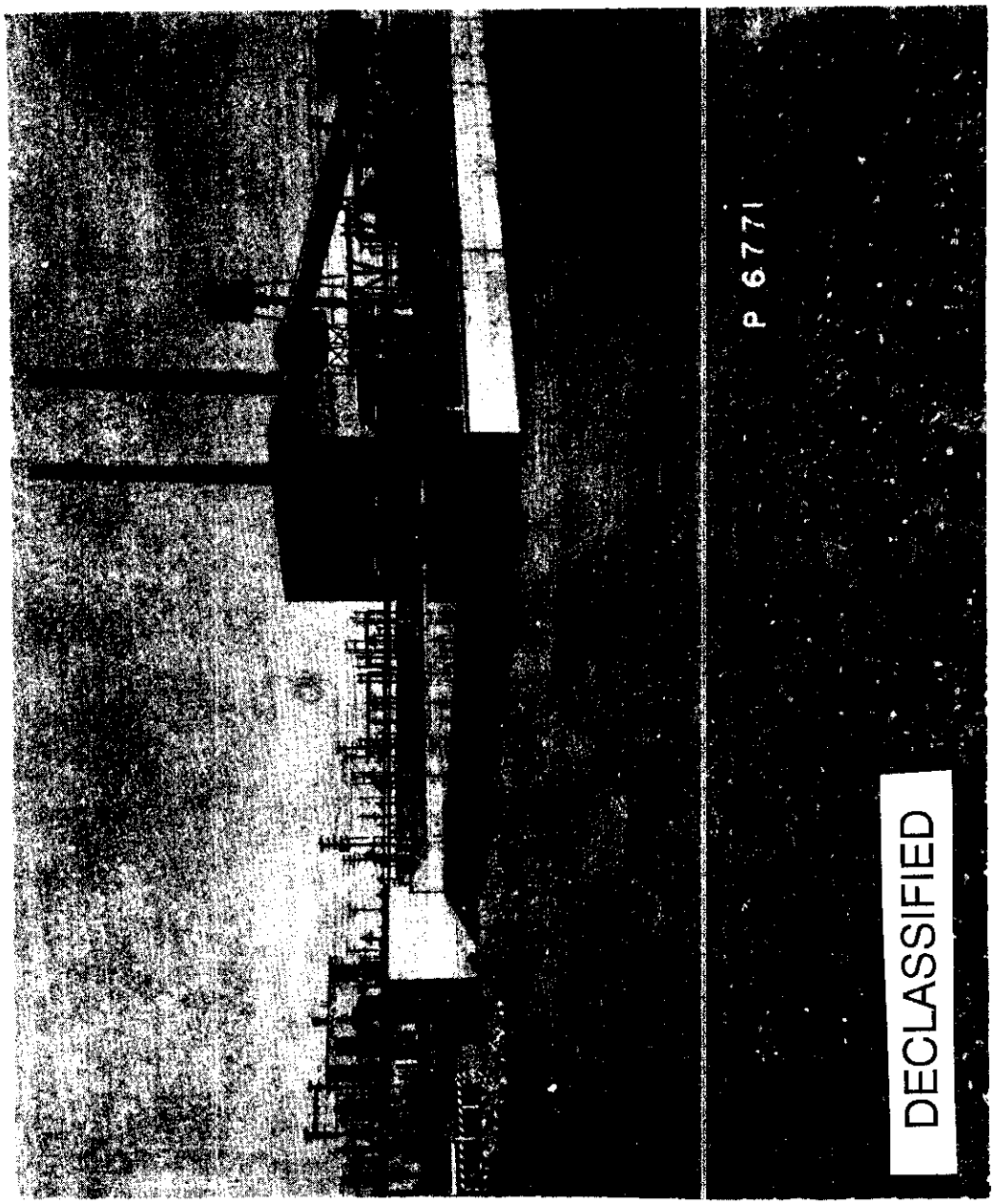

FIGURE 17

283-W FILTER PLANT

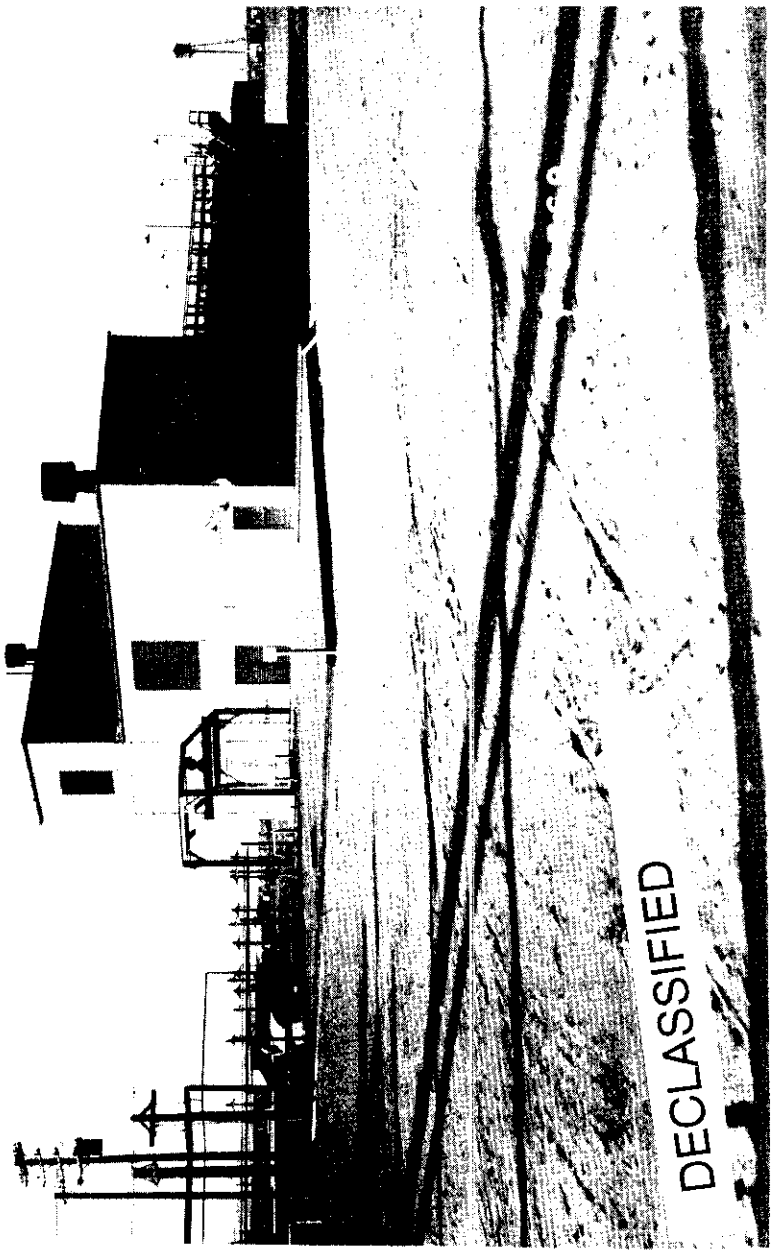

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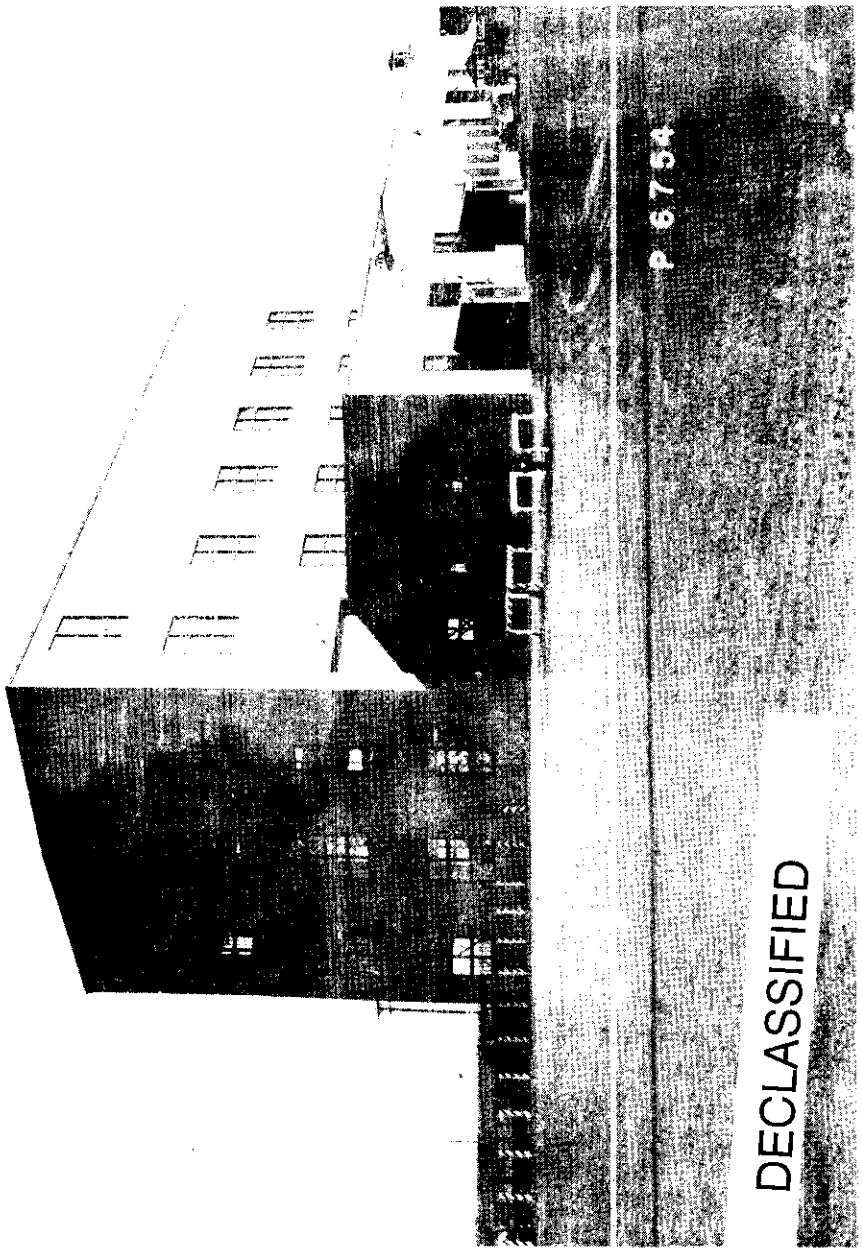
FIGURE 18

272-W AREA SHOP (LOOKING SOUTHEAST)



the major portion of the decontamination operation are carried out in this building. (See Figure 19)

Concentration (224) Building

The concentration operation and the remainder of the decontamination operation is carried out in this building. (See Figure 20)

Waste Disposal (241) Building

The radioactive by-products of the separation process are stored here in shielded tanks, and innocuous wastes are disposed of by introduction into reverse flow wells or open surface ditches. (See Figures 21 and 22)

Fan House and Process Stack (291) Building

This building provides the facilities for handling the main ventilation requirements for Buildings 221 and 224 and for disposing of gaseous radioactive by-products into the atmosphere. (See Figures 23 and 24)

Stack Monitor (292) Building

This building houses the health monitoring equipment for the 291 Building stack. This equipment is described in Chapter X. (See Figure 25 for view of this building in the T Area)

Tank Farm (211) Building

This provides for the bulk storage of chemicals used in the area in connection with the above buildings. (See Figures 26 and 27)

Service (271) Building

Provides facilities for preparation of process chemicals and serves as administrative headquarters for the operation of the process. (See Figure 28)

Control Laboratory (222) Building

This building houses the control laboratories for the process. (See Figure 29)

One unit known as Isolation (231) Building is located in the 200 West Area and serves the three separation units. Shipping of the product is carried out from this building. (See Figure 30)

[redacted]

FIGURE 19

221-T CANYON BUILDING

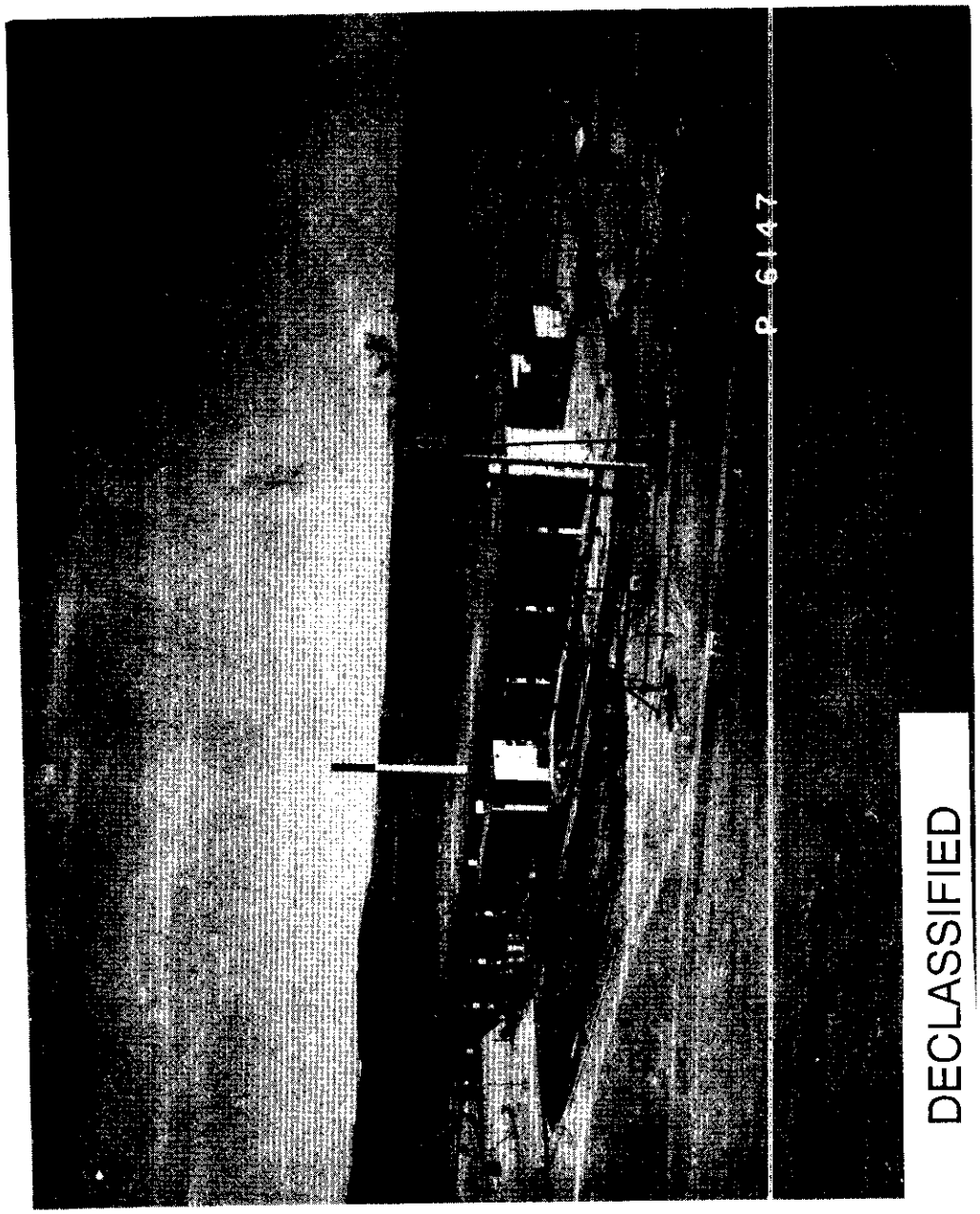


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FIGURE 20

224-T CONCENTRATION BUILDING

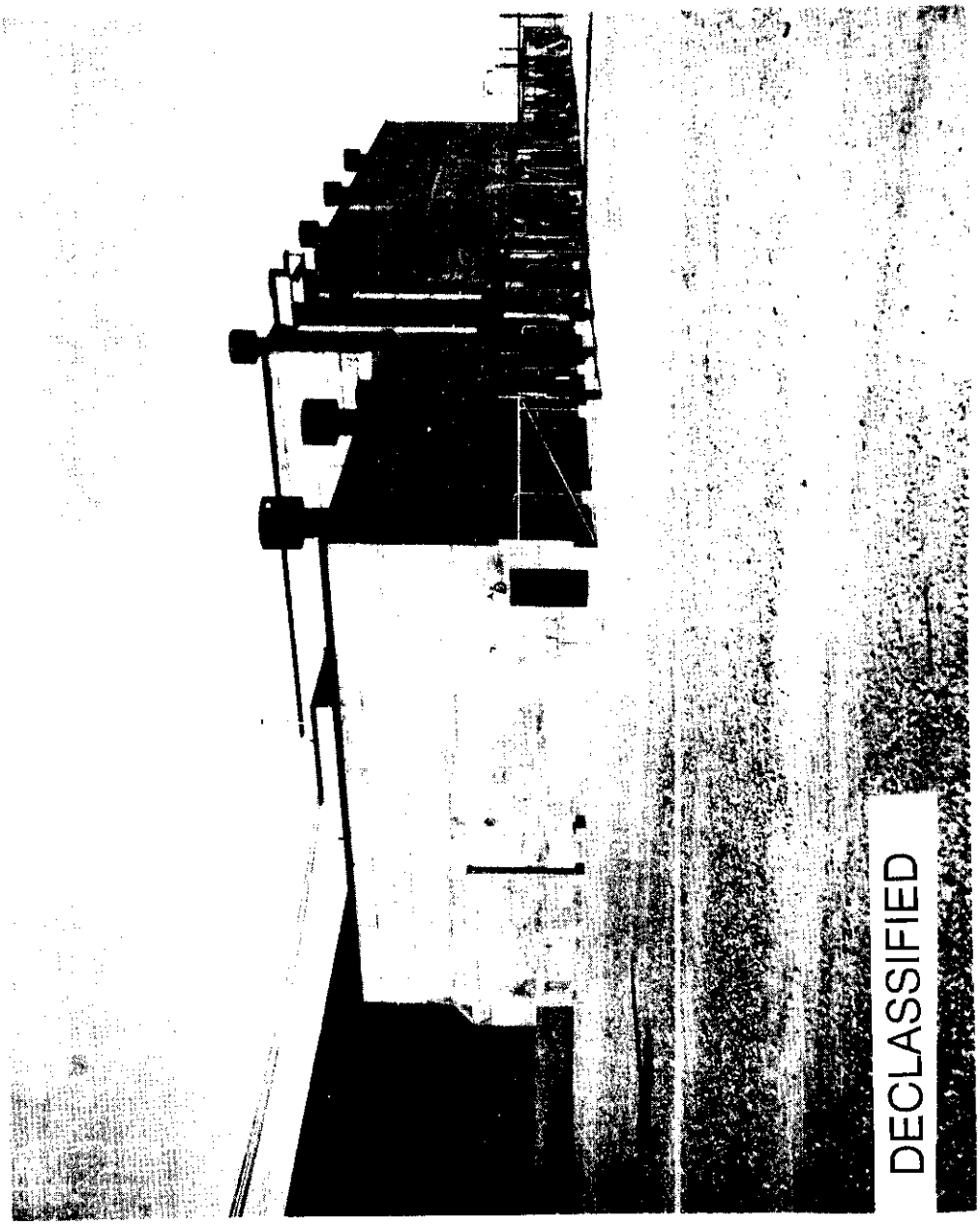

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FIGURE 21

241-T WASTE DISPOSAL BUILDING

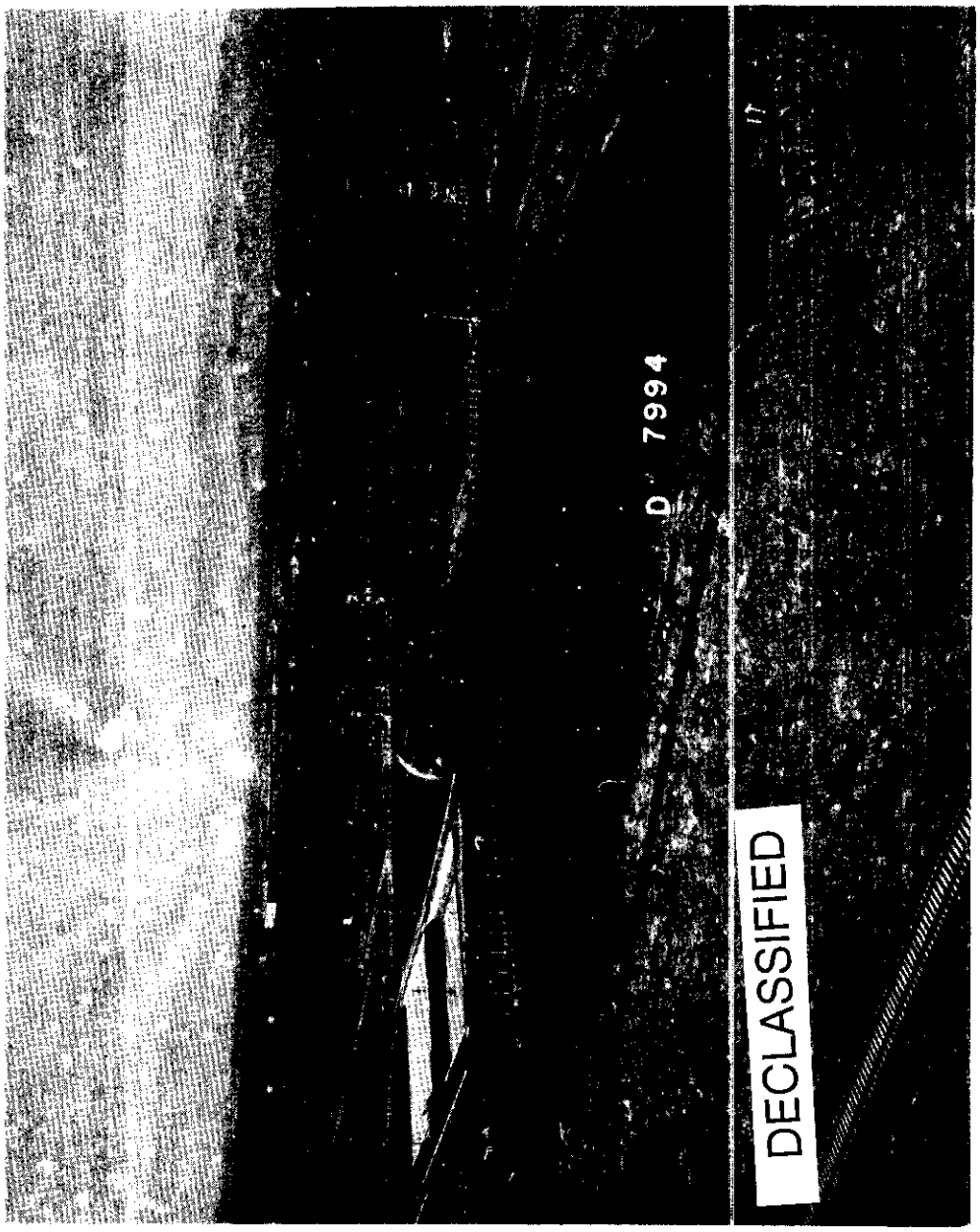
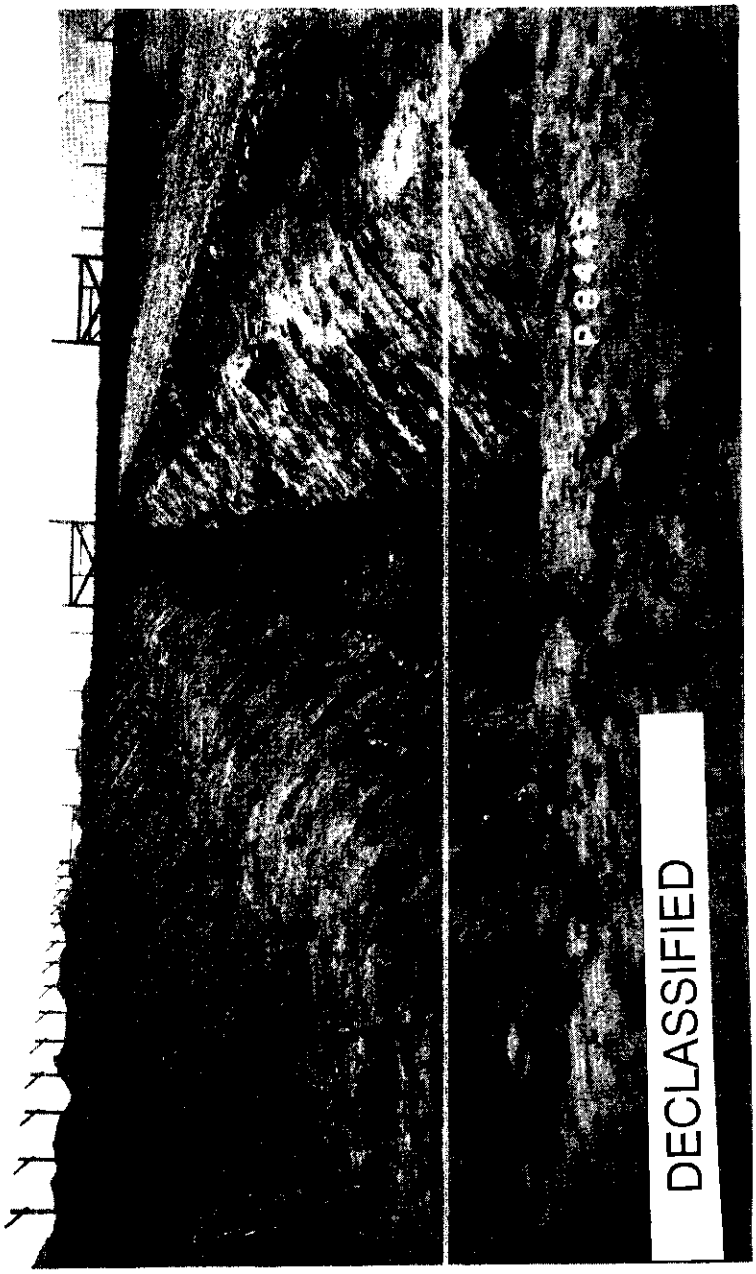


FIGURE 22



216-E PROCESS WASTE AREA TRENCH

FIGURE 27

291-T FAN HOUSE AND PROCESS STACK

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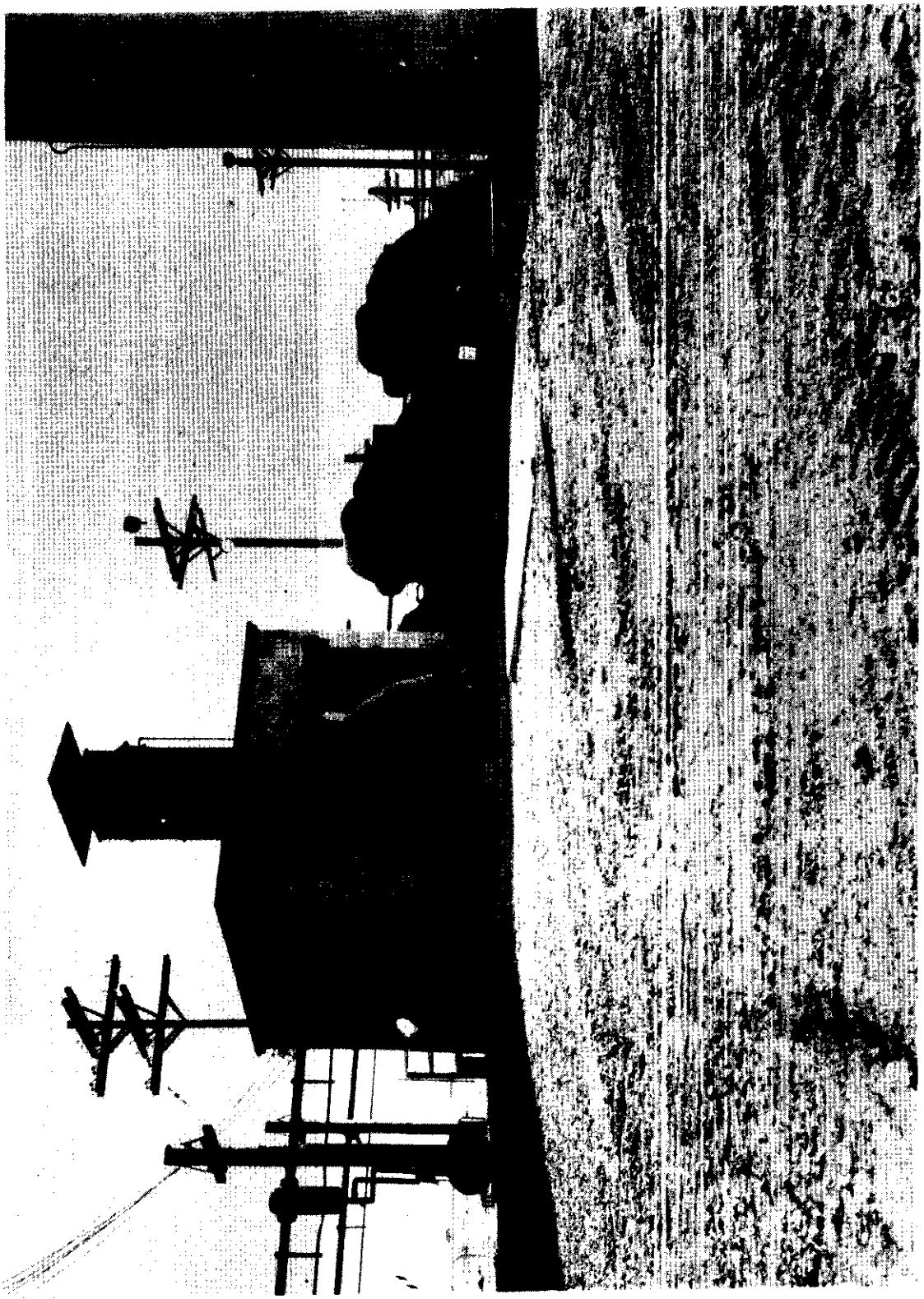
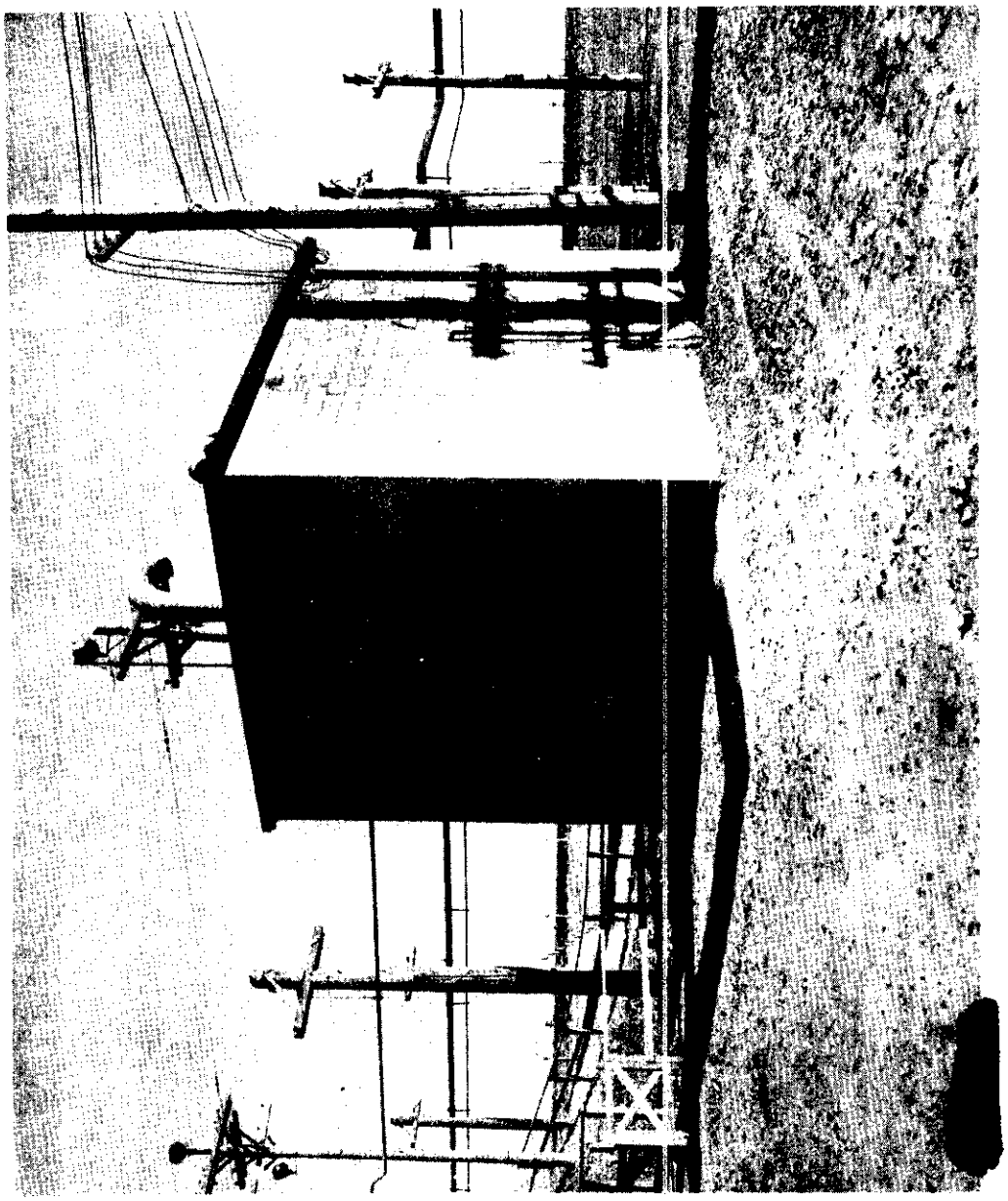
FIGURE 24



291-T FAN HOUSE AND PROCESS STACK

FIGURE 25



292-T STACK MONITOR BUILDING

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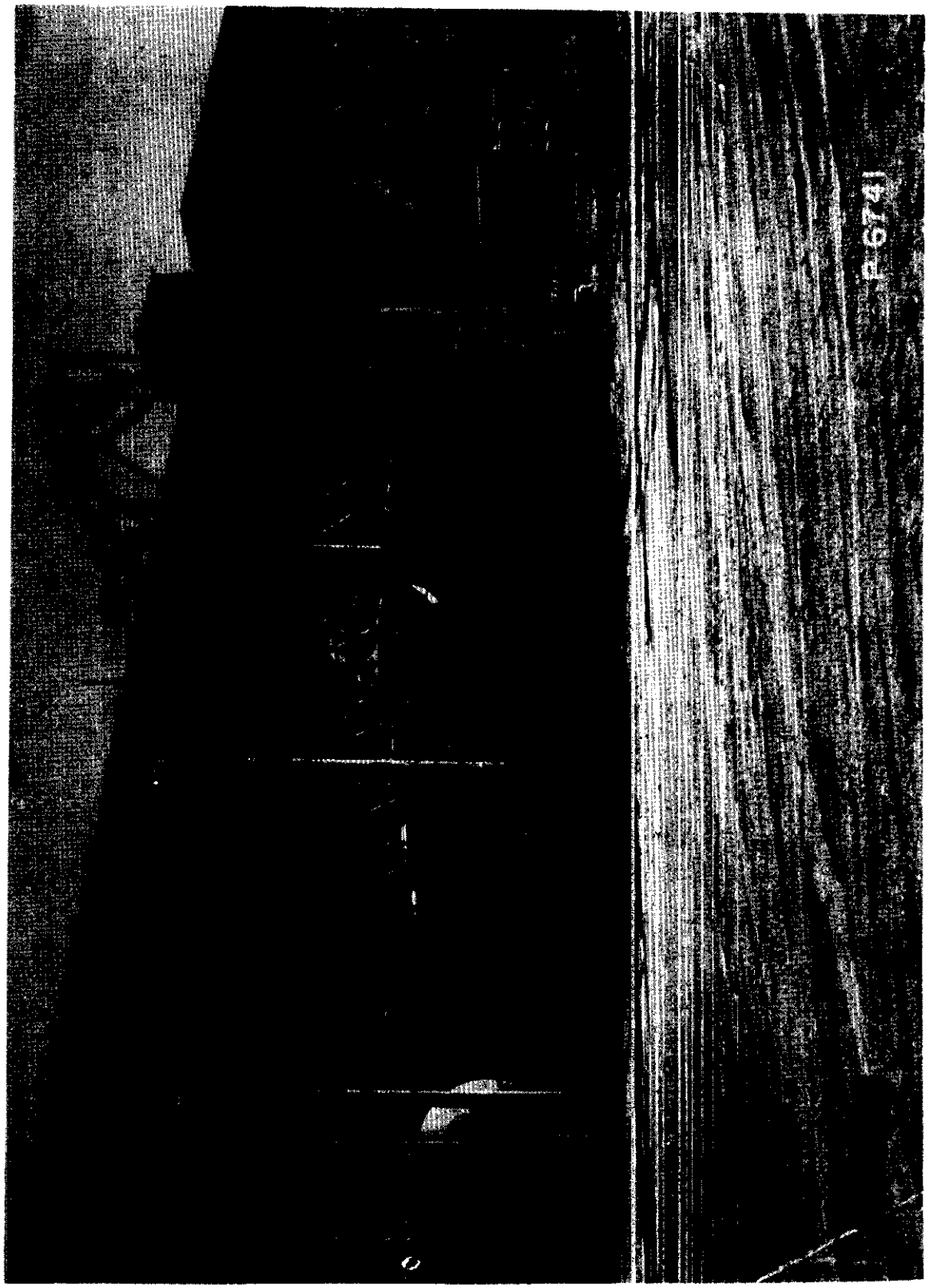
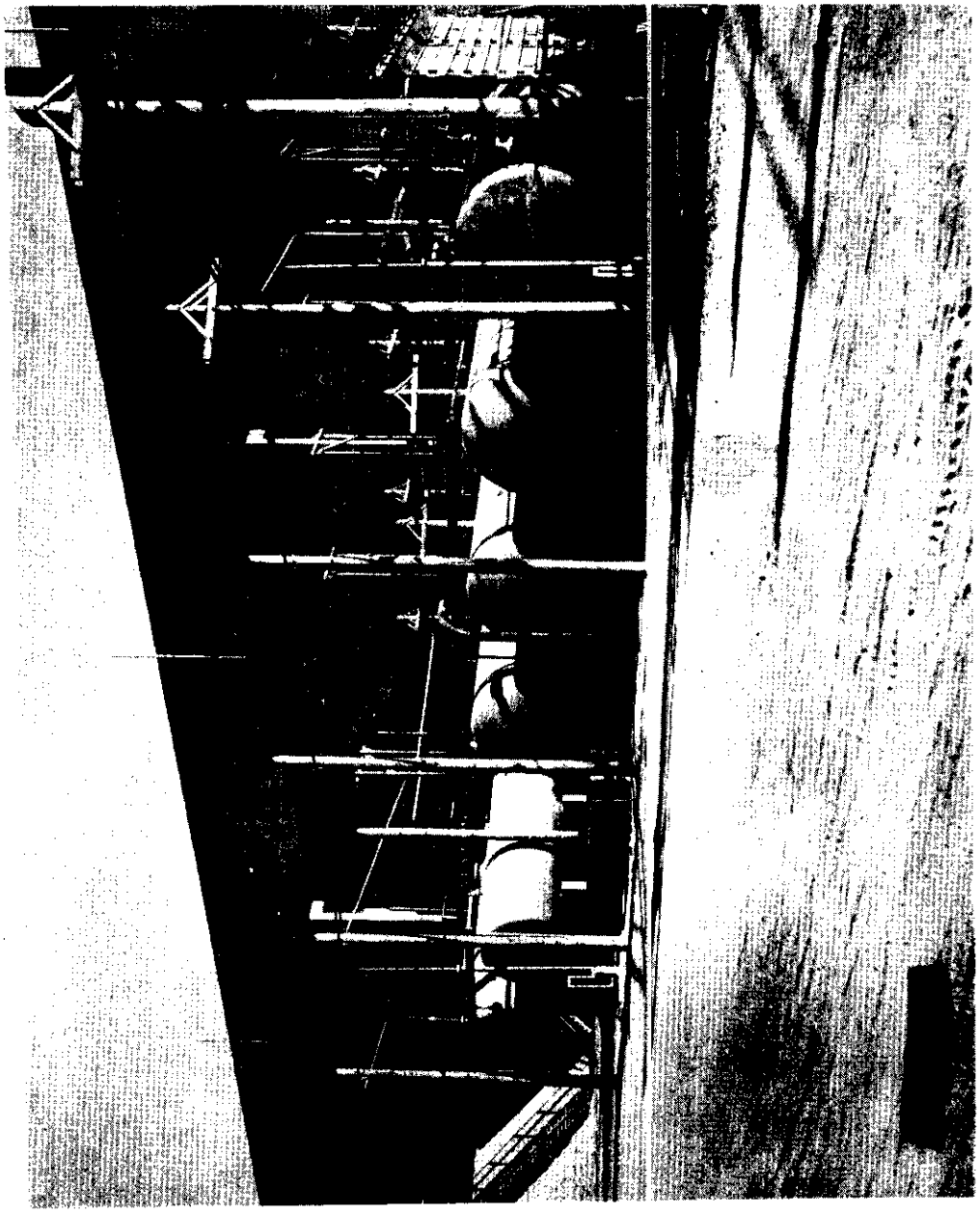
FIGURE 26



211-T TANK FARM

FIGURE 27



211-T TANK FARM

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FIGURE 28

271-T CHEMICAL PREPARATION AND SERVICE BUILDING

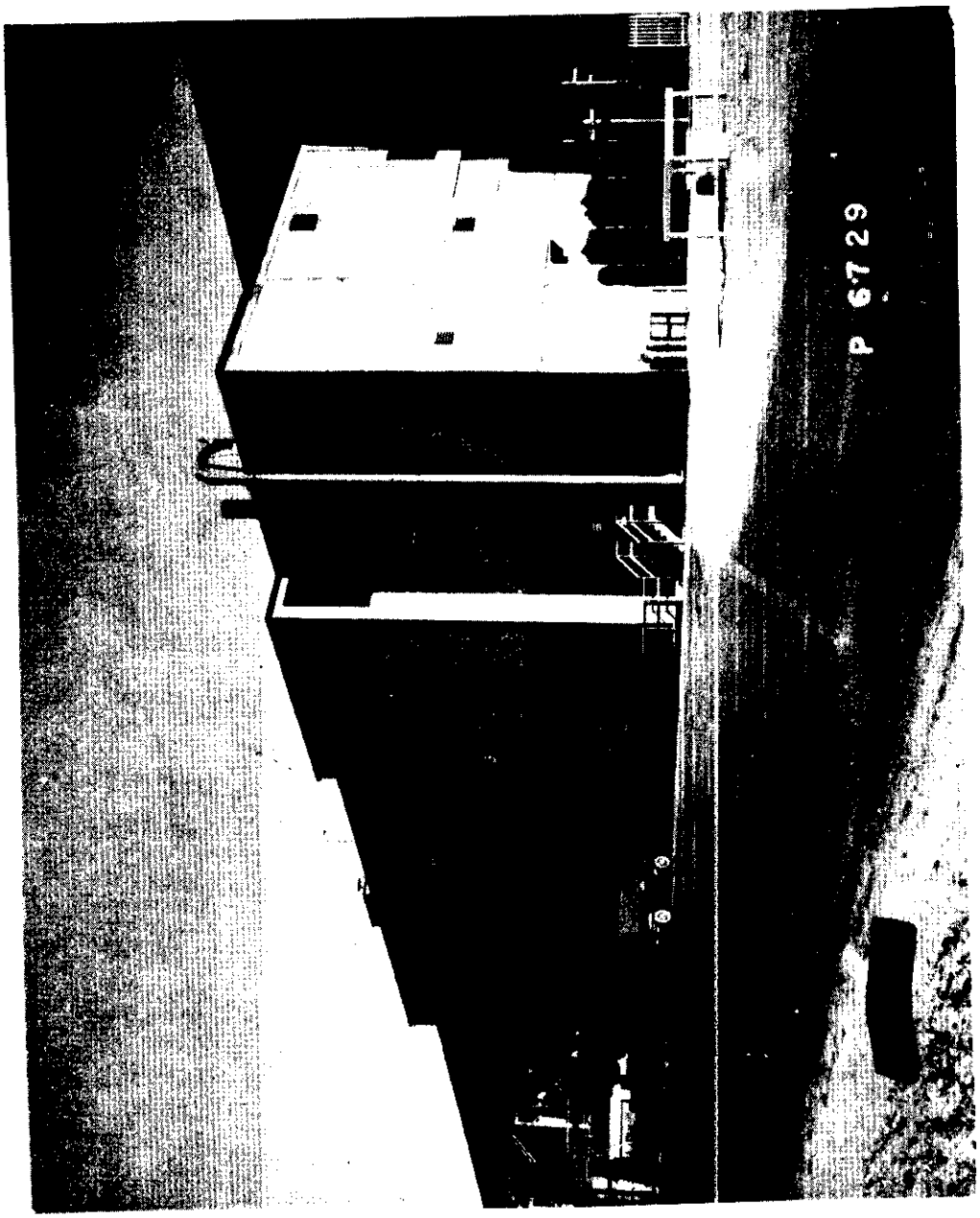
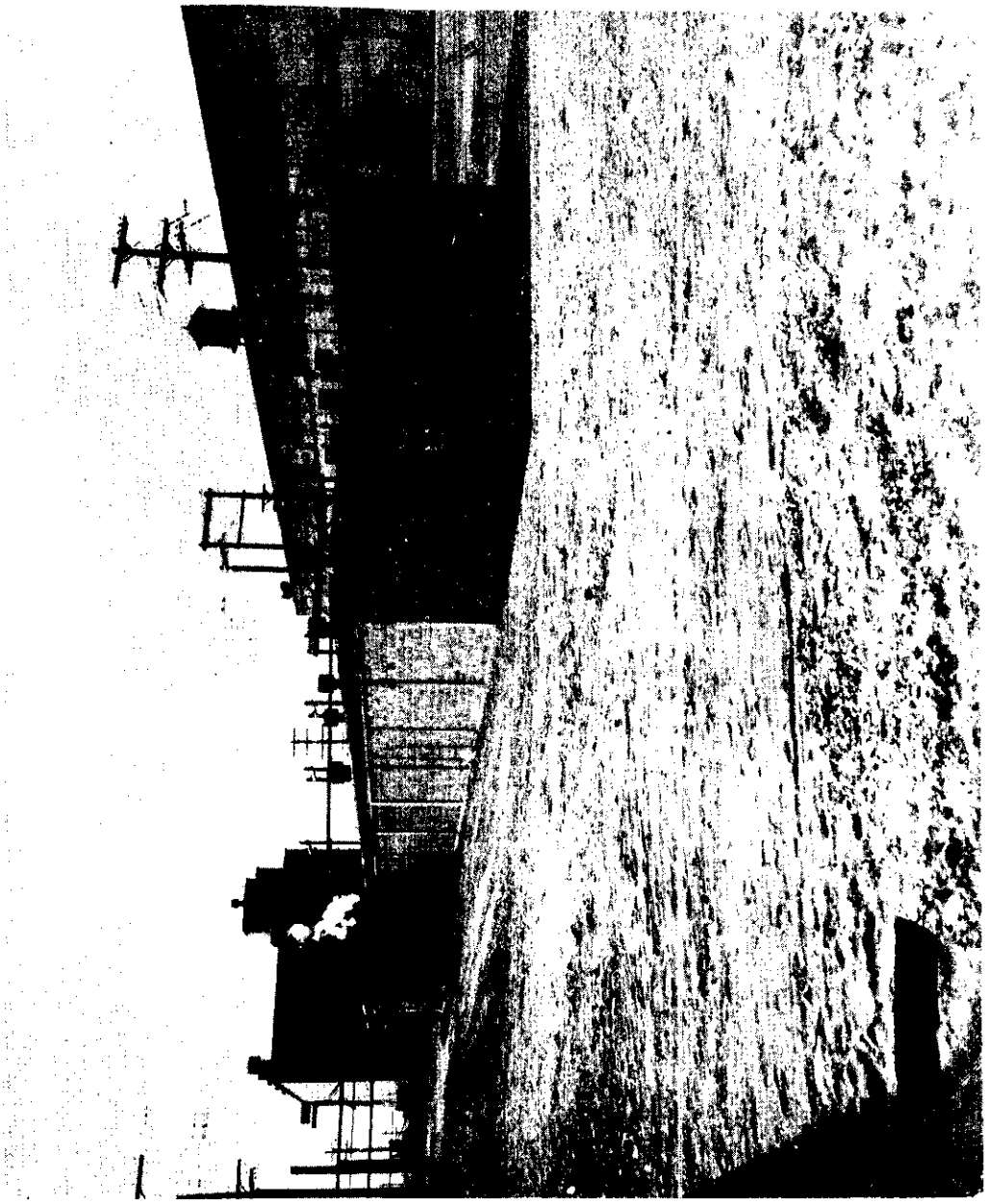


FIGURE 29



222-T CONTROL LABORATORY

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FIGURE 30

231 WEST ISOLATION BUILDING

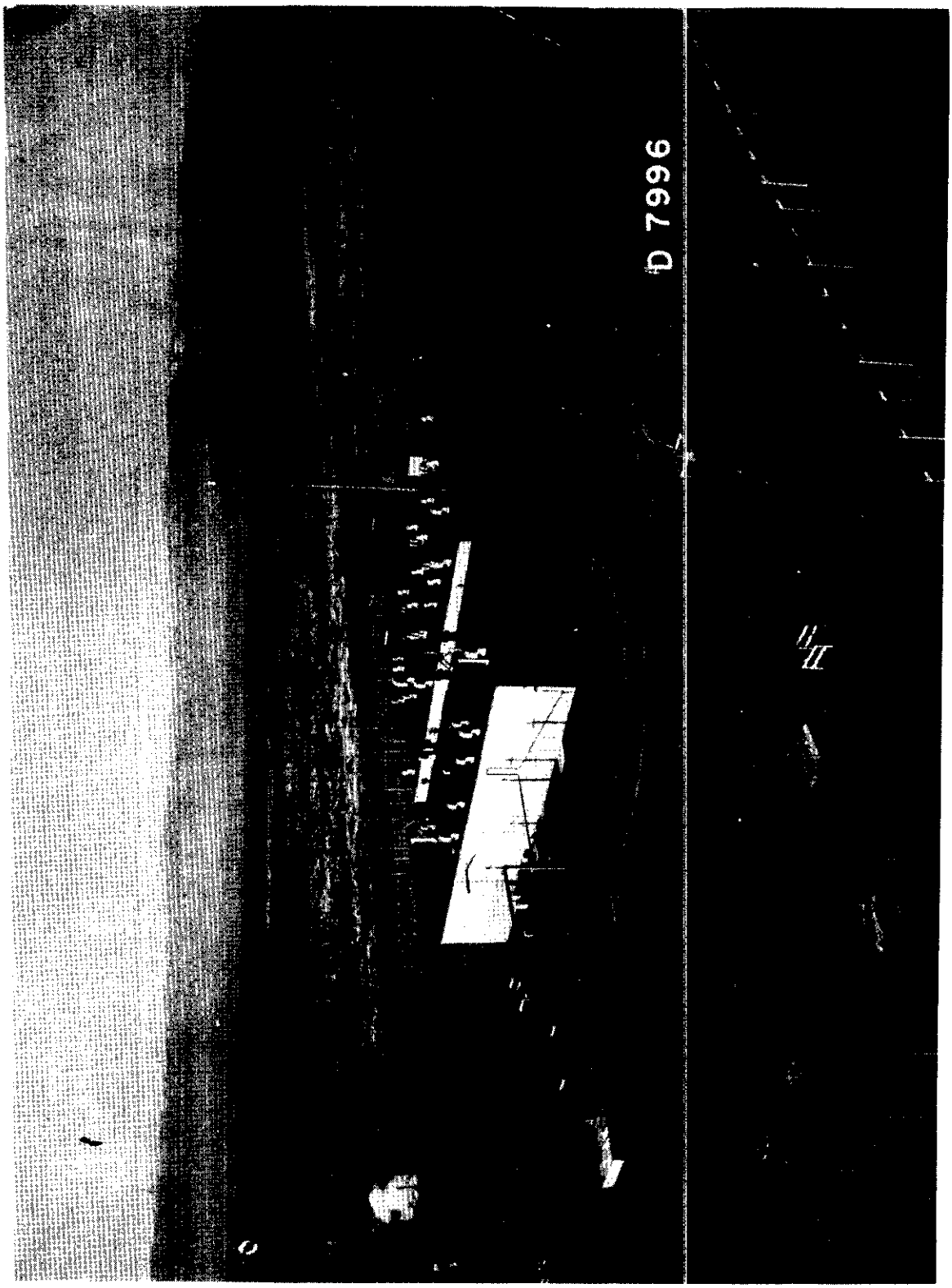

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FIGURE 3

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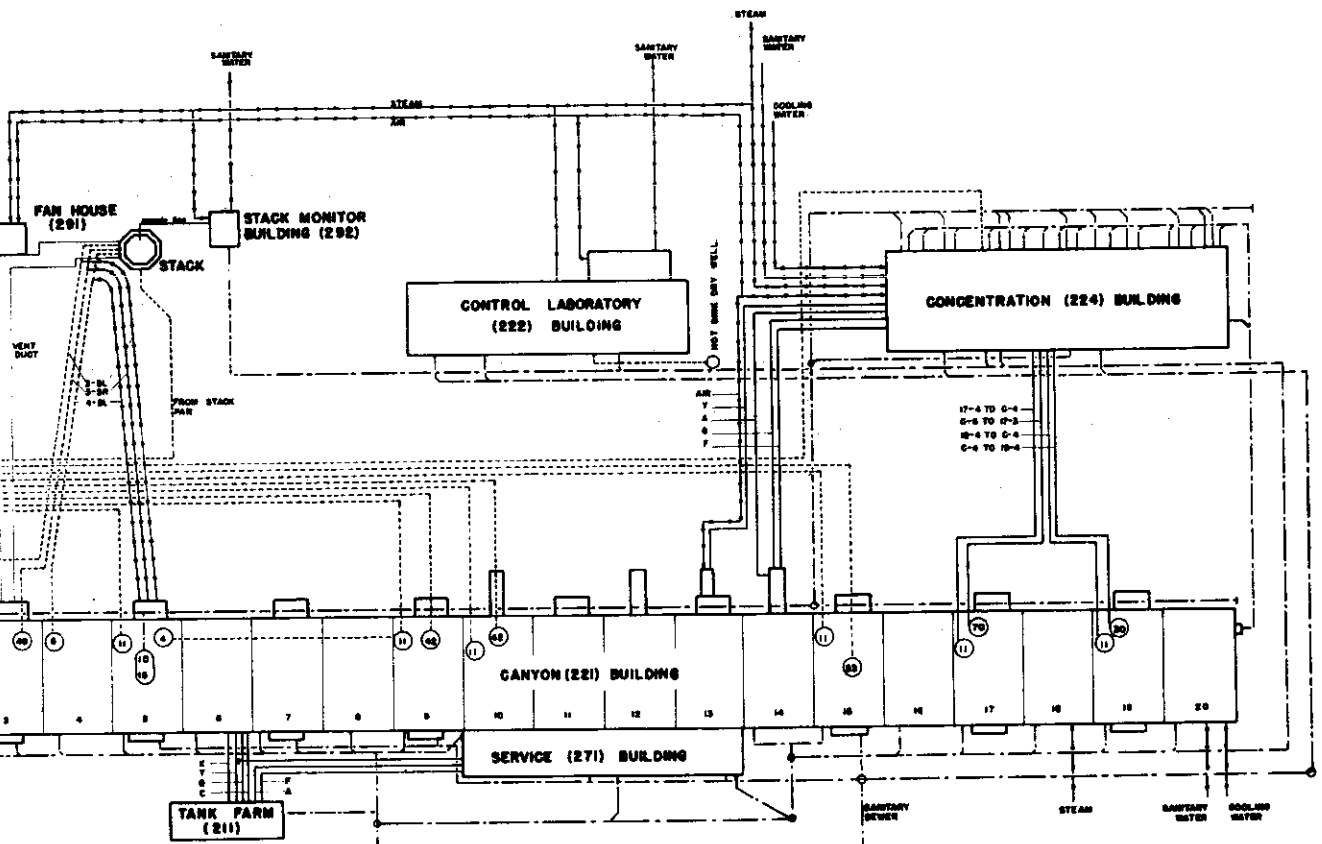
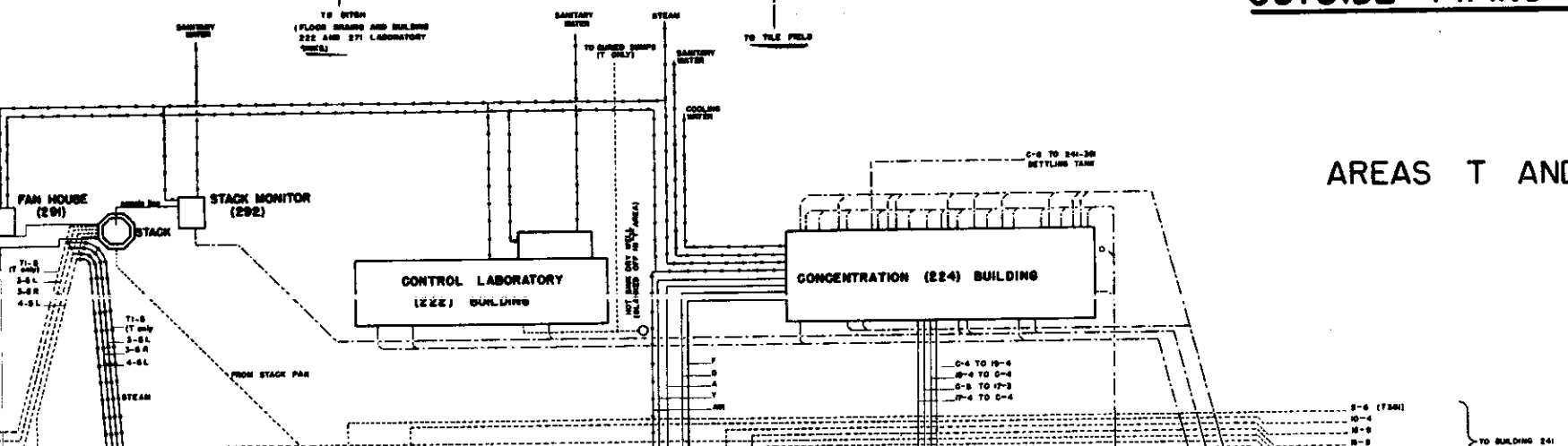


AREA B

OUTSIDE PIPING DIAGRAM



AREAS T AND U

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METAL STORAGE BASIN (212 BUILDING)

During its decay period the pile metal is stored in 212 Building (Figures 32 and 33). The metal is kept in a concrete pool under 16-1/2 to 20 feet of water. The water serves a dual purpose; absorbing the heat generated by the active metal, and shielding personnel from the radiation.

Metal is brought into the building in a water tank on a railroad car. The tank carries two casks each holding one bucket of metal (107 slugs weighing approximately 845 lbs.). The cask (Figure 34) is a massive lead container, with holes in the lid and walls so that water can circulate around the metal when the cask is immersed in the tank or transfer pits. The lid of the cask is locked into place by sliding bars actuated through a ratchet arrangement by an impact wrench.

In 212 Building, the cask lid is unlocked and the cask is then lowered into one of the transfer pits by the crane. Near the bottom of the pit, the four projecting arms of the lid are caught on steel supports. The cask is lowered free of the lid and moved forward in the pit a short distance to bring it out from under the lid. The bucket is then picked up with a yoke suspended from a small hand operated trolley and lifted out of the cask by a hydraulic lift section of the trolley track. As shown in the drawing the bucket can then be moved, under water, from this transfer pit to a transfer track section and then to the monorail system over the storage pool. The storage area has a floor over the pool with slots under the monorail tracks through which the yokes pass. During storage the buckets remain hanging in the pool, suspended from the overhead trolleys.

The building is provided with monitoring equipment to check the activity of the water in the storage pool tank car. The water to be monitored is pumped through a chamber in the apparatus. Hence, the cooling water adjacent to the various buckets in the storage basins can be monitored and buckets with fractured slug coatings giving rise to excessive activity in the water can be located. These are removed and placed in Cell 4 of 221 Building.

The transfer of metal to the Canyon (221) Building, whether because of fractured coatings or in the normal course of operation, is accomplished with the cask and tank car reversing the procedure by which metal is brought into 212 Building.

DESIGN FEATURES OF THE CANYON (221) BUILDING

The design of the separation plant was based on four essential considerations:

1) Adequate protection of operating personnel from radiation.

2) Remote operation of process equipment.

FIGURE 32

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BUILDING 212- LAG STORAGE

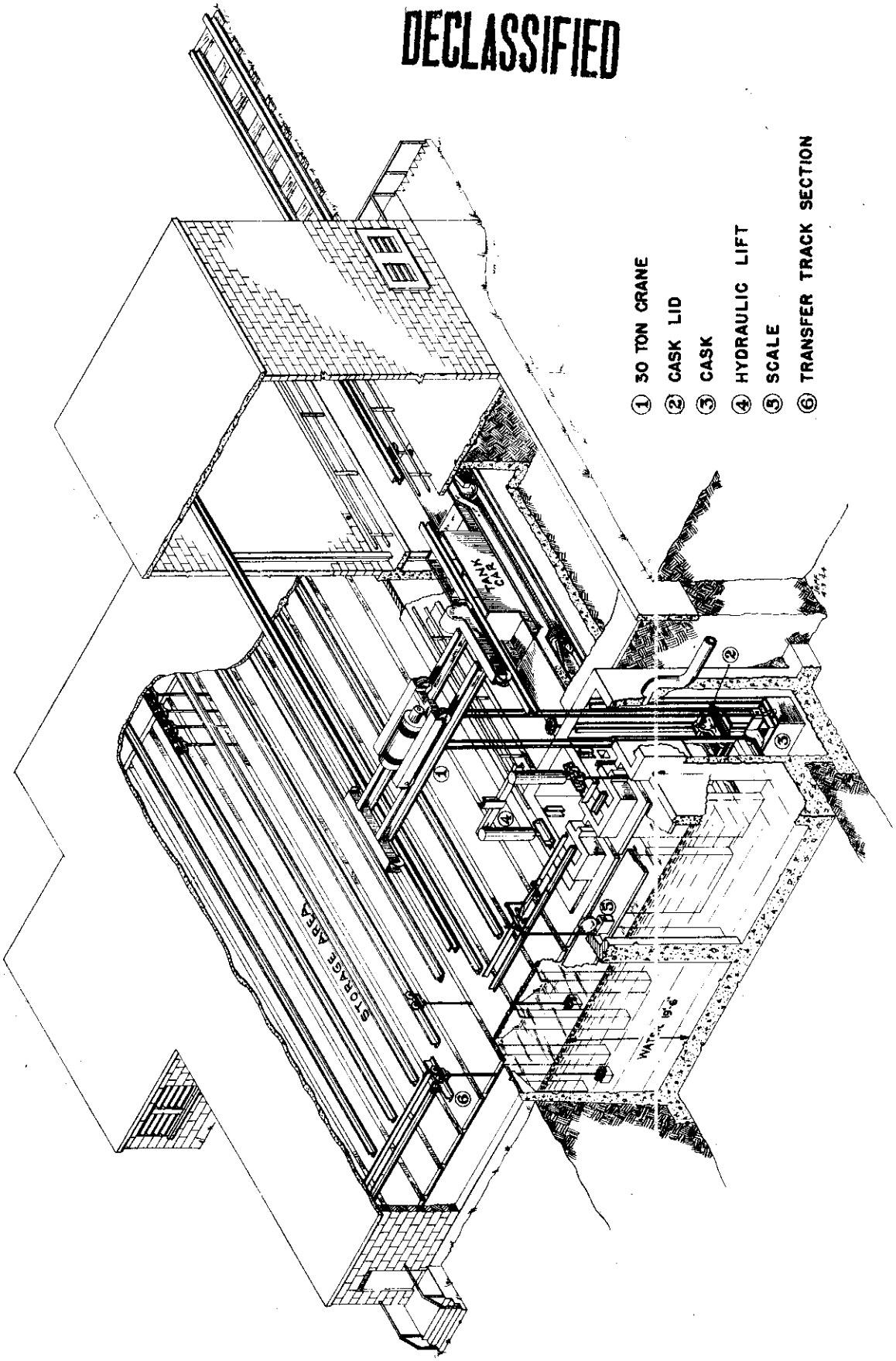

FIGURE 33

BUILDING 212-P - METAL STORAGE BASIN

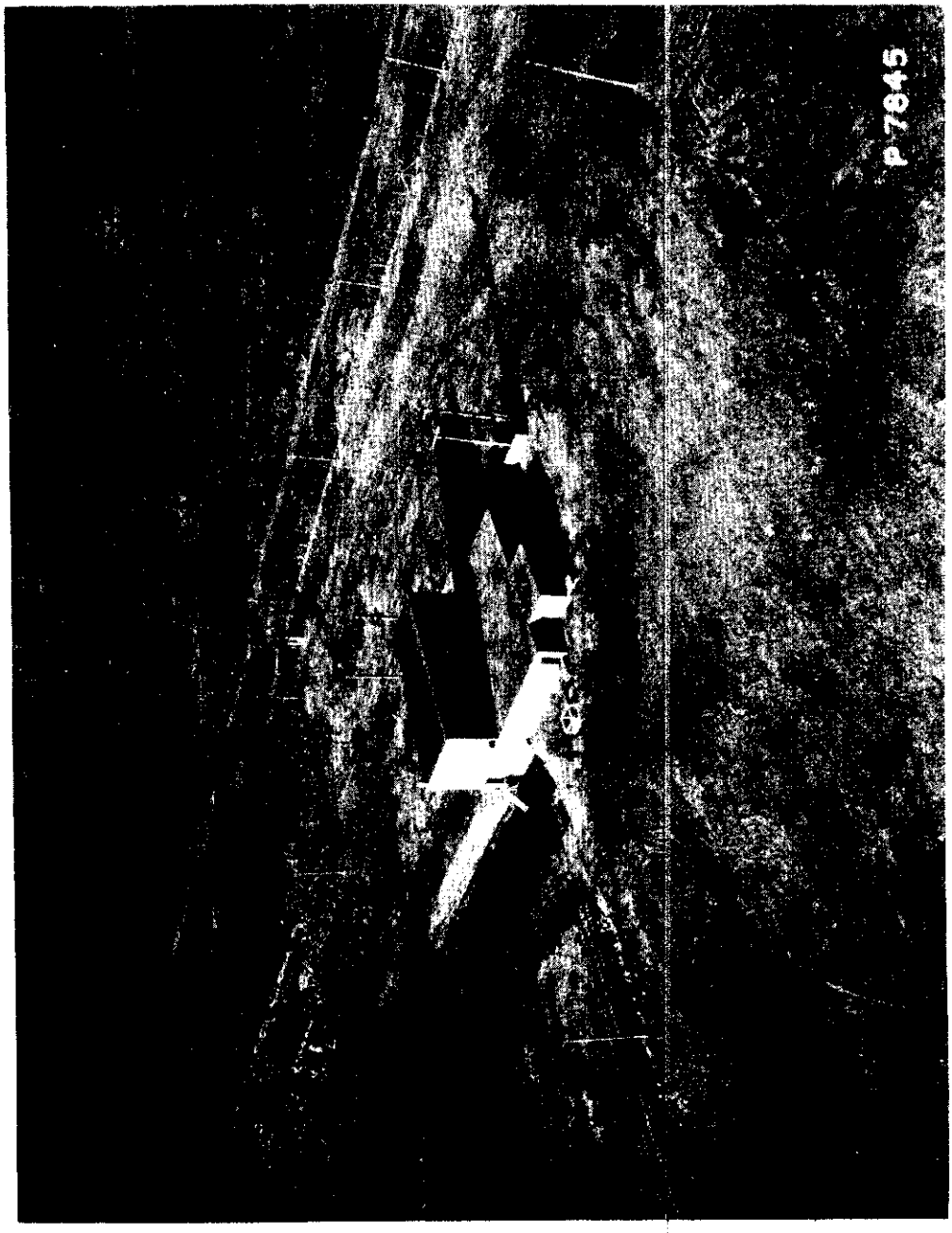

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FIGURE 34

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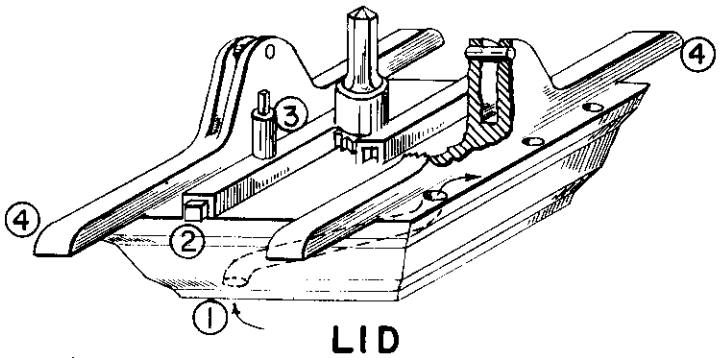
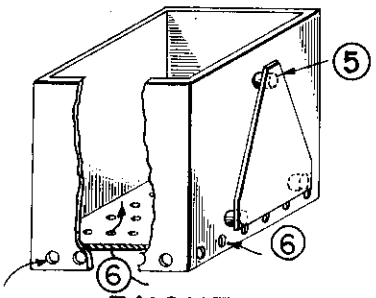
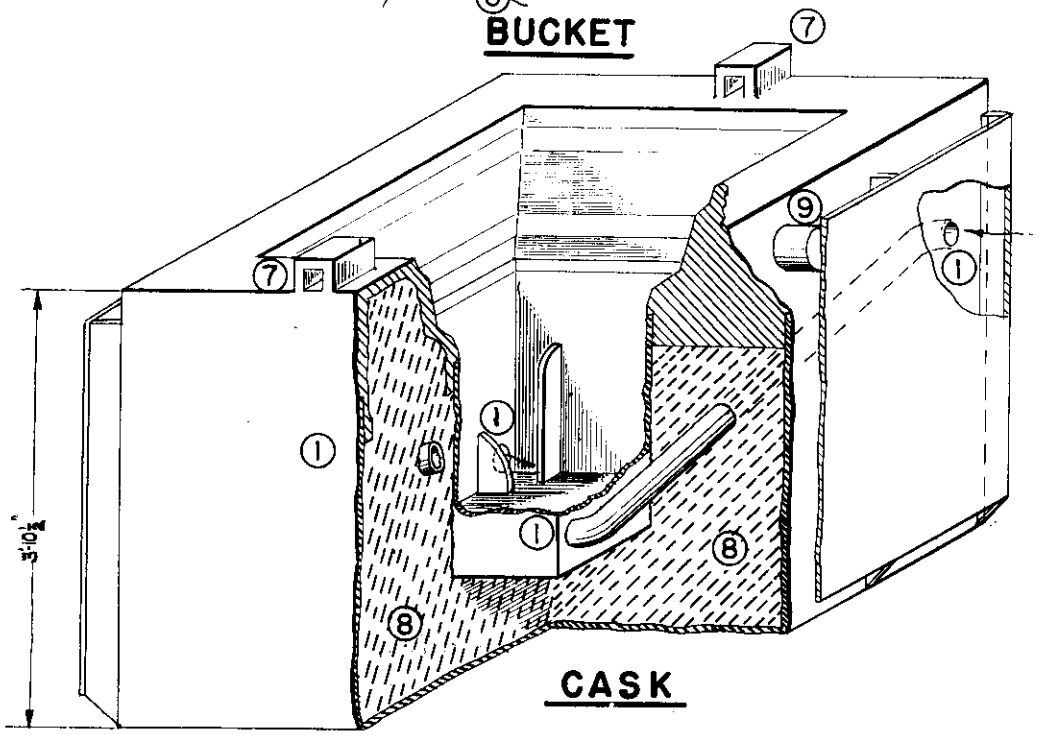
CASK ASSEMBLY



LID



BUCKET



CASK

1. WATER CIRCULATING TUBE
2. LOCKING BAR
3. BALL LOCK
4. LID ARMS
5. LUG FOR LIFTING BUCKET
6. HOLES FOR WATER CIRCULATION THROUGH BUCKET
7. CATCHES FOR LOCKING BARS
8. LEAD FILLED CASK WALLS 11" THICK
9. LUG FOR LIFTING CASK

[redacted]

3) Remote maintenance of process equipment due to the presence of high radiation levels.

4) Flexibility of arrangement and layout so that a wide range of process steps could be undertaken without major redesign or rebuilding of the plant. This last consideration was necessary because of the undeveloped state of the separation process when design was initiated on the project.

Protection of Personnel

The radiation hazards existing in the separation plant are those normally associated with radioactive materials - penetrating gamma radiation and intense, but relatively non-penetrating, beta and alpha radiation. Protection from all three sources of radiation can be obtained by a suitable combination of distance and shielding between personnel and the source of radiation. In the separation plant, shielding is obtained almost entirely by the use of massive walls of concrete which also serve as structural elements of the buildings themselves. In general, the concrete shielding is heavy enough so that protection by distance is of secondary importance. Equipment placed behind the massive concrete walls must be operated by remote control.

Remote Operation of Process Equipment

The remote operational control features of the design present no unusual aspects. Recording and indicating instruments are used to follow temperature and density changes in process equipment, while motors and other moving parts are controlled electrically in the usual manner.

Remote Maintenance of Process Equipment

Most of the unusual design features of the separation plant originate from the necessity for remote maintenance. This type of maintenance is necessary in most instances, due to the great difficulty of decontaminating a piece of process equipment which has once been contaminated by radioactive materials. Therefore, it is necessary to be able to replace and service remotely all of the process vessels and transfer lines which become contaminated. In addition, it is essential that the process equipment contain no valves, pumps, stuffing boxes or other items that require periodic inspection and maintenance, or that in the course of ordinary operation might leak or drip process solutions. This requirement is met by designing the process piping as single lines without T's or multiple connections, and designing the vessels to contain no bottom outlets. Pumps are eliminated by using steam jet ejectors for all process transfers.

The process vessels themselves were designed to be removed or installed by a specially developed crane. The operator of this crane is protected in a heavily shielded cab and views the operations through a periscope. Piping connections were designed which can be made or broken by means of a remotely controlled electrically operated impact wrench which in turn is carried on the

crane. The piping itself is made up in standard prefabricated units which can be dropped into place by the crane and then made up with the automatic connector.

Flexibility of Arrangement

An important consideration in the design of the Separation Building was flexibility of arrangement. At the time design was begun, the process itself was in many respects largely undeveloped. This required that the layout be such that not only minor changes, but even very fundamental alterations in the equipment arrangement and process flow could be made. In order to achieve this flexibility, the 221 Building was designed, as far as possible, as a group of standard units in which different types of process vessels, pipe connections, and instrument hookups could be installed without requiring structural modification. The various equipment pieces were likewise designed to permit installation at various locations in the standard units, as changing process requirements might dictate.

ELEMENTS OF THE DESIGN OF THE CANYON (221) BUILDING

The important elements of the Canyon (221) Building design are illustrated in the cut-away view shown in Figure 35.

The Connector

The most important single feature as far as remote maintenance is concerned is the connector. This is a special device by which pipes, conduits or instrument leads can be connected together by tightening a single nut. In the remotely maintained areas, removable sections of piping have a connector fitted on both ends for attachment to connector flanges on process vessels and to fixed piping built into the structure.

The design of the connector is shown in Figure 36. The piping ends in a short elbow provided with a machined and gasketed connecting surface and fixed jaws for holding the pipe in place on a connector flange until the connector is tightened. Proper rotation of the pivoted screw in the connector head moves a collar carrying three hooks, or latches, which catch the connector flange and force the connecting surfaces together. The latches pass through a yoke in the connector and are so shaped that in making a connection the yoke guides them in against the flanges, while the reverse operation drives them back and out away from the flange so that the connector can be lifted off. Directly back of the connector flange is a kick plate. In breaking a connection the last few turns of the screw bring the latches against the kick plate and force the gasketed connecting surfaces apart, thus overcoming any tendency to stick.

This connector is used in all locations that are exposed to high levels

FIGURE 35

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BUILDING 221 - CUTAWAY VIEW

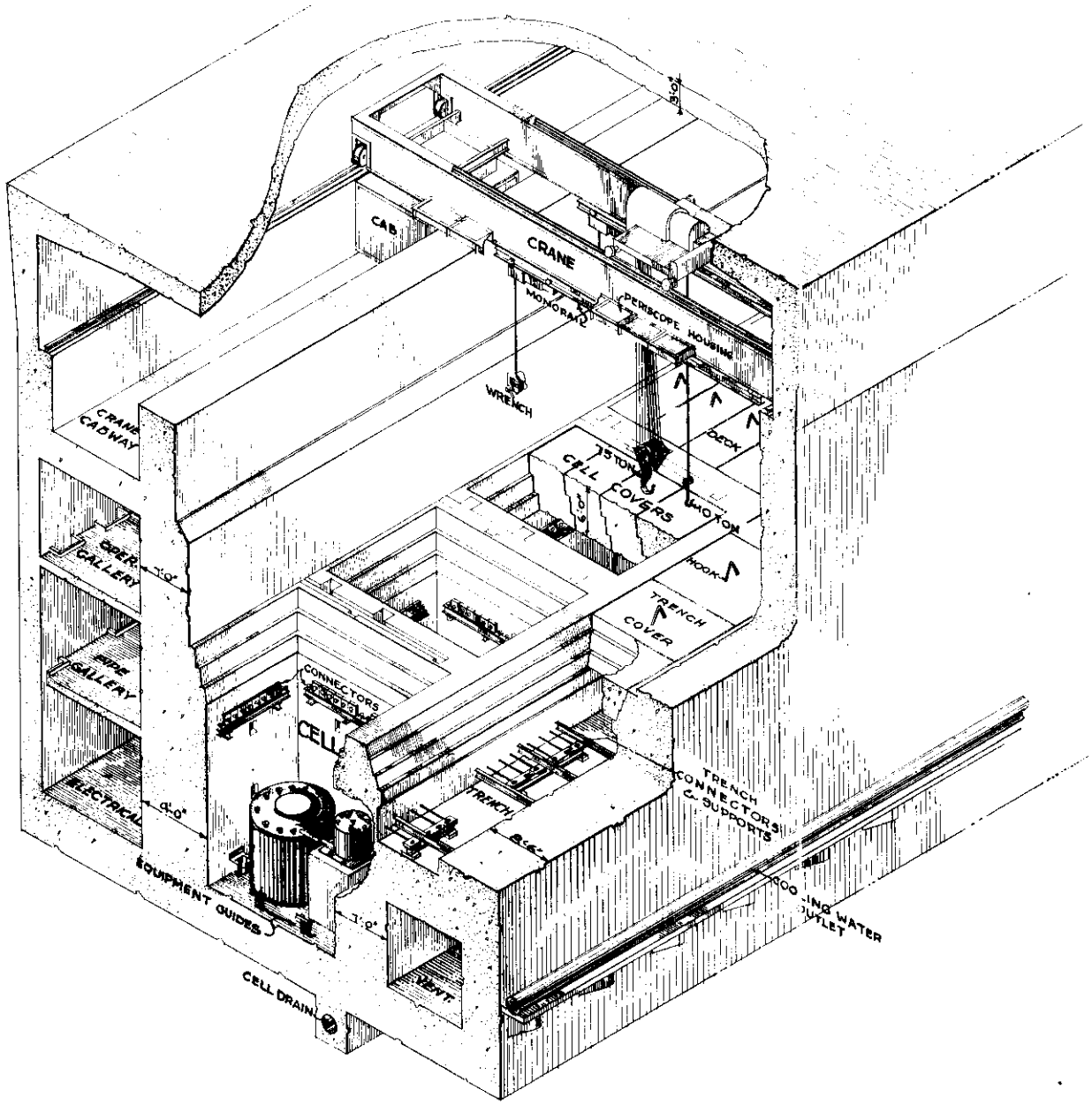

FIGURE 36

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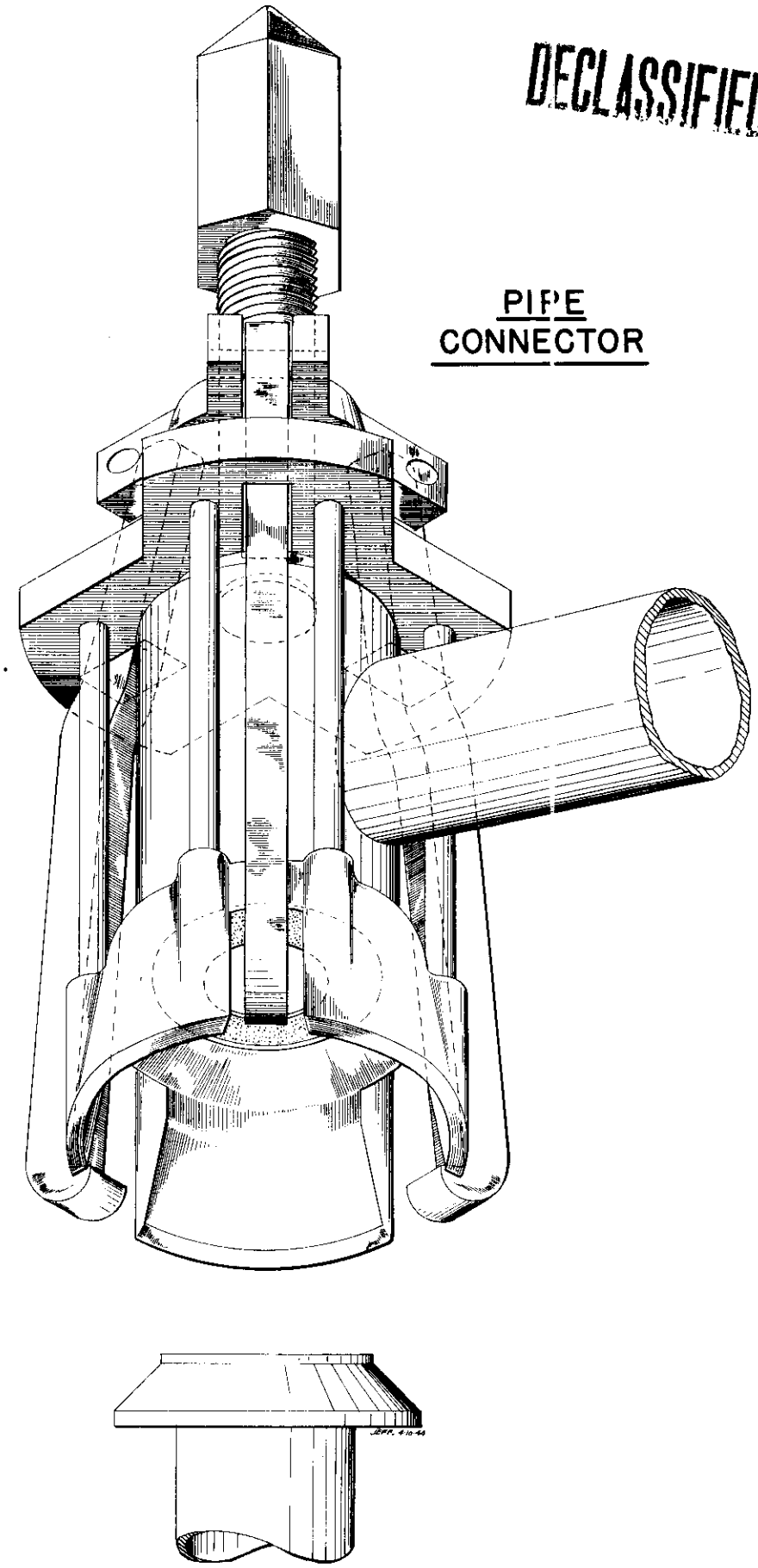

of radiation from process solutions. Two variations of the fundamental designs are used:

1) A pipe connector essentially as illustrated above which serves to connect two pieces of process piping, and

2) An electrical connector in which the piping leads are replaced by electrical conduits and the electrical circuit is continued through suitable spring loaded contacts made up in the body of the connector.

The pipe connector carries single 2-inch or 3-inch pipes, or four smaller pipes of 1/8 to 1/2 inch in diameter. The electrical connector which may carry up to six separate cables with a corresponding number of contacts serves not only to carry current for motors, but to make electrical contacts for instruments such as resistance thermometers, microphones, and motor tachometers.

Impact Wrench

The connectors are operated by means of a specially constructed electrically driven impact wrench carried on the crane. This wrench can be lowered and placed on the actuating nut of the connector and turns it in a direction either to tighten or loosen the connector as desired. The impact feature of the wrench enables a stubbornly turning fitting to be pounded loose in much the same way as a sledge hammer would act on a persuader.

The combination of connector and impact wrench enables flange connections to be made up and broken with the use of the overhead crane. The connectors are welded on to prefabricated piping, the whole assembly being so designed that it can be lowered by means of the crane on to the process vessel and make the desired connection, either to another vessel, or to process and instrument lines installed in the walls of the building.

The motor speed of the originally designed impact wrench was 1560 rev./min. Initial use of this wrench indicated control of the tightening of the connectors to be very difficult. In many instances it was found difficult to avoid complete rupture of the gaskets used in the connectors. For this reason the motors driving the impact wrench were rewound to give a motor speed of 560 rev./min. resulting in 300 to 320 impacts/minute. Using this slower speed impact wrench an impacting period of 20 seconds was found to be satisfactory for tightening the connectors unless they were improperly aligned.

Crane

A 75-ton electrically operated overhead railway crane is provided in each Canyon (221) Building (See Figure 35). This crane is provided with a heavily shielded cab which in turn operates behind a 5-foot thick wall; thus, providing adequate shielding against radiation. The crane cab is

provided with two adjustable periscopes and television equipment to permit viewing of the canyon deck without exposure to radiation. (These instruments are described in more detail in Chapter X) Together with the usual hook for handling equipment this crane also carries two impact wrenches. All remote maintenance work in the canyon is carried out using this crane.

A second overhead 10-ton capacity electrically operated railway crane is provided for use in maintenance work where radioactive radiation is not present.

Gaskets for Connectors

Original plans called for the use of gaskets for the connector joints to be made from the polymer, poly-tetrafluorethylene - sometimes known as "Teflon" or G-X. This polymer possesses excellent resistance to all of the chemicals and lubricants employed in the separation process. However, it was found that gaskets made from the presently available types of this material flowed excessively when used in connectors tightened by the impact wrench, although it functioned very satisfactorily in the hand tightened flanges used in the Concentration (224) Building. For this reason, gaskets for the connectors on all lines in the Canyon (221) Building are made from about 1/16 inch thick compressed sheeting which contains 75-80% long blue african asbestos fiber, about 13% Buna S synthetic rubber binder, and the remainder fillers such as carbon black, barites, china clay, or zinc oxide. This gasket material is generally designated as G-9. Tests have indicated that the G-9 gaskets may require occasional replacement when in contact with high concentration nitric acid solutions. The G-9 type gaskets are coated with flake graphite before installation in order to prevent their adhesion to the flanges when disconnecting the connectors.

Transfer Jets

The transfer jets used for transferring all process solutions in the Canyon (221), Concentration (224), and Isolation (231) Buildings are illustrated in Figure 37. Four different sizes of steam jets are used, all of which were built from 25-12 stainless steel, and are designated by their rated water syphoning capacity of 3, 10, 20 and 75 gallons/minute. Figure 38 gives the results of actual performance tests using the 10, 20 and 75 gallon/minute jets at various steam pressures for the transfer of water. Figure 39 gives the calculated values for dilution of the solution being transferred by the same jets, based on the data summarized in Figure 38, and other performance tests.

Gang Valves

The steam and venting compressed air supply to the transfer jets, and also the steam and compressed air supply to process vessel spargers in both the Canyon (221) and Concentration (224) Buildings is controlled by means of gang valves illustrated diagrammatically in Figure 40.

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FIGURE 37

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STEAM JET

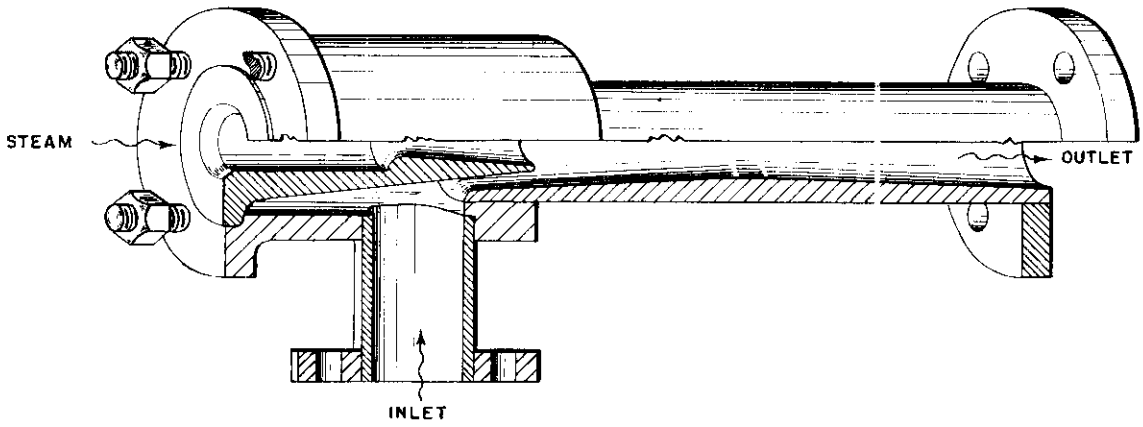

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FIGURE 38

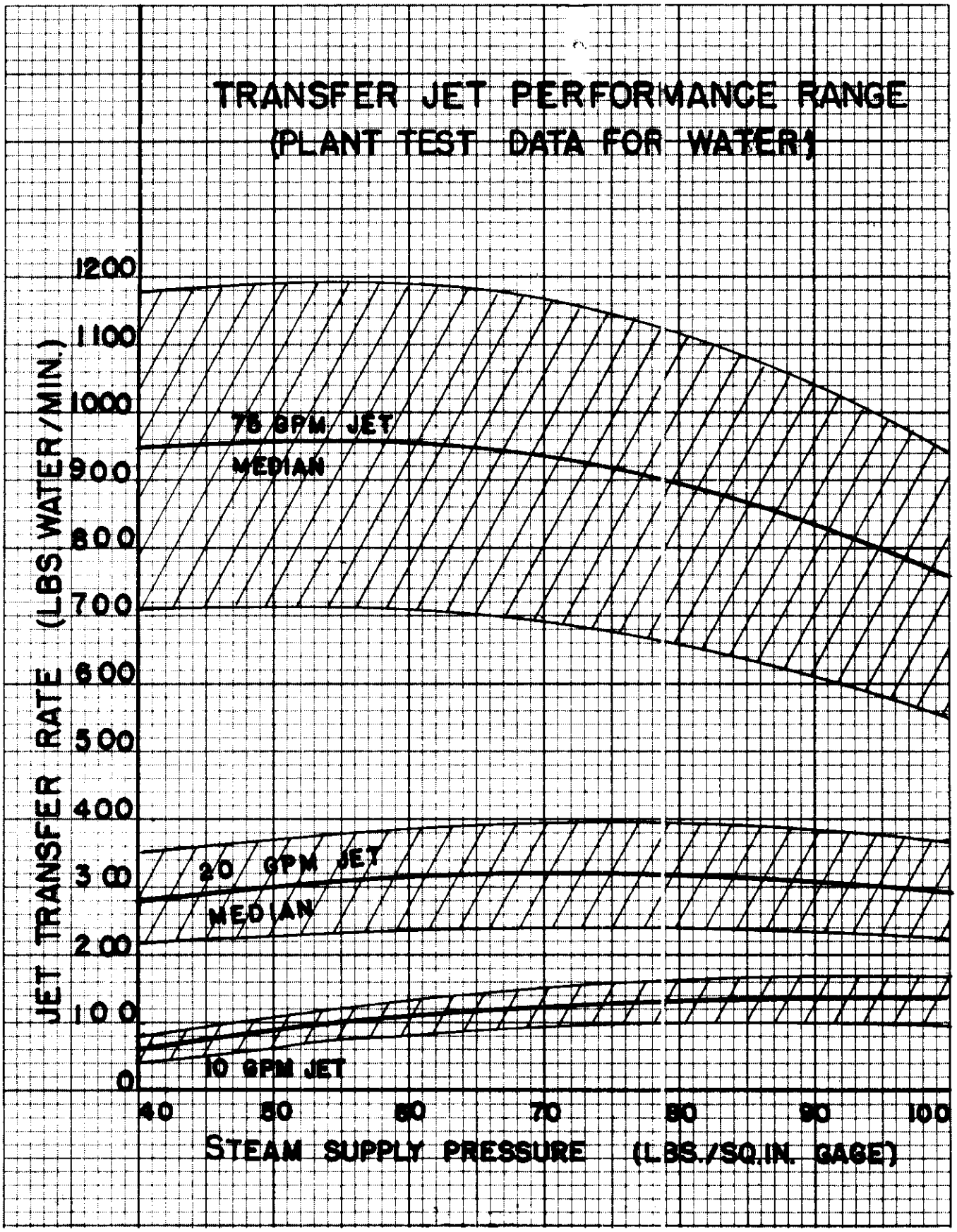

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FIGURE 39

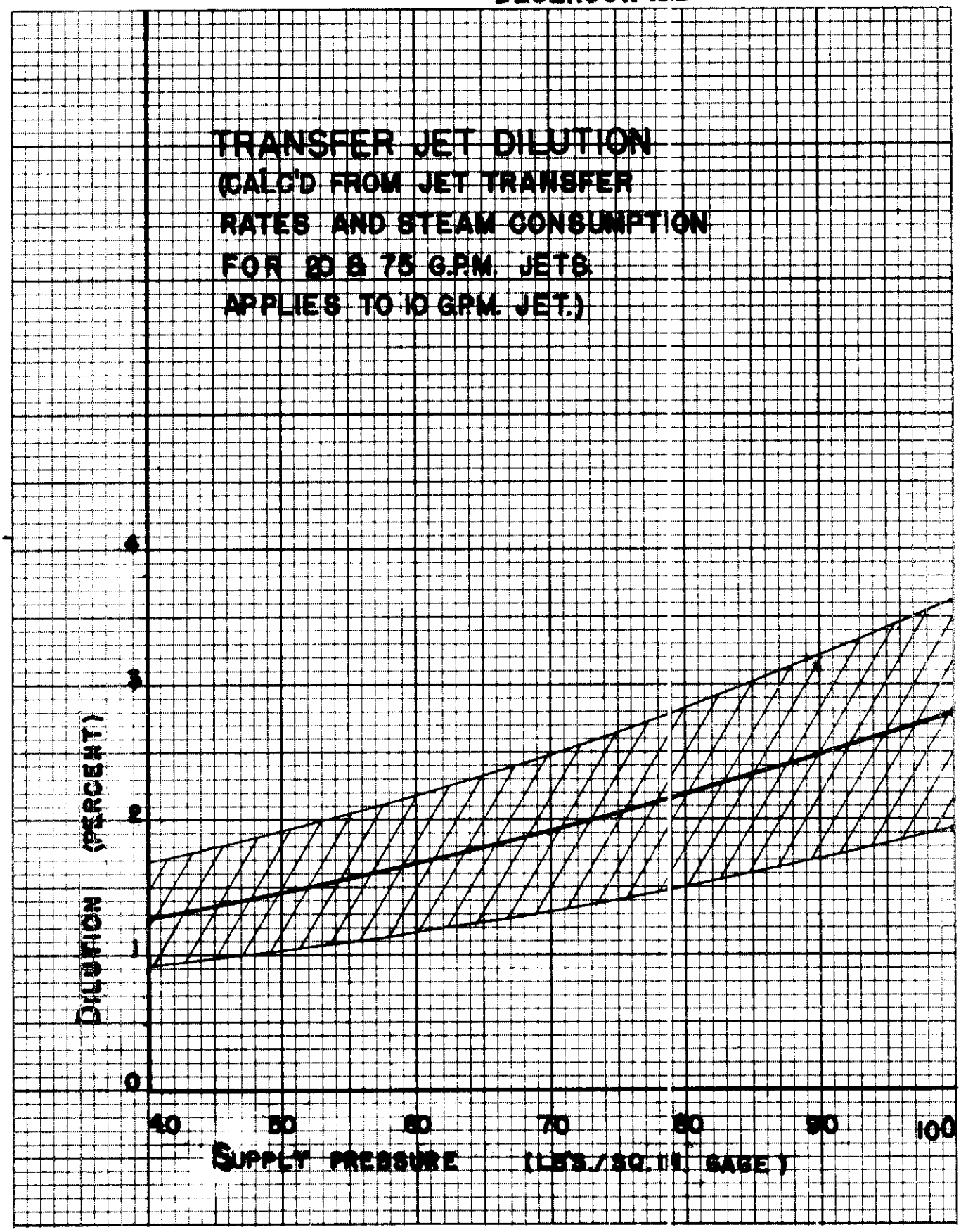

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FIGURE 40

OPERATING DIAGRAM OF GANG VALVE

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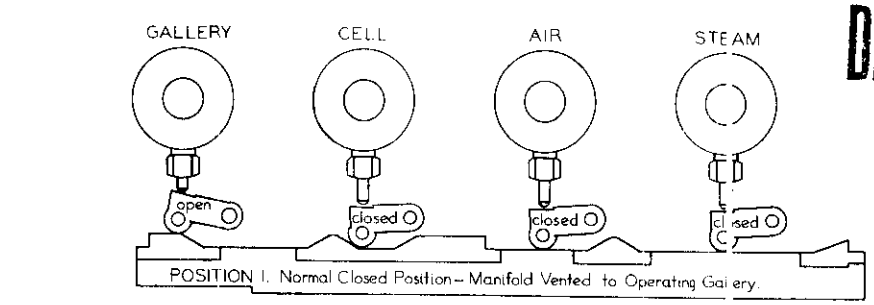

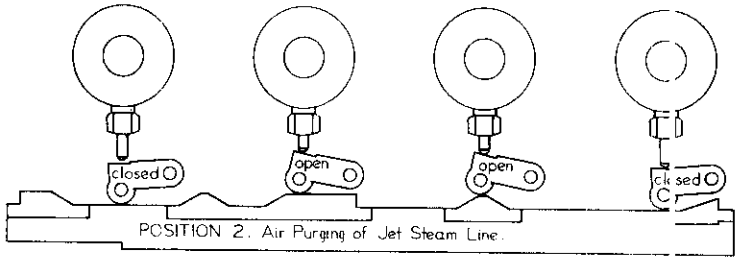

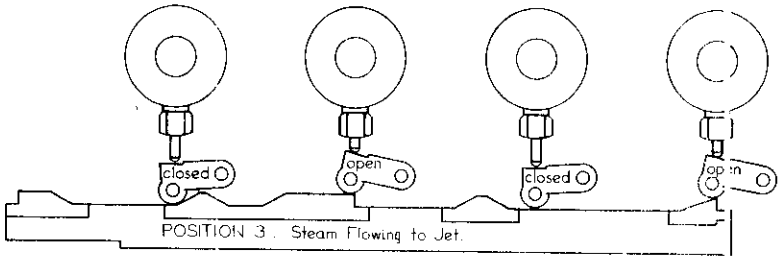

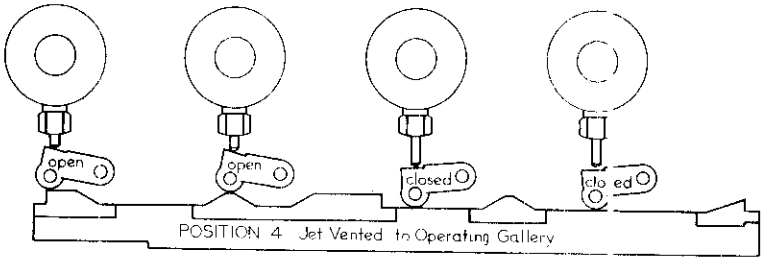

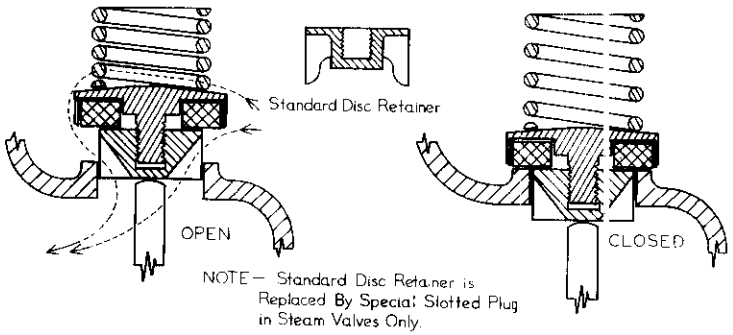

CROSS SECTION OF STEAM VALVE.

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The individual valves making up the gang valves are of the spring closed globe type with all working parts being constructed from bronze. On the valves supplying steam to the transfer jets from the precipitators to the centrifuges the guide on the standard disc is replaced by a plug containing a milled slot as illustrated in Figure 40. The purpose of this slot is to permit more accurate control of the steam supply to these jets with resultant more uniform feeding of the process solutions to the centrifuges.

As indicated in Figure 40 the gang valves permit the following operations to be carried out dependent upon the setting of the valve:

Position 1 — Normal Closed Position

Valve to operating gallery closed, valve to jet is open, steam and air valves closed.

Position 2 — Air Venting of Jet or Sparger

Gallery valve closed, jet and air valve open, steam valve closed.

Position 3 — Steam Being Supplied to Jet or Sparger

Gallery valve closed, jet and steam valves open, air valve closed.

Position 4 — Jet Vented to Operating Gallery-Maintenance Position

Gallery and jet valves open, steam and air valves closed.

Basic Process Vessel Requirements

For the most part, the bismuth phosphate process in the Canyon (221) Building can be carried out in a series of identical equipment units which are well adapted to the feature of flexibility arrived at in design and to the standardization of arrangement, which is almost essential for successful remote maintenance.

Standard Section

The Canyon (221) Building consists of a row of twenty approximately 40-feet long concrete sections each containing two cells illustrated by Figure 42 as indicated in the process piping diagram, Figure 41.

Twelve of these concrete sections each contain a standard group of process equipment consisting of four pieces; precipitator, catch tank, centrifuge, and solution tank illustrated in Figure 43.

Most of the Canyon (221) Building operations, the extraction and decontamination cycles, can be carried out in this standard grouping. For example,

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FIGURE 41

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CANYON BUILDING PROCESS PIPING DIAGRAM

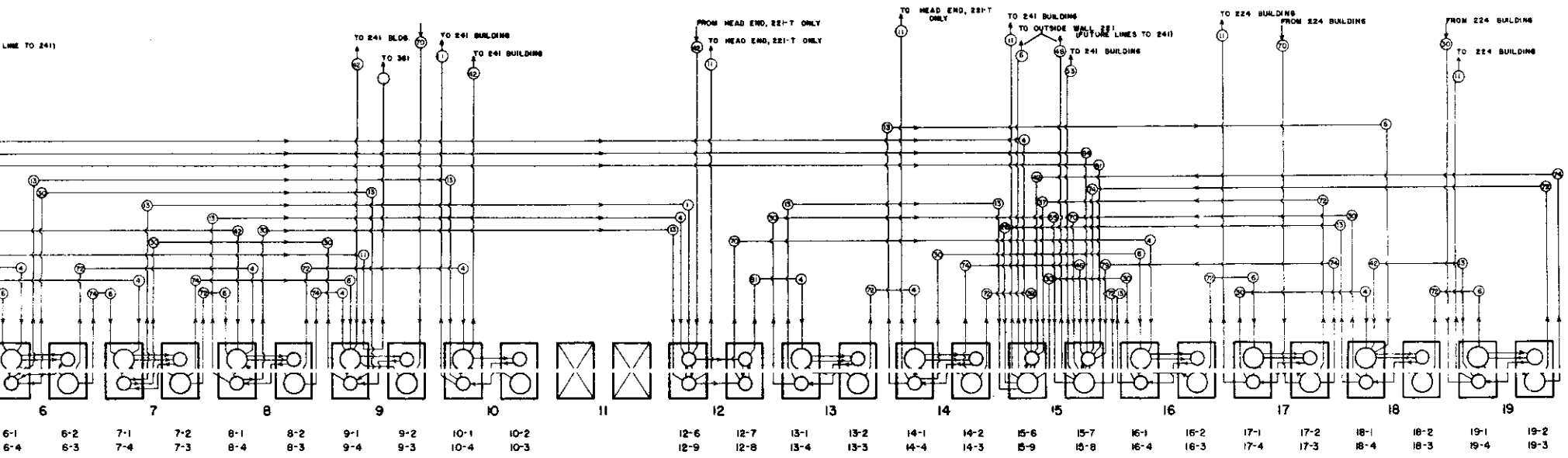

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FIGURE 42

CANYON BUILDING

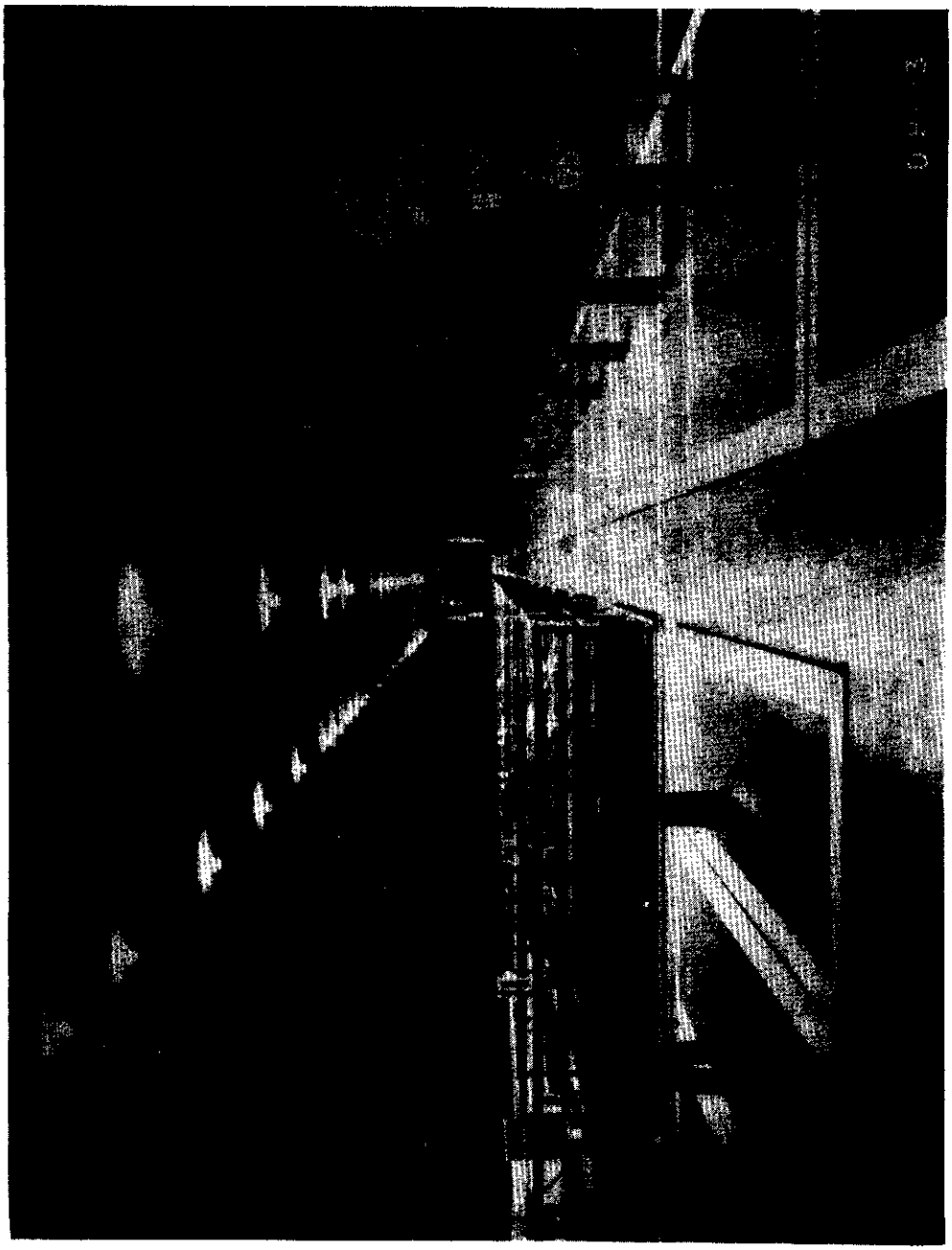

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FIGURE 43

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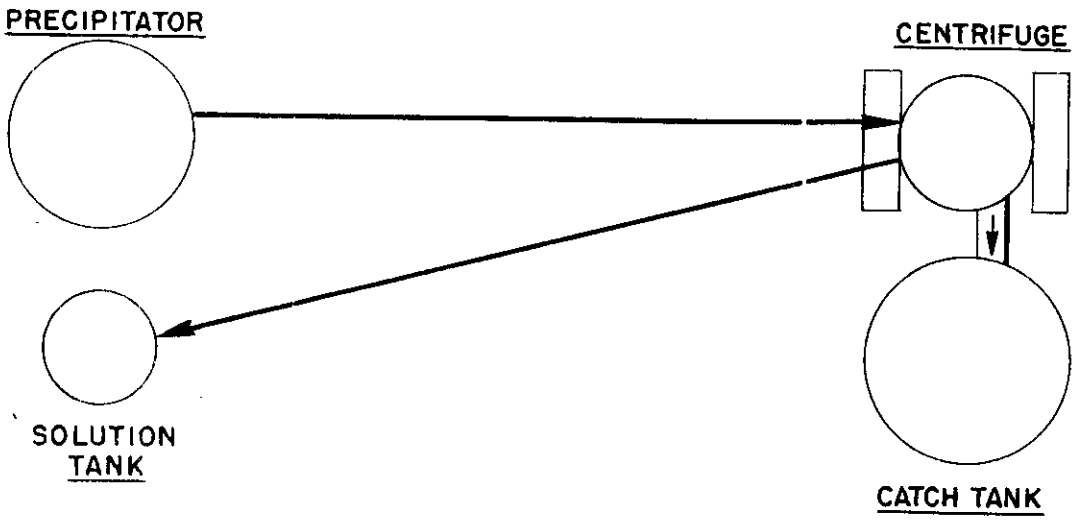
STANDARD EQUIPMENT GROUP



[redacted]

in an oxidation and by-product precipitation, the product is oxidized and the by-product precipitate formed in the precipitator. The cake is separated out in the centrifuge and sent to waste via the solution tank, while the product solution is received in the catch tank and sent on to the next operation.

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Standard Cell

The standard cell is a 13 foot by 17 foot 8 inch room. This room is 22 feet high, with 7-foot thick concrete walls, and has a 6-foot thick cover. The cover is in removable sections and is the only means of access to the cell. The massive walls and cover shield personnel against radiation from process materials within the cell. The cover sections have stepped interlocking edges so that there are no straight cracks through which radiation can pass.

All pipe, instrument, sampling and control lines into the cell are buried in the concrete and terminate in connector flanges on the cell walls. These flanges are installed with a high degree of precision, and the cell walls and floor are finished accurately to standard dimensions so that the connector arrangement in the cells is fixed and uniform. The connector arrangement in a standard section of two cells and the lines through the concrete are shown in Figures 44 and 45. Each of the electrical lines contain six leads. The other instrument, hydraulic, and lubrication lines contain four small pipes, and the chemical feed, steam and water lines consist of a single 2-inch or 3-inch pipe. The section in Figure 45 shows how piping to the gallery is brought up in an S shape rather than straight through the concrete in order to minimize the escape of radiation from the cell.

As shown in Figure 35, equipment is placed on the cell floor and held in position by guides built into the cell, thus establishing a standard relationship between the connector flanges on vessels and cell walls. It is this standard relationship which makes remote maintenance possible, since piping can be prefabricated to fit.

The equipment arrangement together with the services supplied by the various connectors for a standard section consisting of two standard cells is illustrated in Figures 46, 47, and 48. Operating flexibility is provided for by the spare connector flanges on the cell walls which can be used to supply additional chemical feeds, instrumentation, process transfers to or from the pipe trench, and the like. Process transfer lines between cells in the section are run directly through the cell walls. Because of difficulties created by the expansion joint which separates adjacent sections, no piping is run through the walls between sections.

Details of piping and equipment arrangement within each process cell is indicated in the chapters describing the individual steps in the separation process (Chapter III - IX).

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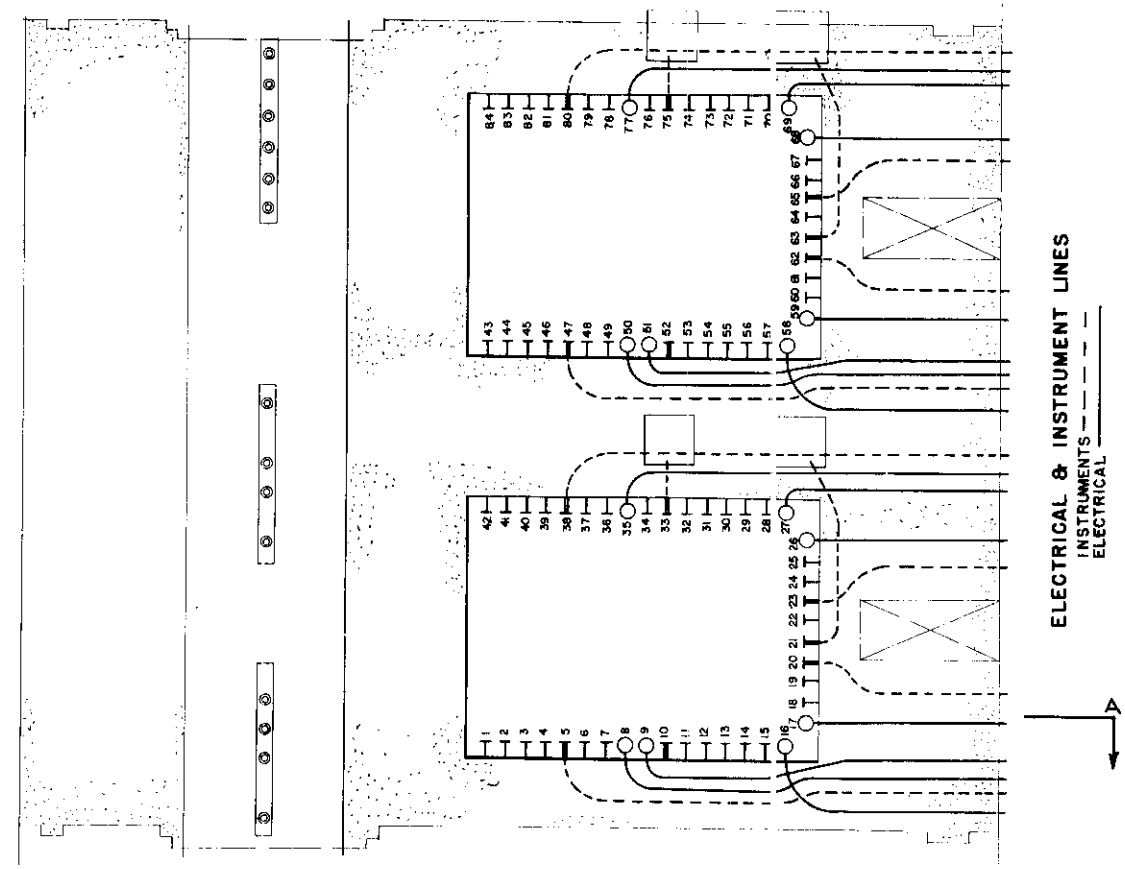
FIGURE 44

CELL SECTIONS



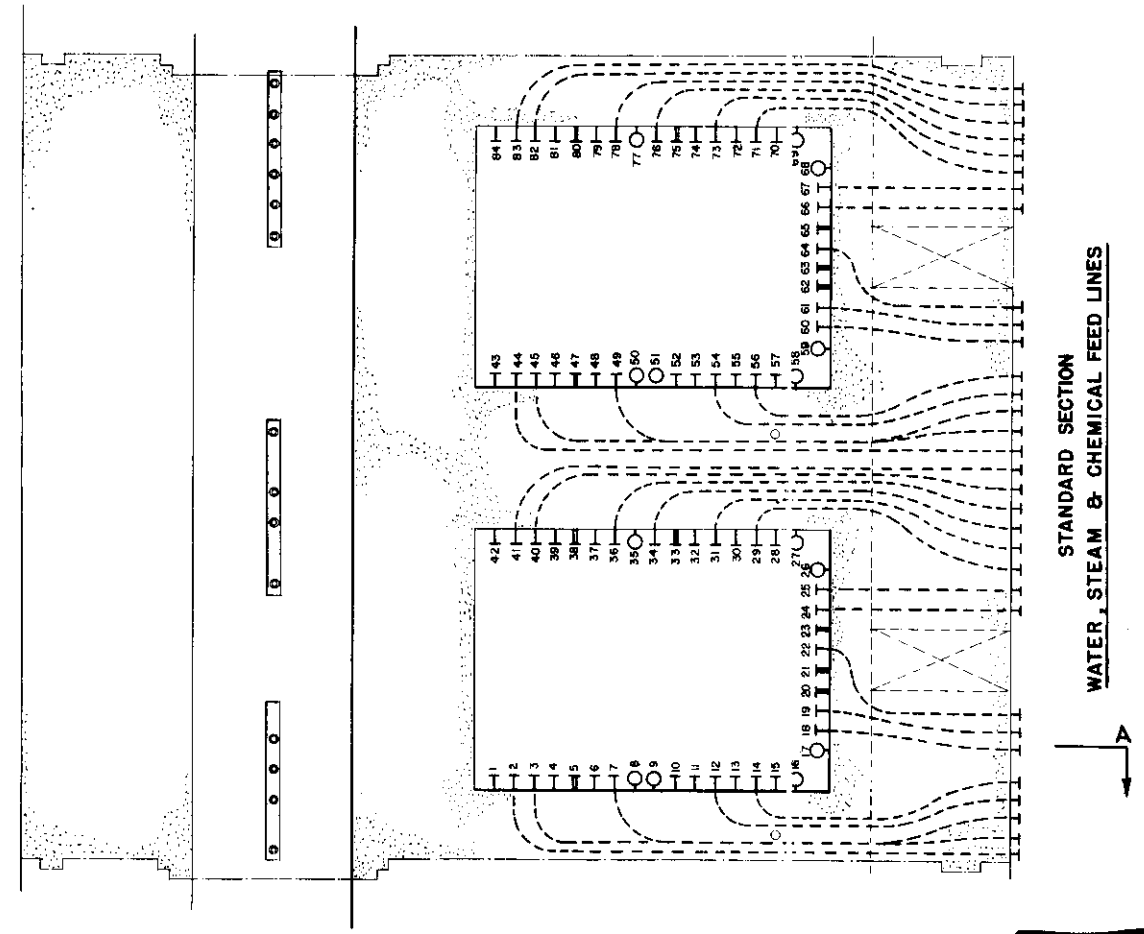


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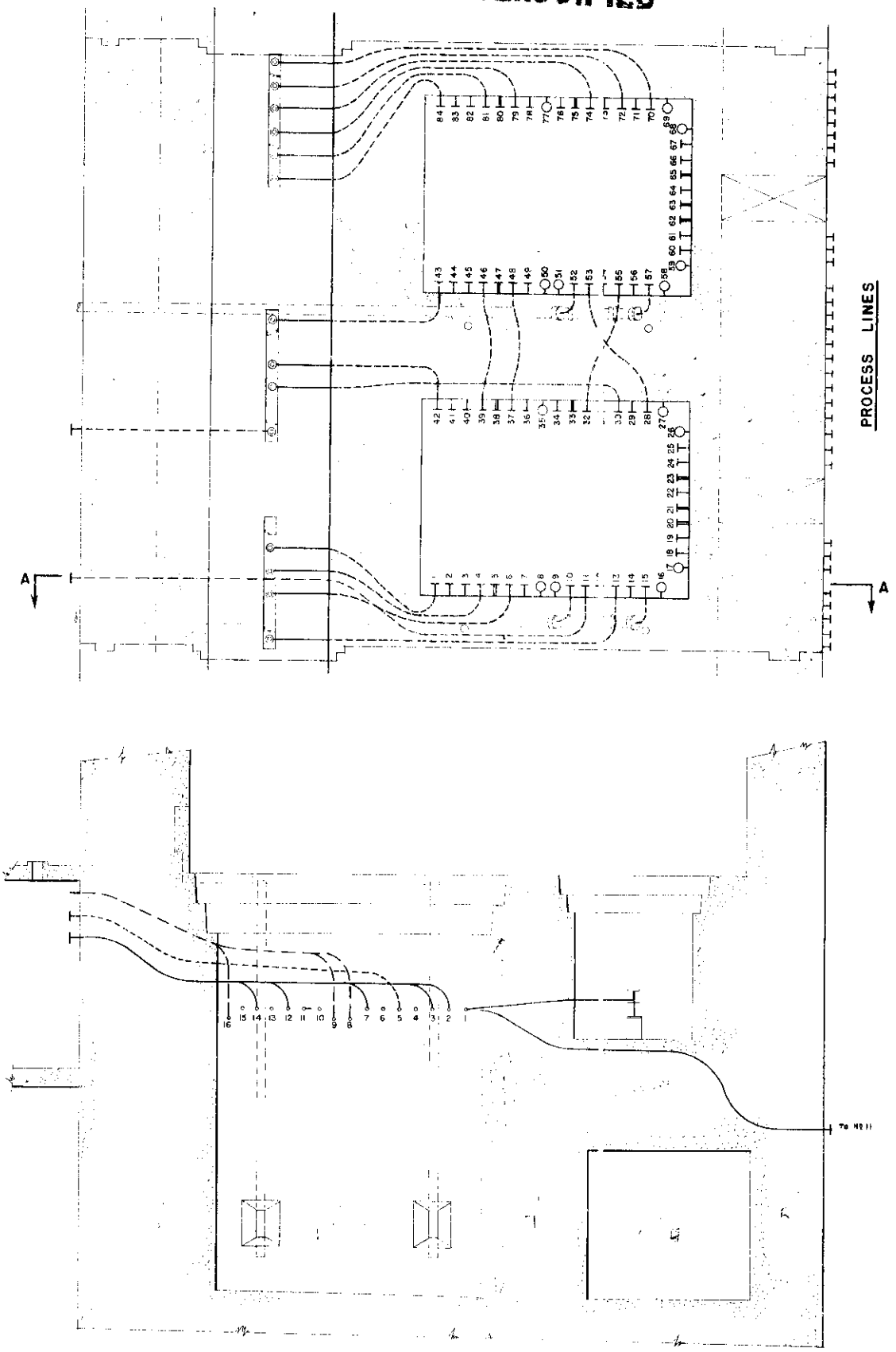
FIGURE 45

CELL SECTIONS



SECTION A-A

ELECTRICAL
INSTRUMENTS
STEAM, CHEMICAL

[redacted]

FIGURE 46

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PLAN OF STANDARD EQUIPMENT ARRANGEMENT

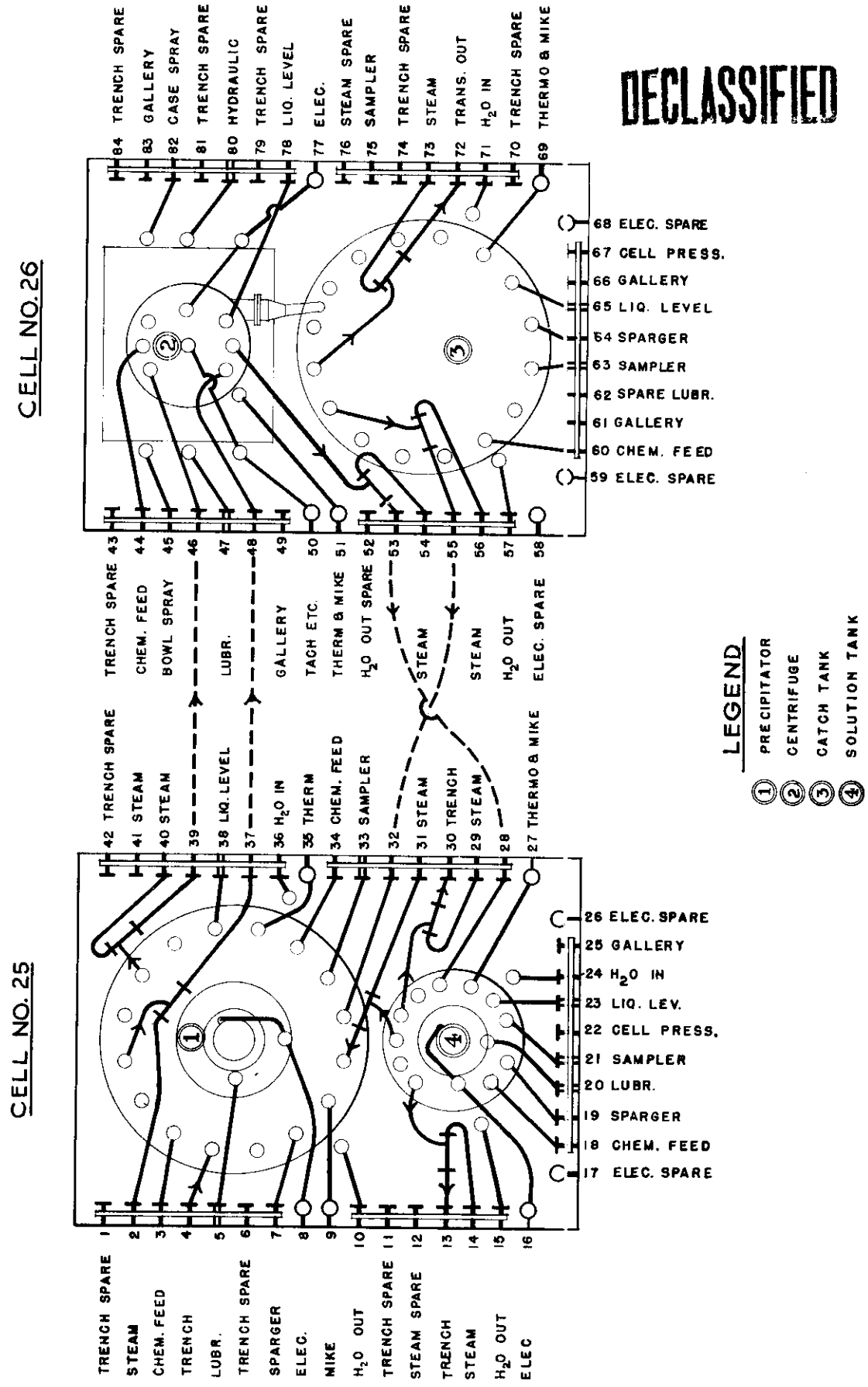

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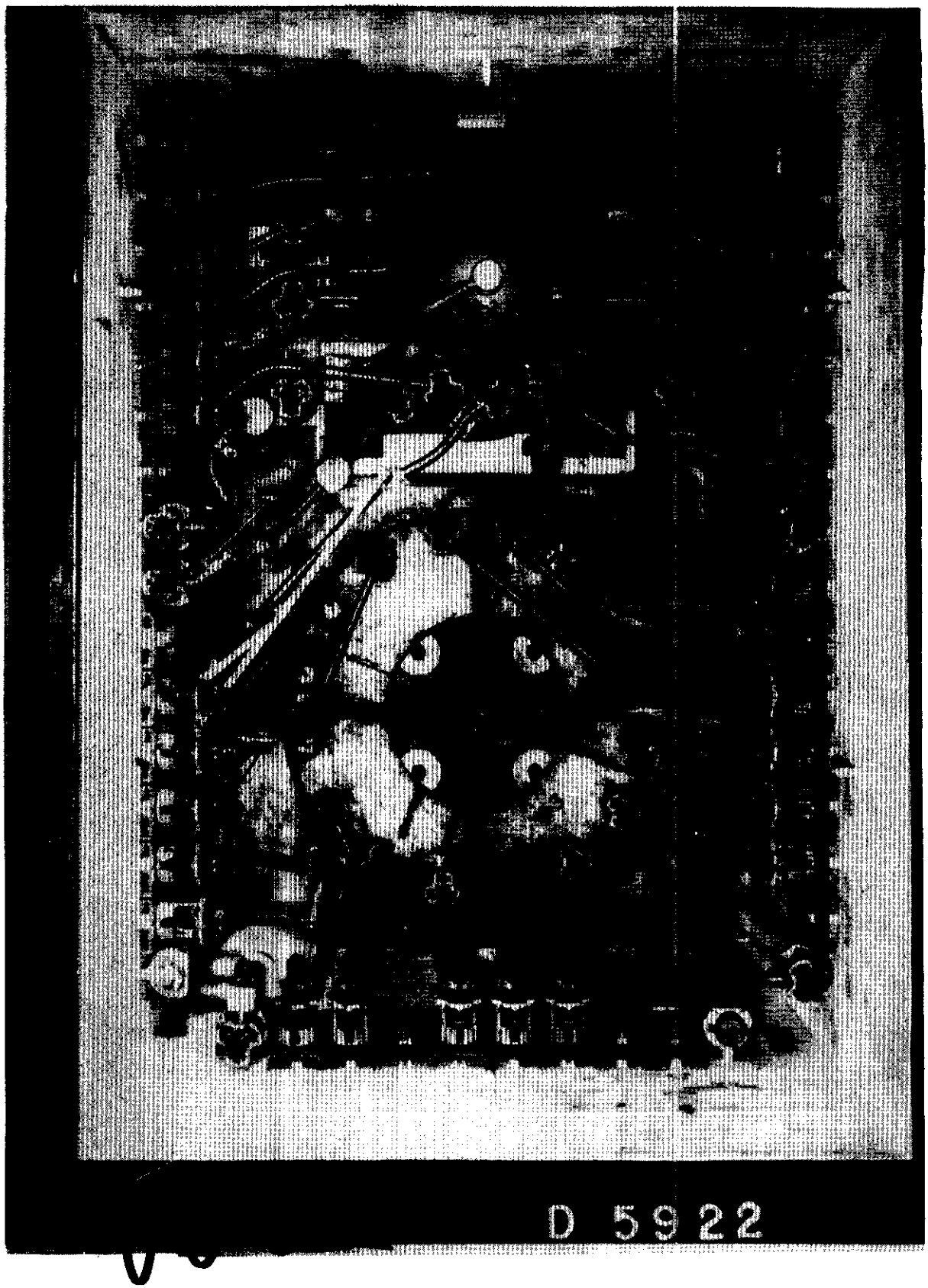
FIGURE 47

STANDARD CELL NO. 11 - SECTION 6

FIGURE 48

STANDARD CELL NO. 12 - SECTION 6



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Cell Functions

The 221 Building facilities provide for carrying out the separation process to the point where a decontamination factor of 10^5 is reached. The following tabulation illustrates the functions of the various cells:

TABLE I

Section	Cell	Function
1	1 & 2	Storage of contaminated discarded equipment
2	3	Railroad tunnel for bringing in metal
2	4	Storage of slugs with fractured coatings. (This cell is kept filled with water). (See 212 Building)
3	5 & 6	Coating removal, metal dissolving and reduction
4	7	Coating removal, metal dissolving and reduction
4	8	Metal solution storage
5	9	Sewage disposal, holding tanks
5	10	Sewage disposal, sewer cell
6	11 & 12	Spare. May be used for a by-product precipitation before extraction
7	13 & 14	Extraction (Spare)
8	15 & 16	Extraction
9	17 & 18	Treatment of waste metal solution
10	19 & 20	Treatment of waste metal solution (Spare)
11	21 & 22	Spare, unequipped
12	23 & 24	Storage and oxidation of metal solutions
13	25 & 26	First decontamination cycle, by-product precipitation
14	27 & 28	First decontamination cycle, product precipitation
15	29 & 30	Treatment of decontamination wastes

[redacted]

Section	Cell	Function
16	31 & 32	Second decontamination cycle, by-product precipitation
17	33 & 34	Second decontamination cycle, product precipitation
18	35 & 36	Third decontamination cycle, (Spare)
19	37 & 38	Third decontamination cycle, (Spare)
20	39 & 40	Spare, unequipped

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Pipe Trench

Process piping which carries active solutions between sections is installed in a pipe trench which runs from Section 3 to Section 20. Lines from the cells pass through the concrete and terminate in connector flanges in the trench. Just as in the cells, the connector flanges are held in fixed standard position by steel supports embedded in the concrete trench floor. The trench piping is in prefabricated sections attached to the flanges with automatic connectors. (See Figures 49 and 50) The trench cover is in removable sections, similar to the cell covers. Alterations and replacements of trench piping can be made with the same remotely operated equipment used for cell maintenance.

In addition to avoiding lines through expansion joints, the pipe trench serves other purposes. It makes process lines accessible for maintenance and contributes flexibility since sections can be hooked up through the trench in different ways to conform to process changes. (See Figure 51 for a diagram of the trench piping.)

Pipe Gallery

All cell piping except process transfer lines is brought up to the pipe gallery, terminating in connections on the wall. From here, connections are made to the weigh tanks and control boards in the operating gallery. Remote maintenance is not required, so all connections are of the normal type and the gallery piping is not buried in concrete. Chemical headers, electrical and steam distribution lines are also located in this gallery. (See Figure 52 for a view of the pipe gallery.)

Operating Gallery

The operating gallery is the control center for cell equipment. At each section is a gauge board from which control and instrument lines run to the cells, via the pipe gallery. Weigh tanks are provided with inlet connections from appropriate chemical headers in the pipe gallery (See Figure 54) and outlets to the cell vessel connections, also located in the pipe gallerys. (See Figure 53 for a view of the operating gallery.)

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FIGURE 49

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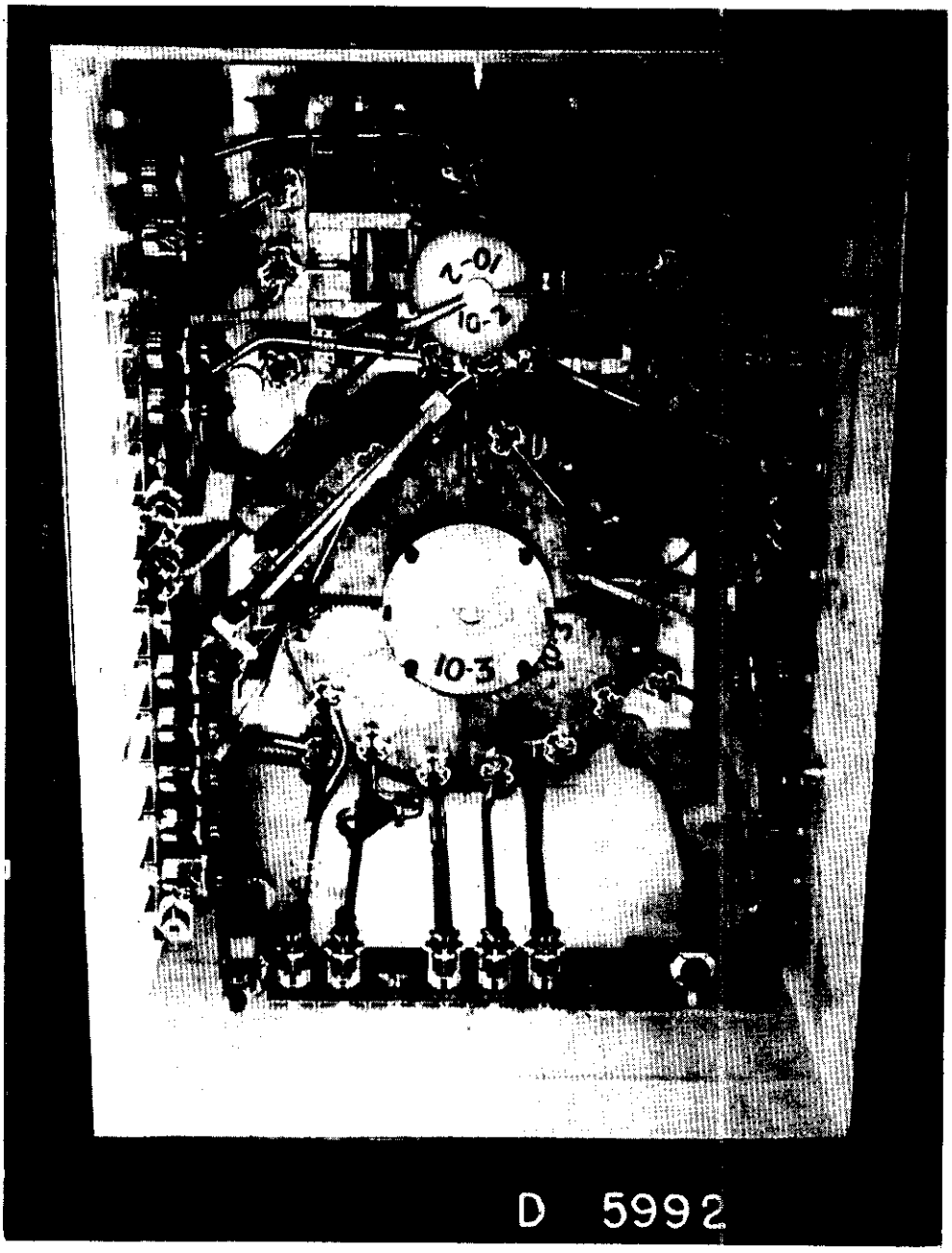

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FIGURE 50

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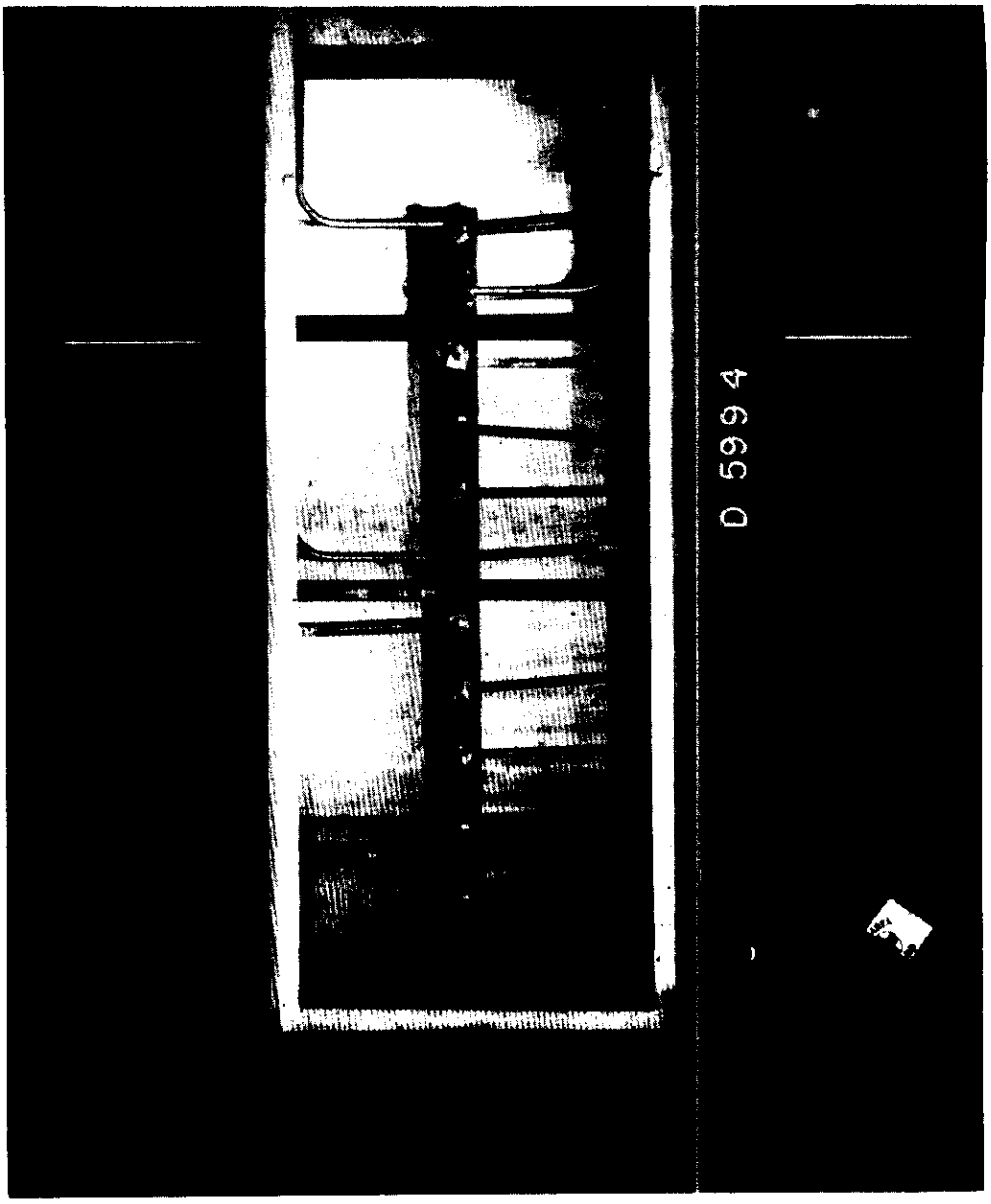

TRENCH PIPING - CANYON BUILDING

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FIGURE 51

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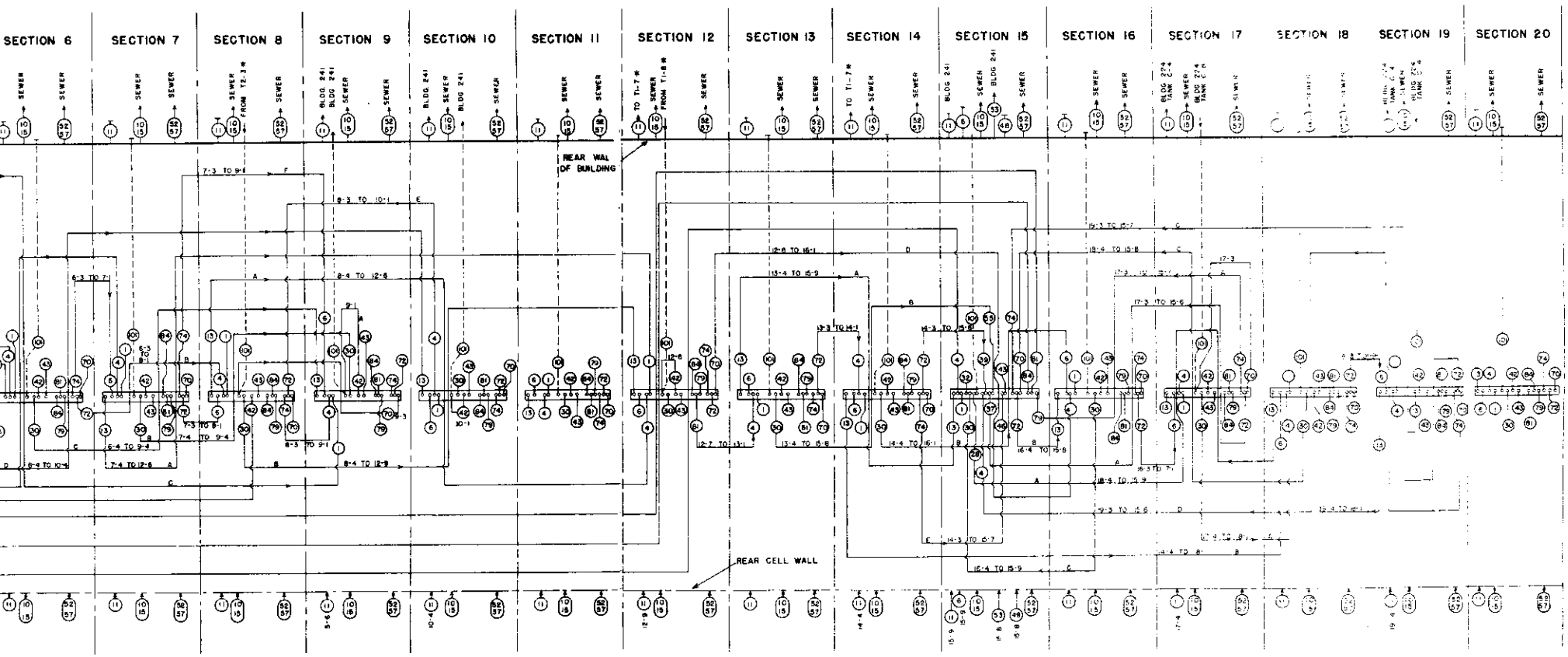
TRENCH PIPING DIAGRAM AND CONNECTIONS TO REAR OF CANYON (221) BUILDINGS.



REFERENCE DRAWINGS

W 73338	TRENCH PIPING, SECTIONS 3 TO 9
W 73339	TRENCH PIPING, SECTIONS 9 TO 15
W 73340	TRENCH PIPING, SECTIONS 15 TO 20
W 76585	ALTERNATE TRENCH PIPING
W 72189	PIPING THROUGH CONCRETE, SECTIONS 1 AND 2
W 74399	VENTILATION AND 24 INCH DRAIN
W 89333-4	CONCRETE, SECTIONS 1 AND 2

FIGURE 52

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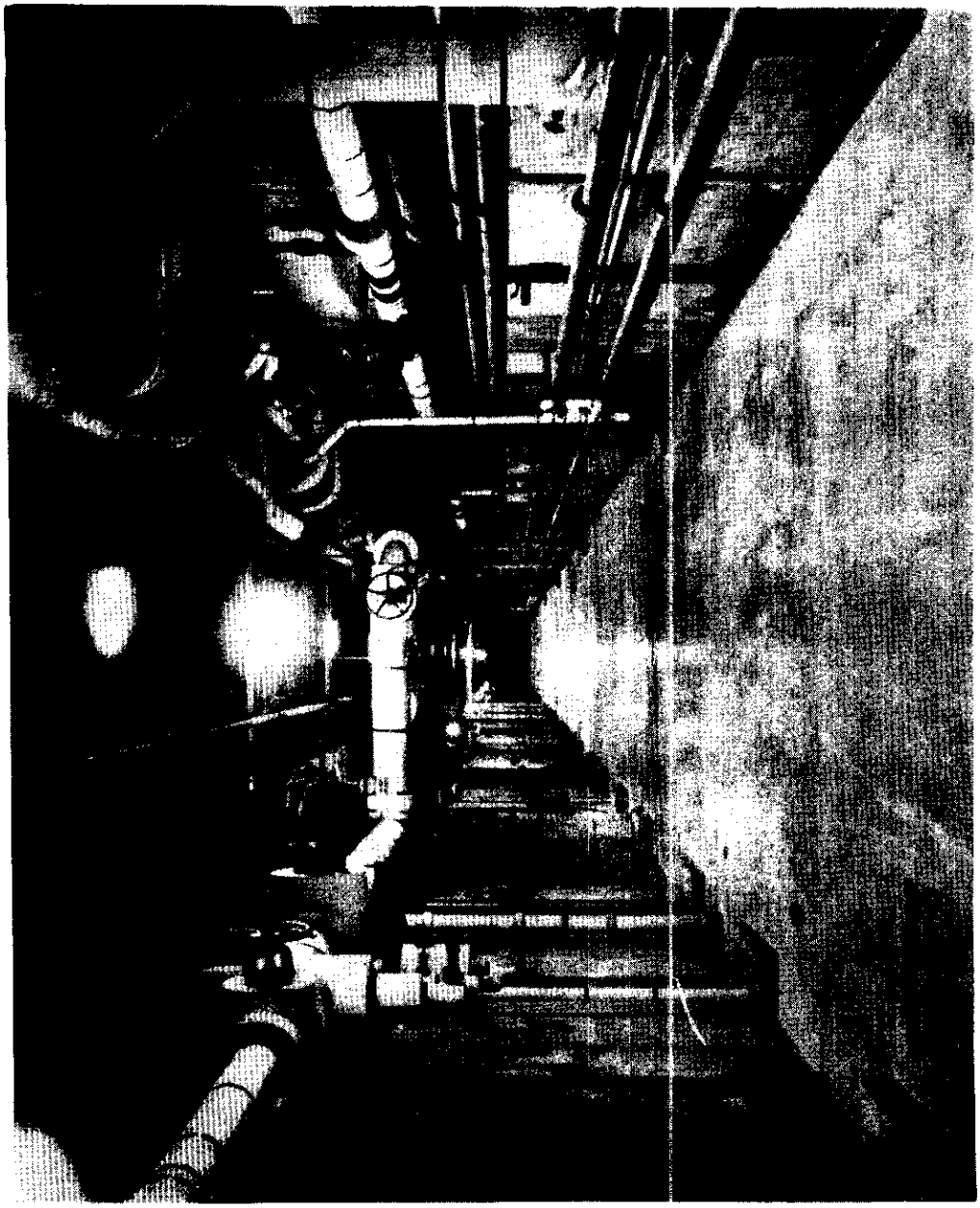

PIPE GALLERY - CANYON BUILDING

FIGURE 53

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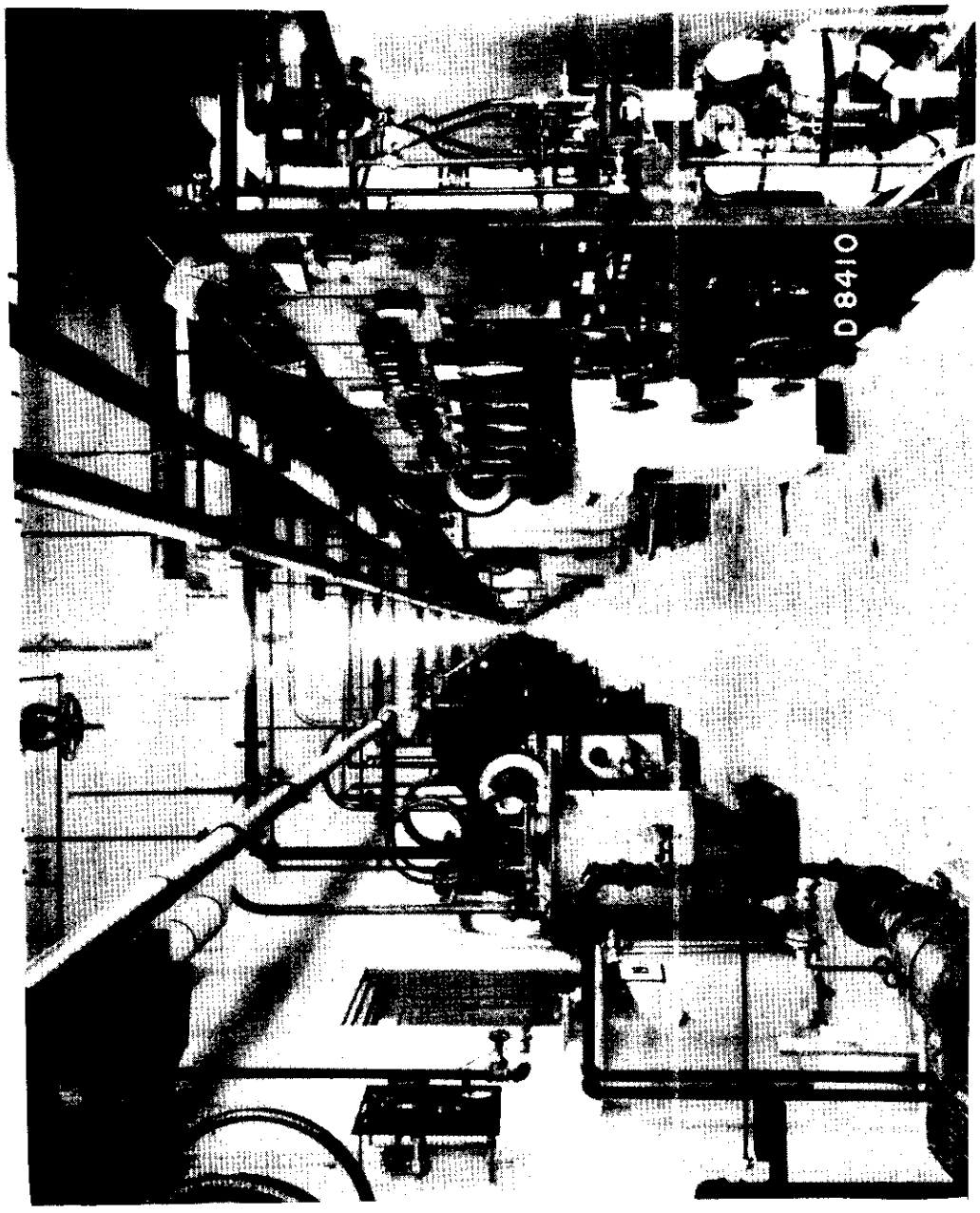

OPERATING GALLERY - CANYON BUILDING

HW 10475-C

FIGURE 54

CHEMICAL HEADER PIPING
BUILDINGS 211, 221, AND 271

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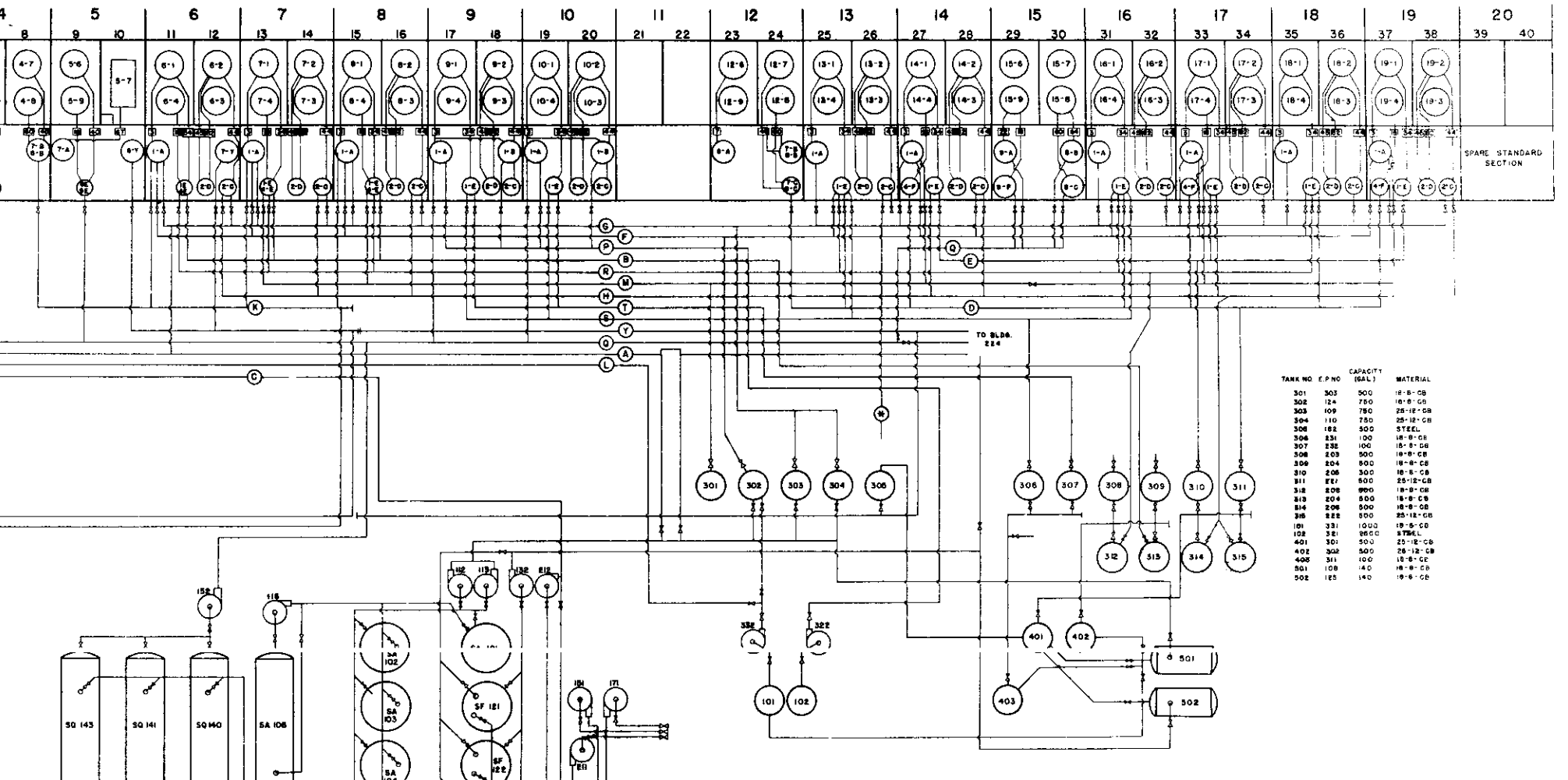

TANK NO.	E.P. NO	CAPACITY (GAL.)	MATERIAL
301	303	500	18-8-CB
302	124	750	18-8-CB
303	109	750	25-12-CB
304	110	750	25-12-CB
305	182	500	STEEL
306	231	100	18-8-CB
307	235	100	18-8-CB
308	203	500	18-8-CB
309	204	500	18-8-CB
310	206	300	18-8-CB
311	227	500	25-12-CB
312	208	990	18-8-CB
313	209	500	18-8-CB
314	208	500	18-8-CB
315	222	500	25-12-CB
101	331	1000	18-8-CB
102	321	9800	STEEL
401	301	500	25-12-CB
402	302	500	25-12-CB
403	311	100	18-8-CB
501	108	140	18-8-CB
502	125	140	18-8-CB

Electrical Gallery

The electrical or basement gallery contains principally electrical lines. The steam main also enters the building through this gallery. (For a view of the electrical gallery see Figure 55.)

BUILDING NO. 271 - SERVICE BUILDING

The Service Building is attached to the Canyon Building on the operating gallery side. The relationship of 271, 221, and other buildings in the immediate neighborhood is shown in Figure 31. The 271 Building serves for the preparation of process chemicals and as the administrative headquarters for the 221 Building. It is a four story building approximately 48 feet by 160 feet. The layout of the building is shown in Figures 56 and 57.

The basement floor of the 271 Building contains storage rooms of various kinds, instrument and maintenance shops, and space to house the ventilating equipment for the building; and in addition, the compressed air supply for Buildings 221, 222, and 224.

The first floor contains change room facilities and additional storage space for solid chemicals.

The second floor includes office space for supervision and the dispatcher's quarters. The dispatcher's office contains all the communication facilities for controlling the movements of personnel and materials in carrying out the operations in the Canyon Building.

Figure 58 illustrates the Canyon Building communication network through which the dispatcher keeps track of operating personnel in all parts of the building. All doors giving access to the canyon deck or the crane cabway are equipped with magnetically operated locks which are controlled at the dispatcher's office. Telephones are also located at these doors so that personnel can obtain the dispatcher's permission to enter the building. A communication network is provided on the main deck of the 221 Building. Operating personnel working in this area are required to carry plug-in telephones which are always connected directly to the dispatcher's desk so that constant telephone communication can be maintained. The crane operator is also in constant communication with the dispatcher, both by a telephone and a two-way loud speaker communication system. An alarm system is also provided at the deck to clear personnel from this area when conditions are unsafe. In addition, to this control of the movement of personnel to the operating building, the dispatcher controls the process steps. Movement of chemicals and process solutions from one part of the building to another are cleared by the operators through the dispatcher's office, using the gallery telephones located on each gauge board. Hence the 271 Building represents the nerve center of the entire process operations taking place in this area.

The third floor of the 271 Building contains facilities for preparing

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FIGURE 55

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ELECTRICAL GALLERY - CANYON BUILDING

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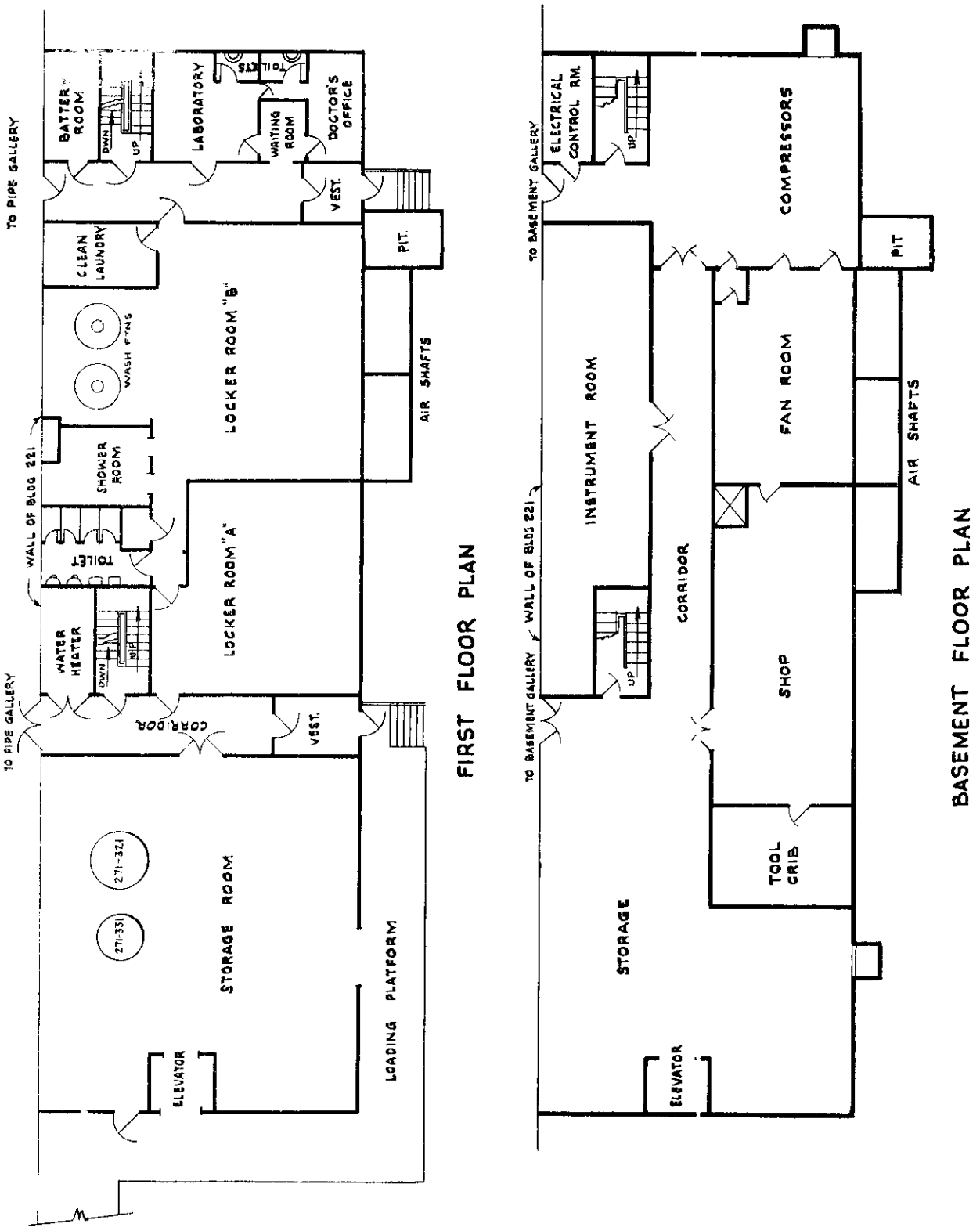
FIGURE 56



271 BUILDING

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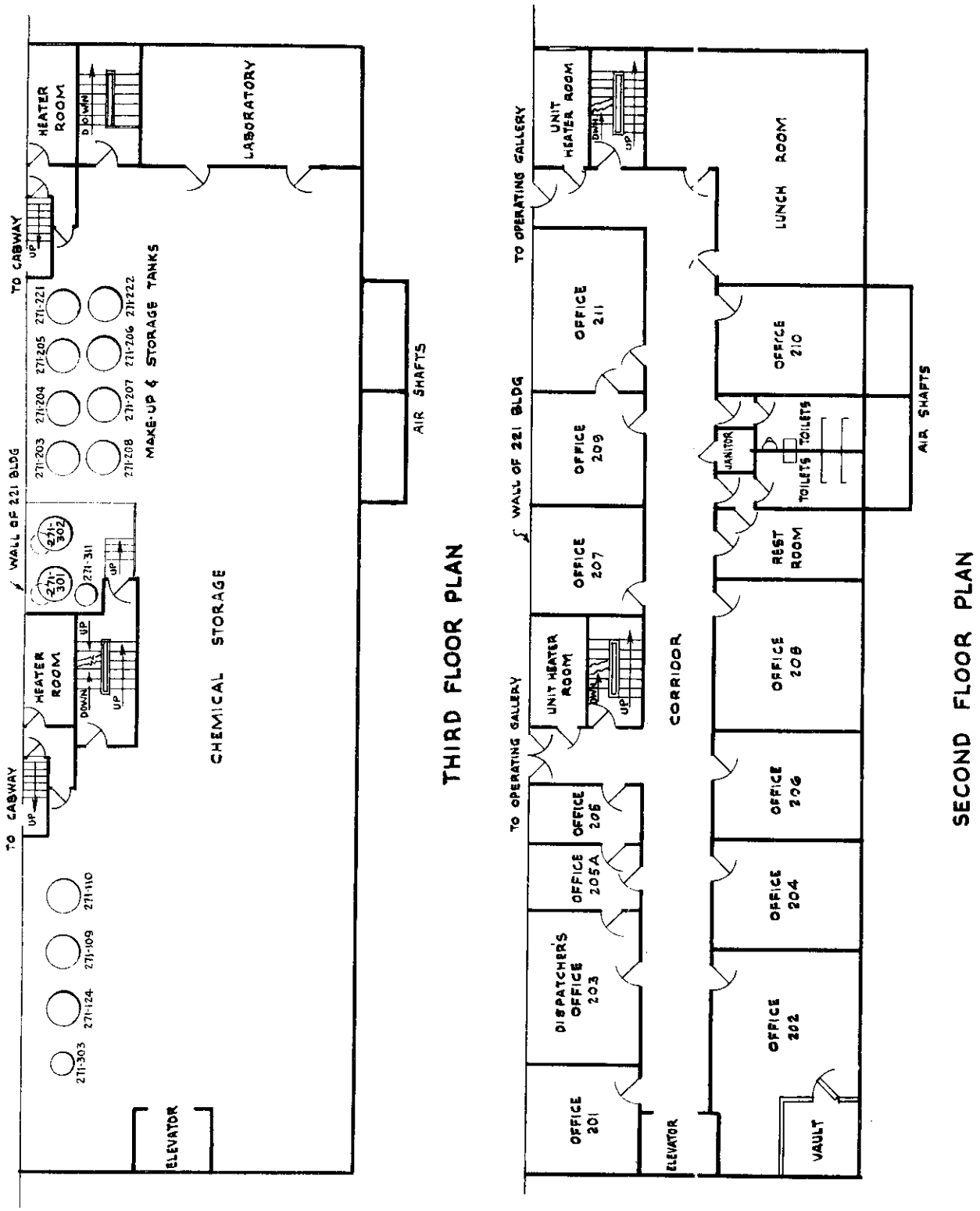
FIGURE 57



271 BUILDING

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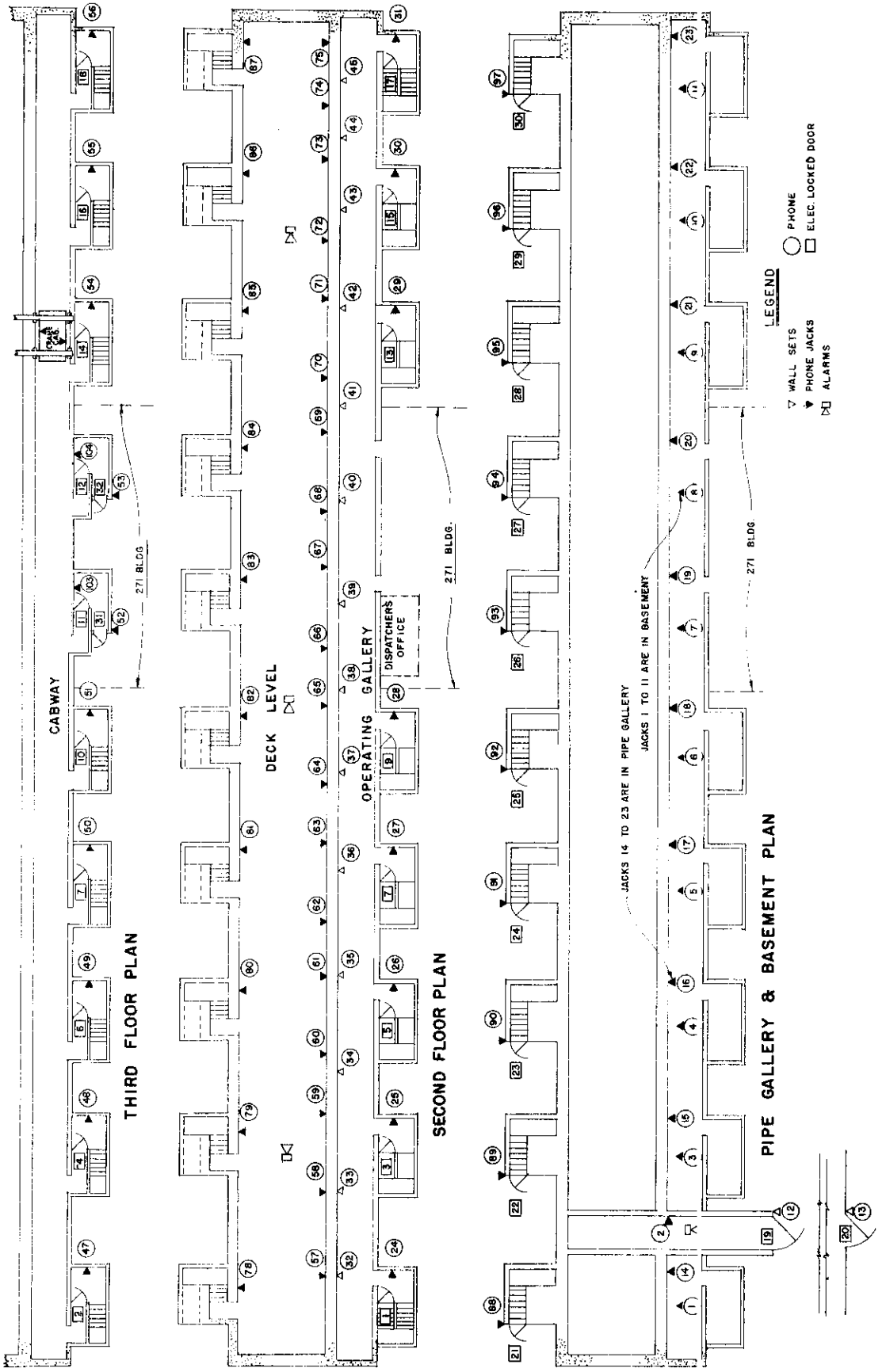
FIGURE 58



CANYON BUILDING—COMMUNICATIONS

process chemicals to be used in the individual steps. The lines carrying chemicals from 271 Building and the Tank Farm (221 Building) are diagrammed in Figure 54. A chemical control laboratory is provided on this floor to check purity and concentrations of various chemical solutions. Access to the crane cabway is also provided by two labyrinth access ways which duplicate those in the outside stairways along the face of the 221 Building.

BUILDING NO. 211 - TANK FARM

The operating storage tanks for bulk chemicals are located adjacent to the 271 Building, as shown in Figures 31 and 59. This consists of five vertical stainless steel storage tanks for handling 60% nitric acid, three similar tanks for handling 75% phosphoric acid, and one for 90% formic acid. A horizontal stainless steel tank is also provided for storage of concentrated nitric acid as received from tank cars together with means for diluting this acid to working strength (60%) before it is discharged to the vertical storage tanks. Three steel tanks are provided for storage of 50% caustic, an adjacent steel tank for the storage of concentrated sulfuric acid, and a steel tank on scales for the storage of anhydrous hydrofluoric acid.

BUILDING NO. 222 - CONTROL LABORATORY

The Control Laboratory (222) Building, illustrated in Figure 28, contains the laboratory and testing facilities required for the necessary process control analyses. The bulk of the analyses are radiochemical and consist in preparing a small aliquot of a radiochemical process solution in a form so that rates of radioactive disintegration can be determined. The product concentration in various steps is determined by counting the alpha disintegrating rate. The extent of decontamination is similarly determined by measuring the beta and gamma disintegration rates.

BUILDING NO. 224 - CONCENTRATION BUILDING

As soon as decontamination has sufficiently reduced the level of radioactivity, it is advantageous to transfer operations out of the massive shielding of 221 Building and into a more normal type of structure, where equipment can be cleaned and maintained in the usual manner. Therefore, the later stages of decontamination, as well as the concentration operation, are carried out in 224 Building illustrated in Figure 20.

The layout of the 224 Building is shown in Figure 59. It contains six working areas termed cells, in keeping with the designation used in the 221 Building. Four of the cells, A, B, D, and E are identical and each contains

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FIGURE 59

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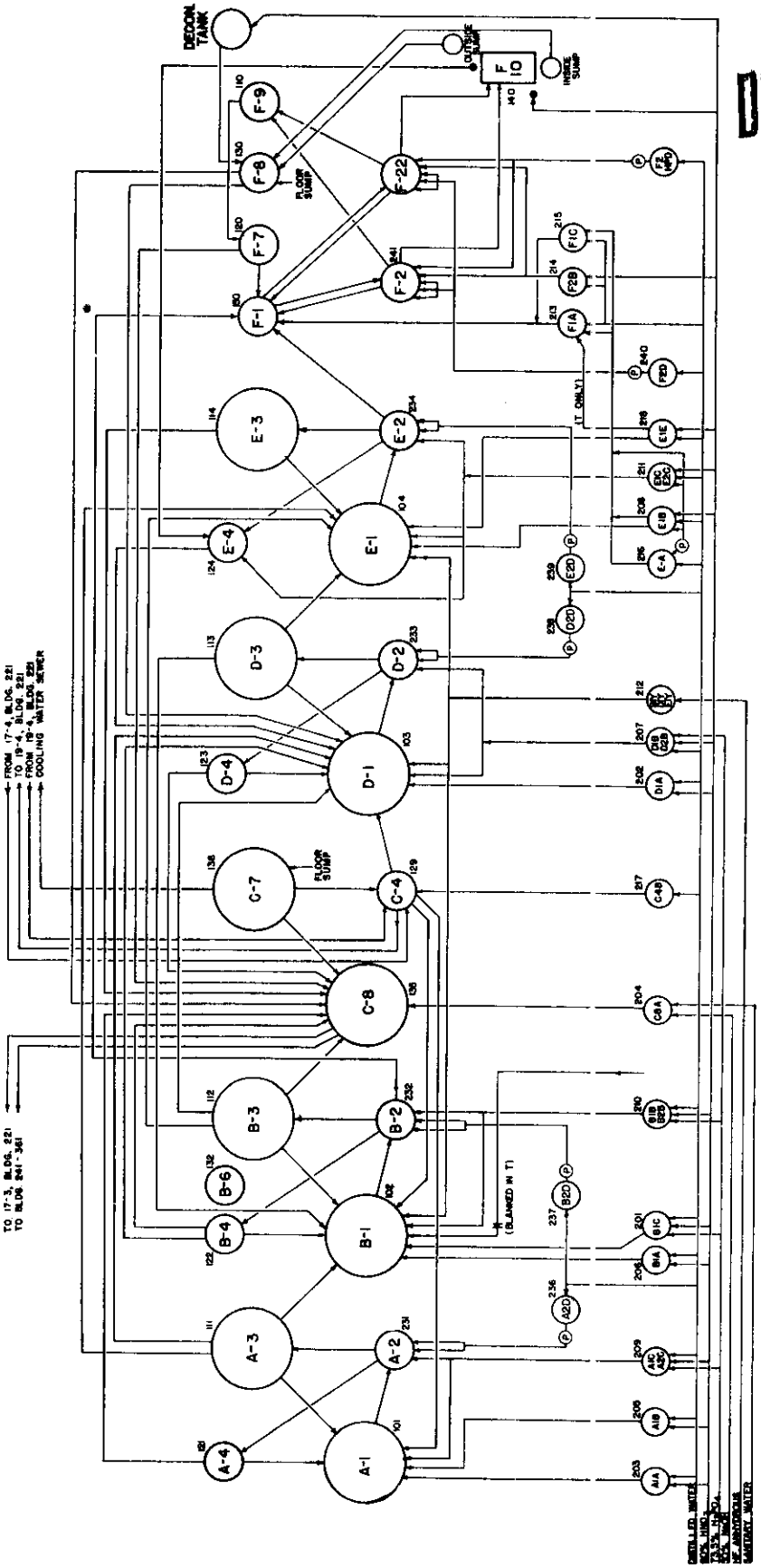
CONCENTRATION BUILDING CHEMICAL AND PROCESS PIPING

PIPING DIAGRAM OF THE CONCENTRATION (224) BUILDING



[redacted]

a standard precipitator, centrifuge, catch tank, solution tank grouping. This equipment in the main is identical in design with that supplied in the 221 Building, except that ordinary bolted pipe flanges are used instead of the remotely operated connectors. The process piping in this building is laid out in the usual manner. There is no pipe trench, and piping is not buried in concrete walls but is brought into or out of the cells through slots.

The residual radioactivity present in the process solution is high enough to make some shielding necessary. The required degree of protection is obtained by placing 12 inches of concrete between operating process vessels and personnel. In addition, the arrangement of process vessels, concrete walls and gallery is such that personnel in the gallery are at least 10 feet from process vessels. This full degree of protection is only required for continuous 8-hour operation. Process vessels can be approached somewhat closer, for example, during the taking of control samples, provided that the length of stay in the vicinity of the vessel is of the order of a few minutes.

The right half of Cell C is 19 feet lower that the other cells in the building. In it is placed a receiving tank of 800 gallons capacity, connected to the solution tanks in Sections 17 and 19 of 221 Building. The process lines from 221 Building are so pitched that they drain freely into the receiving tank of Cell C. In this way, quantitative transfers without line holdup can be made from 221 Building to 224 Building. This pit also contains a sump tank to collect 224 Building drainage. In the left half of Cell C is a waste neutralizing tank similar in design and function to the waste neutralizing tanks provided in Section 15 of 221 Building.

The equipment in Cell A is used to carry out the bismuth phosphate by-product precipitation during the cross-over step. Cell B is a spare cell sometimes used to carry out either lanthanum fluoride by-product or product precipitations in the cross-over step. Cell D is the cell normally used for the lanthanum fluoride by-product step and Cell E for the subsequent lanthanum fluoride product precipitation. The lanthanum fluoride product cake is separated out in the 40-inch centrifuge of Cell E, and is slurried from the bowl of this centrifuge to an 800-gallon holdup tank in Cell F. Cell F is used for the metathesis and solution steps of the concentration process. Figure 60 illustrates the equipment in Cells A to C inclusive, and Figures 61 & 62 illustrate the operating gallery and chemical solution feed tanks for this section of this building.

The equipment in Cell F consists of three agitated tanks, one unagitated tank, and two 26-inch centrifuges. This equipment is illustrated in Figures 63 to 66 inclusive. The metathesis of the lanthanum fluoride precipitate takes place in these vessels and the resulting lanthanum and product hydroxides are separated in the 26-inch centrifuge, and then washed and dissolved in nitric acid. The resulting solution of lanthanum and product nitrates is stored in a small weigh tank (F-10) in a glass enclosed cage in Cell F. (See Figure 66). This tank is rectangular in shape and is only 4 inches thick. The purpose of this shape is to prevent undesirable side reactions, leading to fission and loss of decontamination, by facilitating the escape of any neutrons that may be formed in the solution.

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FIGURE 60

CELLS A, B, C, D AND E - CONCENTRATION BUILDING

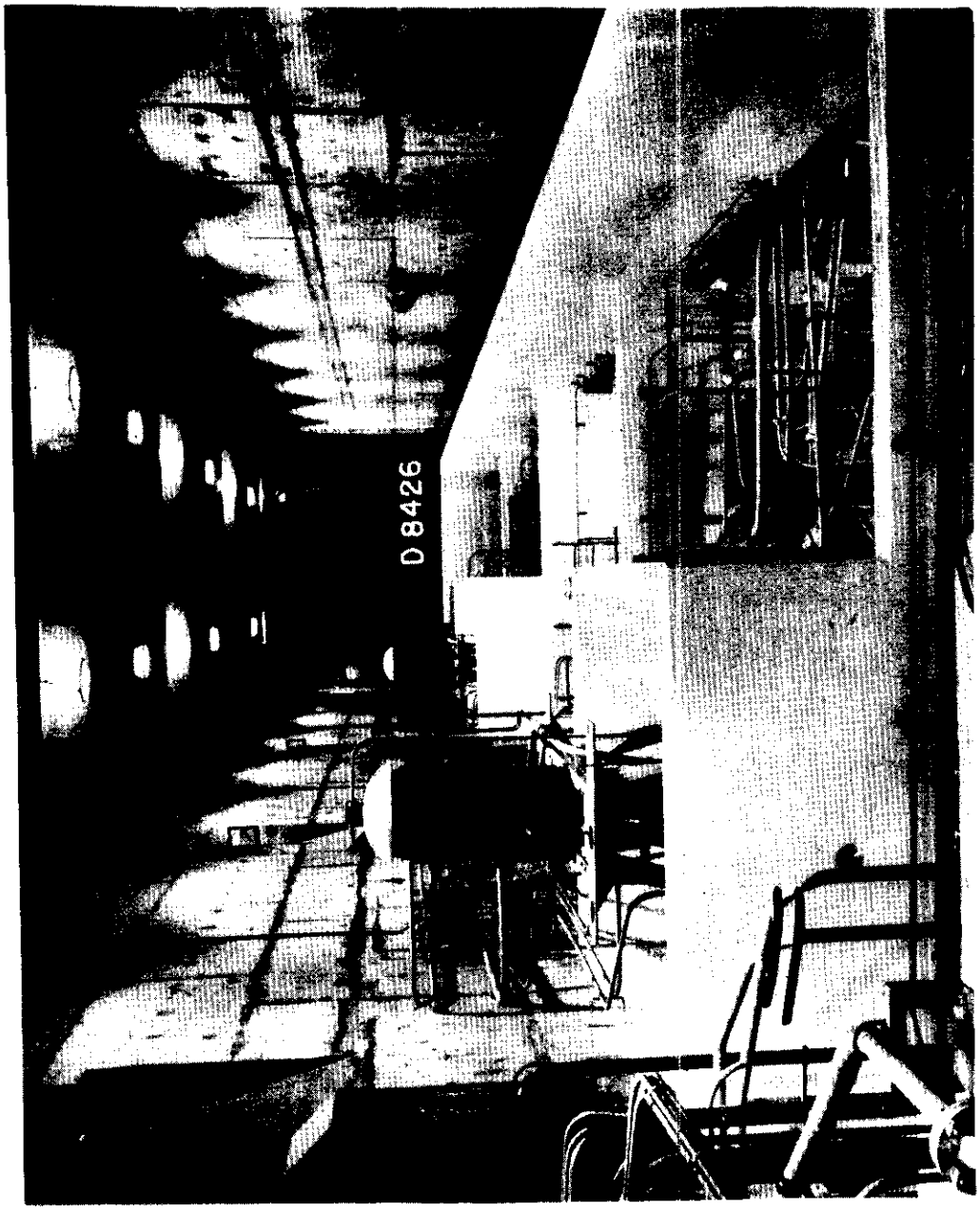

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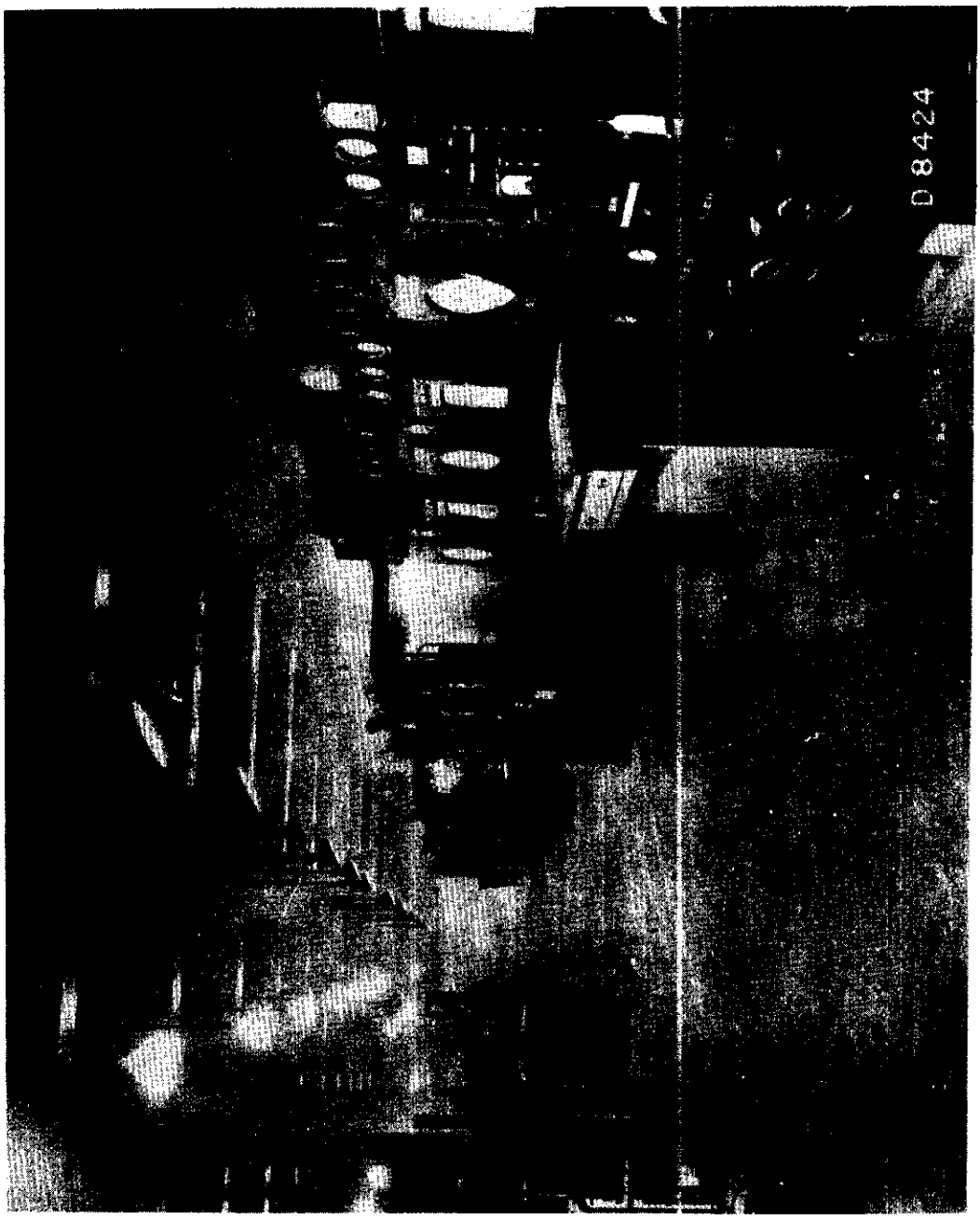
FIGURE 61



OPERATING GALLERY - CELLS A, B, C, D AND E - CONCENTRATION BUILDING

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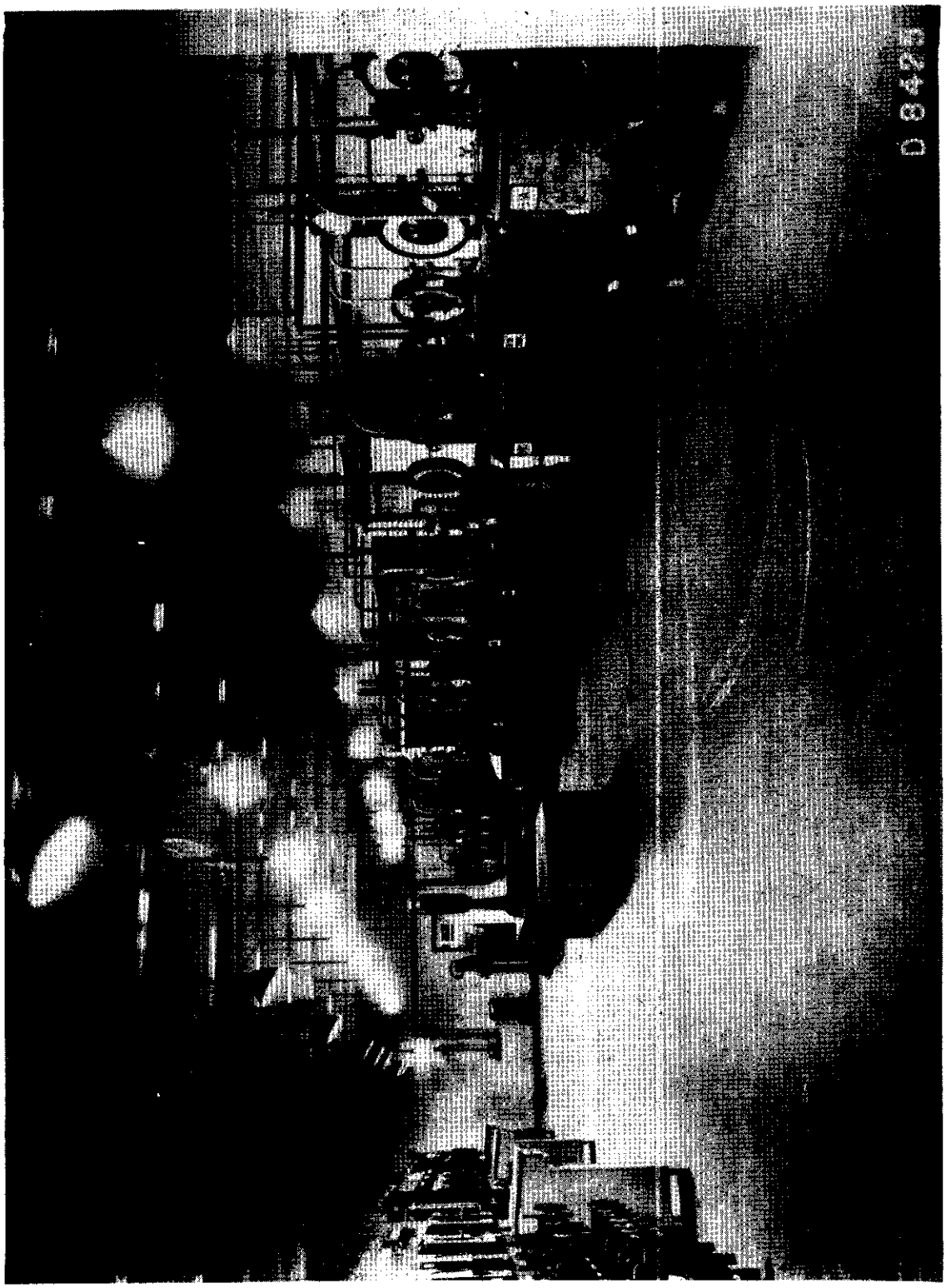
FIGURE 62



CHEMICAL SOLUTION MAKE-UP TANKS - CONCENTRATION BUILDING

FIGURE 63

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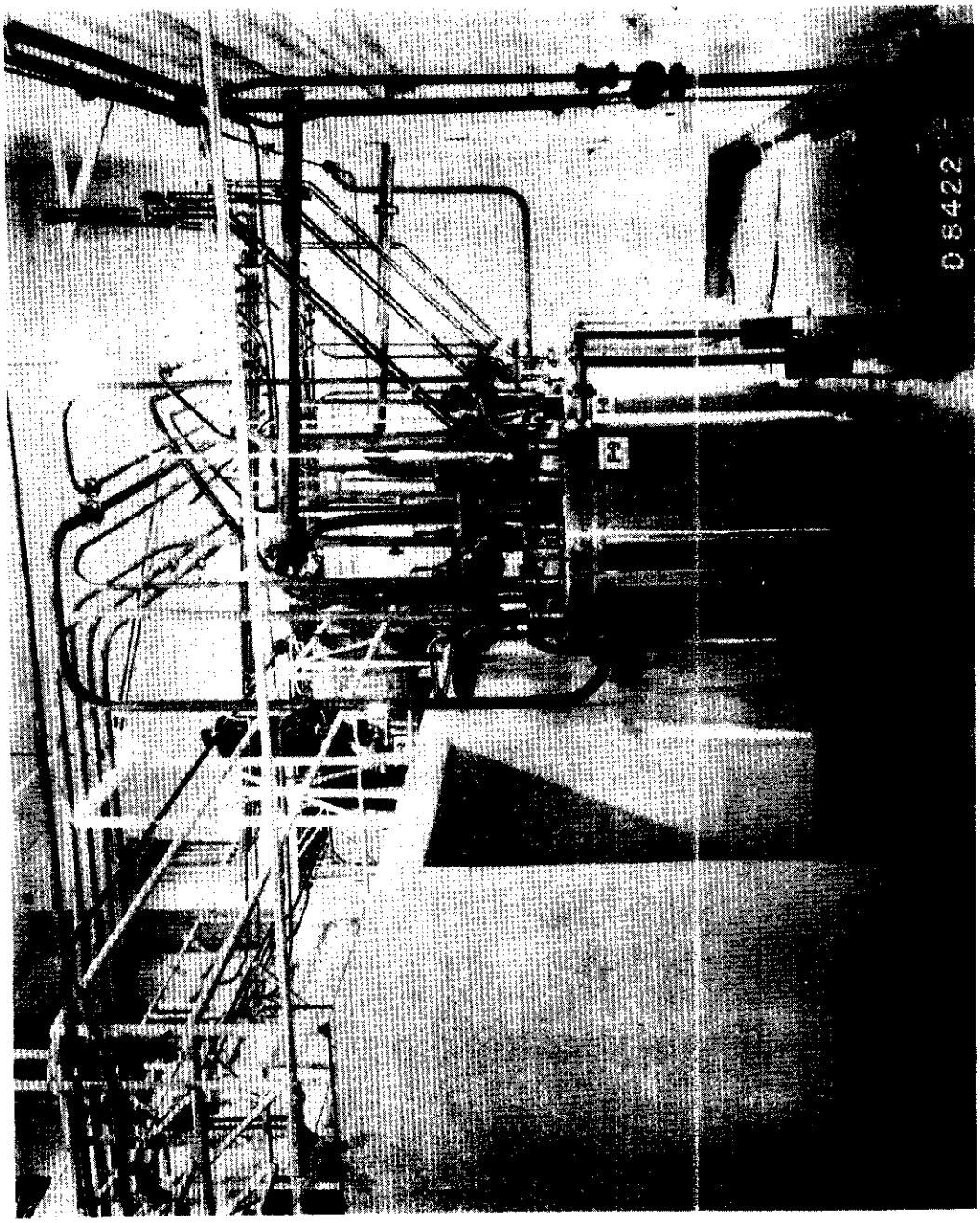

F CELL - CONCENTRATION BUILDING

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FIGURE 64

F CELL - CONCENTRATION BUILDING

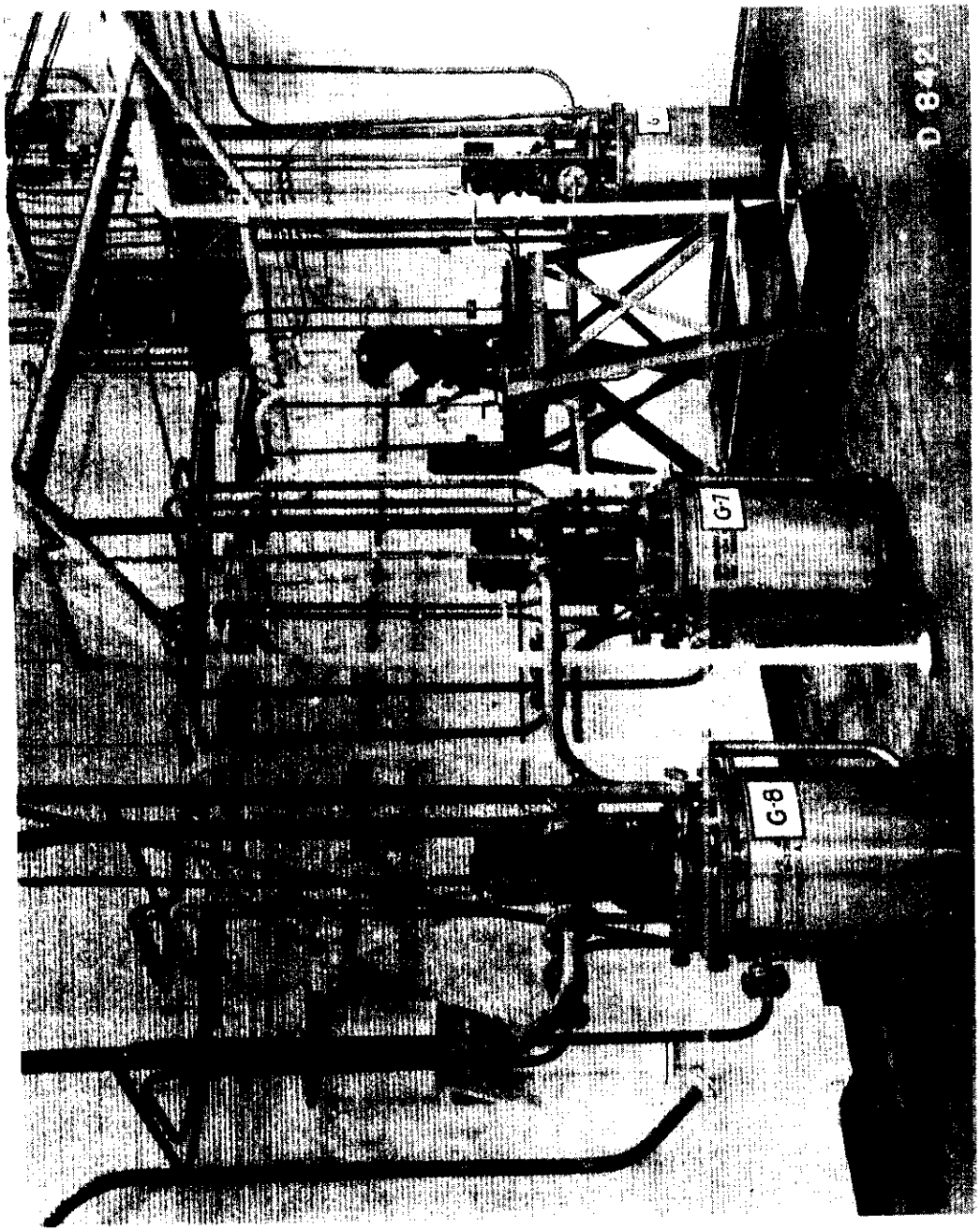

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FIGURE 65

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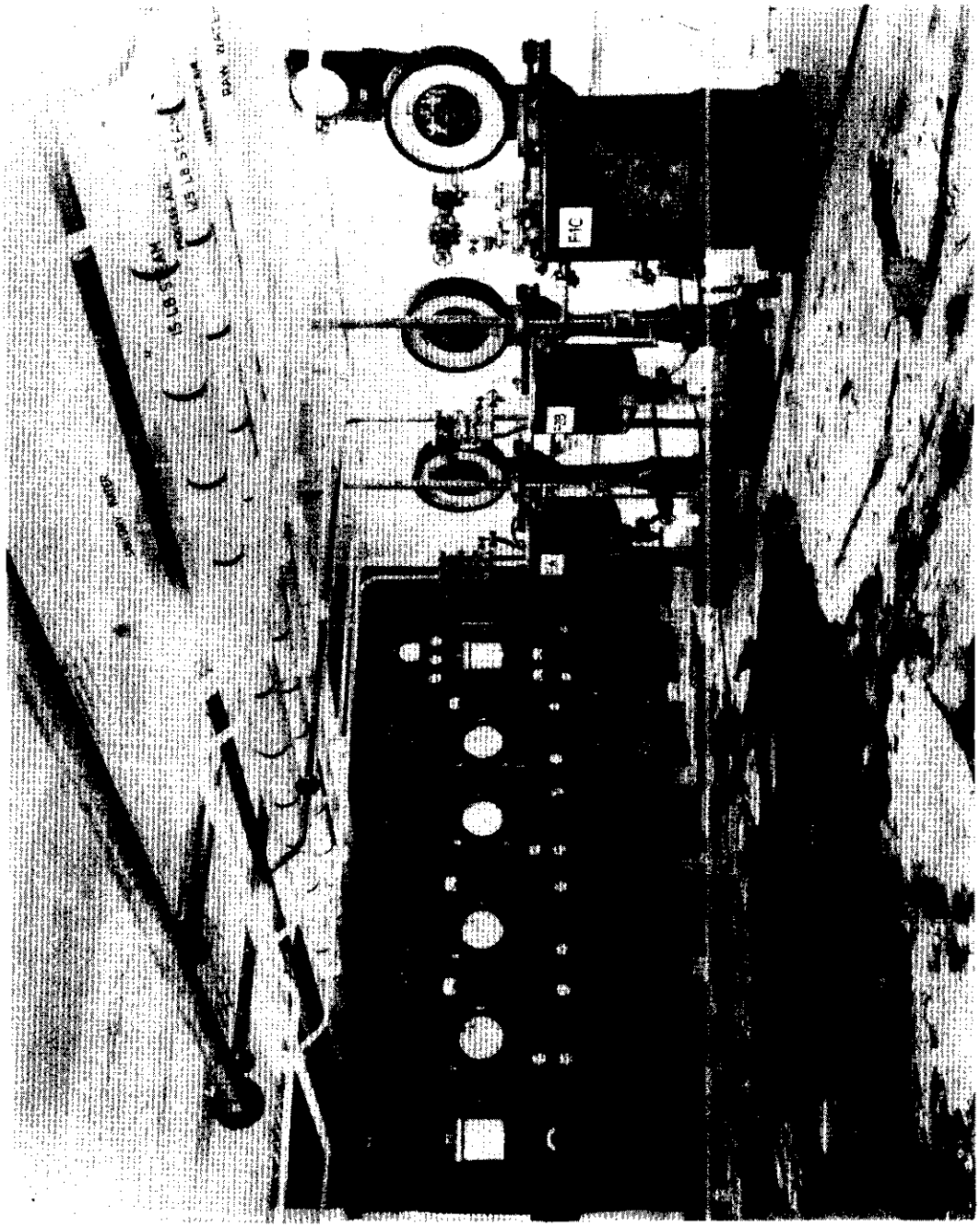

F CELL OPERATING GALLERY - CONCENTRATION BUILDING

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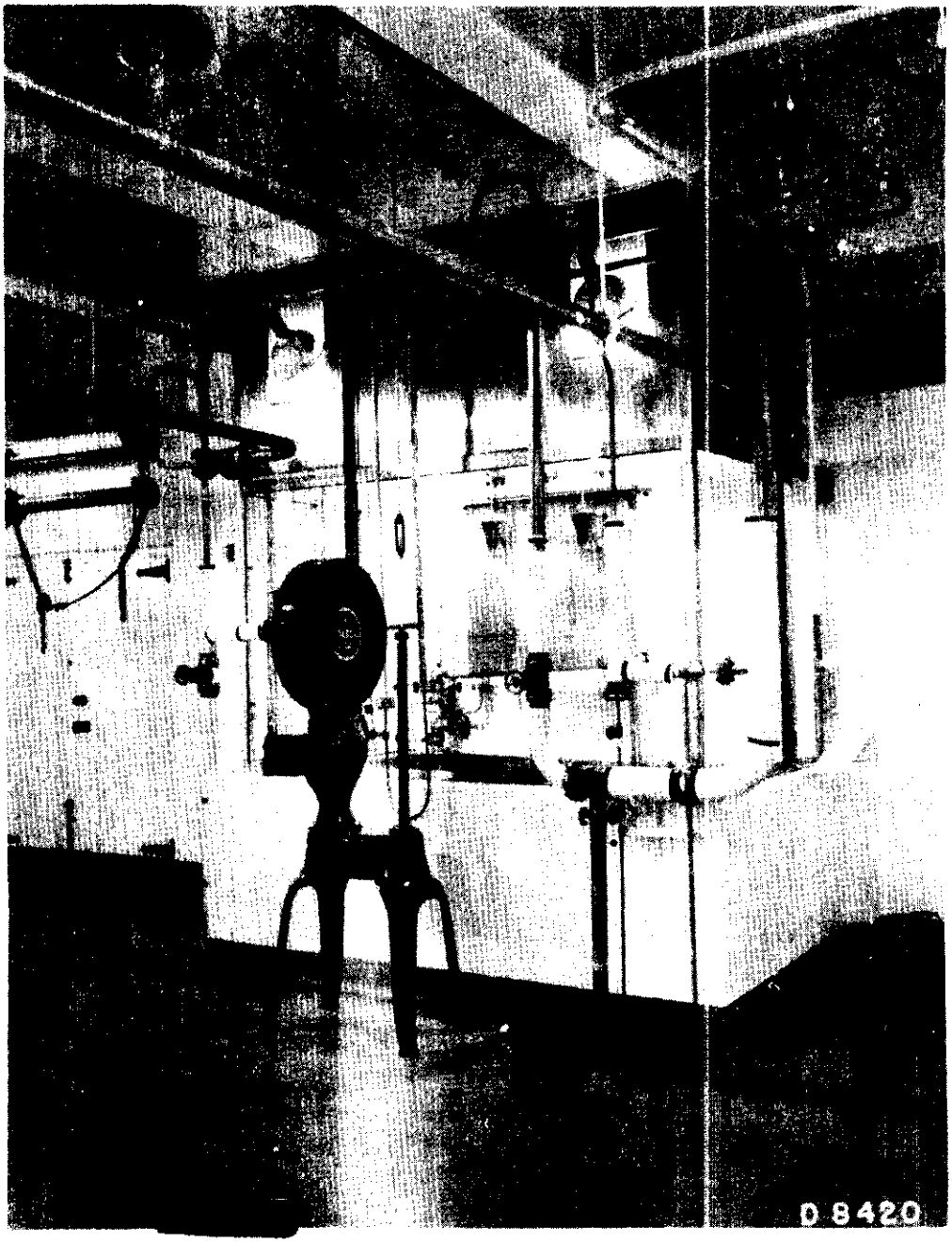
FIGURE 66

F-10 CAGE - CONCENTRATION BUILDING



The centrifuges in the F-Cell are equipped with a high pressure (300 lbs./sq.in.) water spray header containing two flat spray nozzles having a water delivery of about 3.5 gal./min. The nozzles on this header are positioned to cover the two lower shelves in the centrifuge baskets and facilitate the removal of the lanthanum hydroxide cake obtained during metathesis. This high pressure water spray system is in addition to the 100 lb./sq.in. water spray header present in these centrifuges, as in all others in the 221 and 224 Buildings. Water pressure for each spray header on the centrifuges is supplied by individual water pumps.

The contents of the F-10 tank are transferred to the transfer can (F-1) (illustrated in Figure 67) for shipping by truck to the Isolation (231) Building.

Other features of the 224 Building are given in Figures 59 and 60. In general, the layout is somewhat similar to that of 221 Building. The operating gallery is on the third floor so that gravity flow of chemicals can be used for transfer from the weigh tanks to the operating vessels in the cells. Under the operating gallery is a small pipe gallery serving somewhat the same function as the corresponding gallery in 221 Building. The first floor of the building is used for storage, offices, and change room facilities. The end of the building containing Cell F is designed on a somewhat similar principle, although because of the smaller scale of operation at this point of the process, a simpler arrangement of the operating gallery is provided.

200 AREA EQUIPMENT OPERATING NUMBERS

The equipment pieces in the various piping diagrams are identified with the operating numbers used on log sheets, gauge board labelling, and the like.

Vessels in the 221 Building standard sections (6-10 inclusive, 13, 14 and 16-19) are designated by a number which includes the section number and a position number, 1 to 4. Positions are numbered clockwise in a section, starting on the far left. In 221 and 224 Buildings "right" or "left" is referred to an observer in the operating gallery, facing the cells. The dissolvers are all numbered 5, preceded by the section number and followed by L or R for the left or right position of the section, respectively. In the waste and storage section, operating numbers consist of the section number followed by 6, 7, 8, or 9, numbering clockwise from the far left. Example: 3-5-R is the right-hand Section 3 dissolver and 12-9 is the near crude product storage tank in Section 12.

In 224 Building, a process vessel is assigned a cell letter (A to F) followed by number used for that type of vessel in 221 Building, disregarding position. Thus B-2 is the Cell B centrifuge, since 2 is the number of all centrifuges in 221 Building.

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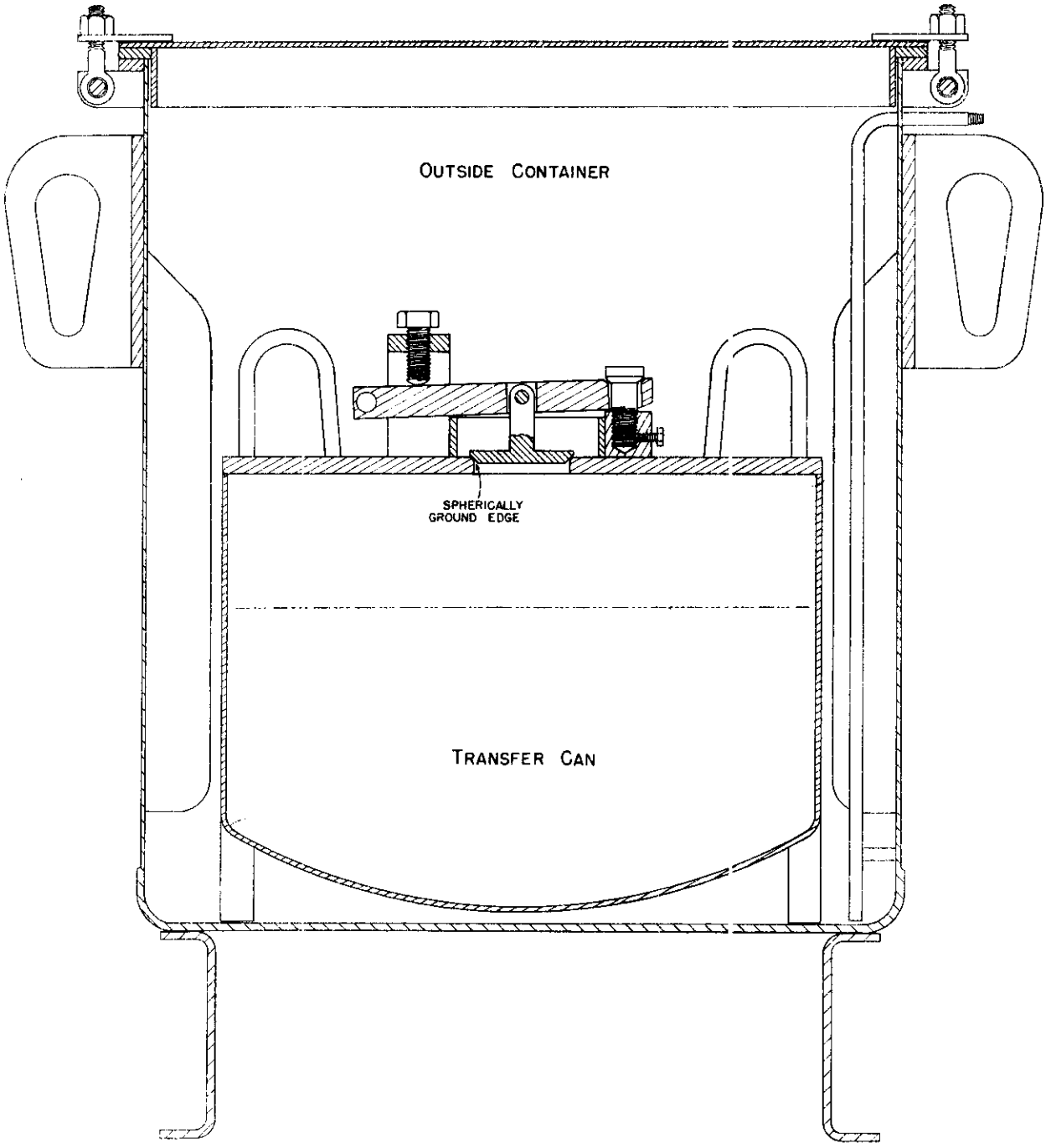
FIGURE 67

SOLUTION TRANSFER CAN AND OUTER CONTAINER



A gallery tank is given the number of the process vessel to which it feeds, followed by a letter indicating the position. The positions are lettered clockwise facing the gauge board with A at the left of the gauge board, B at the right C, D, E, and F along the back wall from right to left. All HF tanks have the position letter replaced by Y. In some instances gallery tanks are not supplied for each position.

In the 211 Tank Farm the numbers are made up of S for all tanks, followed by the code letter for the chemical stored in the tank, and the last position of the engineering equipment piece number, E.P. 211-101 for Chemical A is therefore numbered SA-101.

In the 271 Building the prefixes M or H, for mixing and storage tank respectively, are followed by a number indicating floor and position.

Tank positions and numbers are given in Figures 54 and 59.

BUILDING 291 - FAN HOUSE AND PROCESS STACK

Building 291 includes exhaust fans and a stack for disposal of the various noxious fumes from 221 and 224 Buildings. The gaseous fission products can be safely disposed of to the atmosphere, provided they are sufficiently diluted. The off-gas from the dissolver is diluted with the ventilating air from the 221 Building at the base of the 200-foot stack and discharged to the atmosphere at the top. Approximately 65,000 cu.ft./min. of ventilating air is discharged to the stack. This gives a dilution which is sufficient in some cases to dilute the activity to tolerance levels. However, during the peak dissolving rate of the metal solution step, this is not the case and further dilution by natural convection into the atmosphere must be relied upon to obtain tolerance levels. This means that safe dilution cannot be obtained under certain weather conditions, as for example when strong down drafts exist in the vicinity of the ventilation stack. Hence, the operation of the dissolver depends, for disposal of the waste gases, upon proper weather conditions. Figures 2[illegible], and 68 show the layout of this equipment. The vent lines from the top of the dissolver condensers are run underground from the 221 Building to the ventilation stack. The off gases are transferred to the stack by means of steam jet ejectors located just before the stack. These jets also serve to keep the dissolver under a slight vacuum to prevent leakage of the radioactive gases into the equipment cells.

The ventilation air from the cells in 221 and 224 Buildings is exhausted through a buried duct from 221 Building by the ventilating fans located at the 291 Building. (See Figure 31) The dissolver off-gas is mixed with this air between the fans and the stack as shown in Figure 68, and from there is discharged at the top of the stack. Any drainage collecting in the base of the stack may be radioactive and is, therefore, sent to the 221 Building sewer system through a buried 24-inch earthenware line (See Figure 31).

As indicated in Figure 68 the 291 Building fan equipment includes one steam fan as well as two electric fans. The two electric fans each have a

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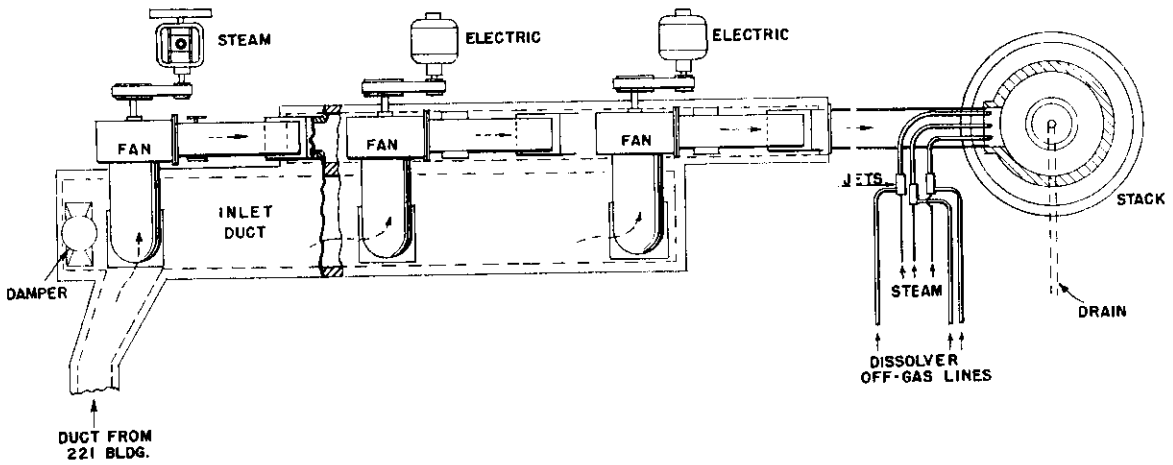
FIGURE 68

BUILDING NO.291



capacity of 32,500 cu.ft./min. or a total of 65,000 cu.ft./min. A damper arrangement in the inlet duct to 291 Building operates in a manner such that about one-fourth of the total air handled by the fans is fresh air from the outside. A vacuum control on the inlet duct automatically starts the steam fan should the vacuum within the duct drop below 2 inches of vacuum, which is the normal operating condition. Thus, the steam fan, which also has a capacity of about 30,000 cu.ft./min., acts as an emergency standby fan in the event of power or mechanical failure of the electric fans, thus assuring continuous exhaustion of the cells in 221 and 224 Buildings.

VENTILATION IN CANYON (221) BUILDING

The Canyon (221) Building contains two separate and distinct systems of ventilation. One system ventilates the process equipment areas, while the other ventilates the operating areas.

In the ventilation of the process equipment areas ten separate wet air washing units, each having a capacity of about 4500 cu.ft./min., feed fresh dust free air to the crane cabway and thus in turn to the Cell Deck Area. The air flows through slots in the cell block covers to the cells and pipe trench, and then through 10-inch diameter terra cotta ducts from each cell and each section of the pipe trench to the 48-inch by 72-inch concrete ventilating tunnel connected to the 291 Building. (See Figures 31, 69, and 70.) The static pressure in the crane cabway on the cell deck is adjusted (dependent upon the number of fans operated) to give about 0.1 inches of water vacuum and that in the cells proper about 0.3 inches of water vacuum.

Ventilation of the operating gallery is accomplished by feeding fresh dust free air from ten air filtering and washing units distributed along the operating gallery proper. Each of these units has a capacity of about 4500 cu.ft./min. Some air also flows from 271 Building into the operating gallery. From the operating gallery the air flows through gratings in the floor to the pipe gallery. The electrical gallery also receives about 5000 cu.ft./min. of fresh dust free air from the 271 Building ventilating system. Air is exhausted to the outside from the pipe gallery by nine exhaust fans and from the electrical gallery by three exhaust fans, one on each end of the gallery, and one in Section 1. The static pressure in the operating gallery and pipe gallery is about 0.02 inches above atmospheric pressure, while that in the electrical gallery is about 0.01 inches above atmospheric air pressure.

With the ventilating systems described above the flow of air is always away from the operating areas towards the outside or into the process area.

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FIGURE 69

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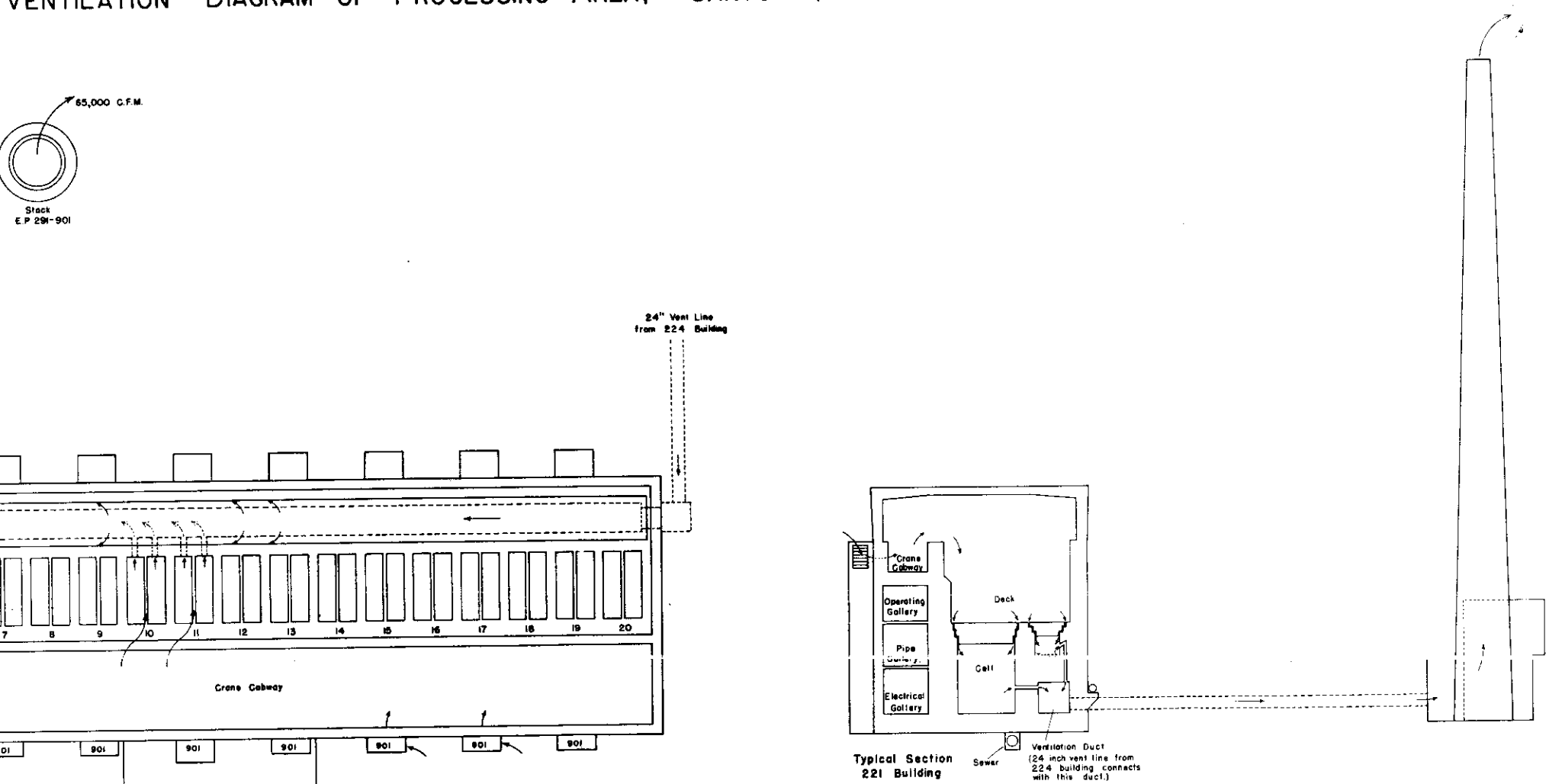
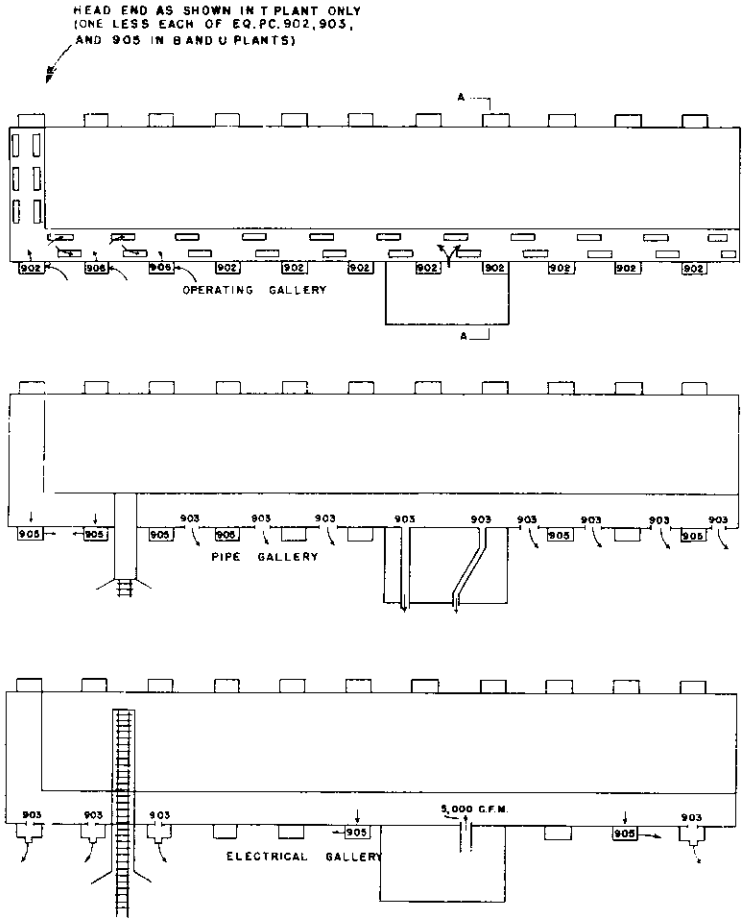
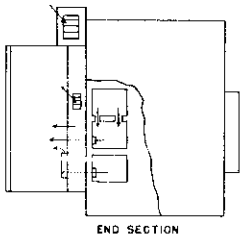
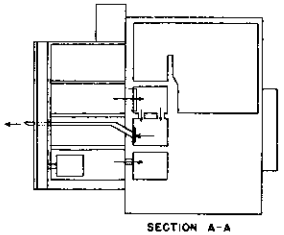
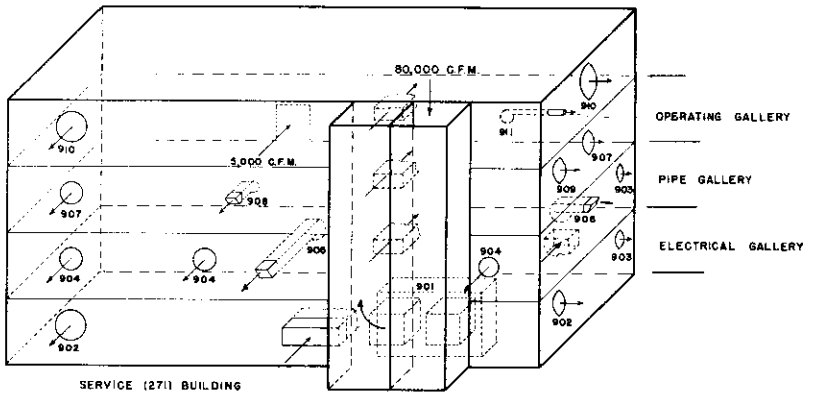
VENTILATION DIAGRAM OF PROCESSING AREA, CANYON (221) BUILDING.











LEGEND: 221 BUILDING.

E.P. 902 CARRIER HUMIDIFYING UNIT 4500 C.F.M.
E.P. 903 GALLERY EXHAUST FANS 4000 C.F.M.
E.P. 905 TOILET AND BATTERY ROOM FANS 1000 C.F.M.
E.P. 906 CARRIER HUMIDIFYING UNIT 4500 C.F.M.

REFERENCE DRAWINGS:

W 73347 EXHAUST FAN ARRANGEMENT 221
W 76165 UNIT HEATER ARRANGEMENT 221
W 71174 VENTILATION 271
W 71175 VENTILATION 271

LEGEND: 271 BUILDING.

E.P. 901 BUFFALO HUMIDIFYING UNIT 80,000 C.F.M.
E.P. 903 BATTERY ROOM FANS 1000 C.F.M.
E.P. 904 FIRST FLOOR VENTILATING FANS 6,500 C.F.M.
E.P. 905 SHOWER ROOM BLOWER 3,200 C.F.M.
E.P. 907 SECOND FLOOR VENTILATING FANS 3,500 C.F.M.
E.P. 908 REST ROOM BLOWER 1,900 C.F.M.
E.P. 909 LUNCH ROOM FAN 4500 C.F.M.
E.P. 910 SOLUTION ROOM FAN 10,000 C.F.M.
E.P. 911 HOOD BLOWER 800 C.F.M.

VENTILATION OF OPERATING AREAS
CANYON (221) AND SERVICE (271) BUILDINGS

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HW-10475-C

FIGURE 70

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VENTILATION OF SERVICE (271) BUILDING

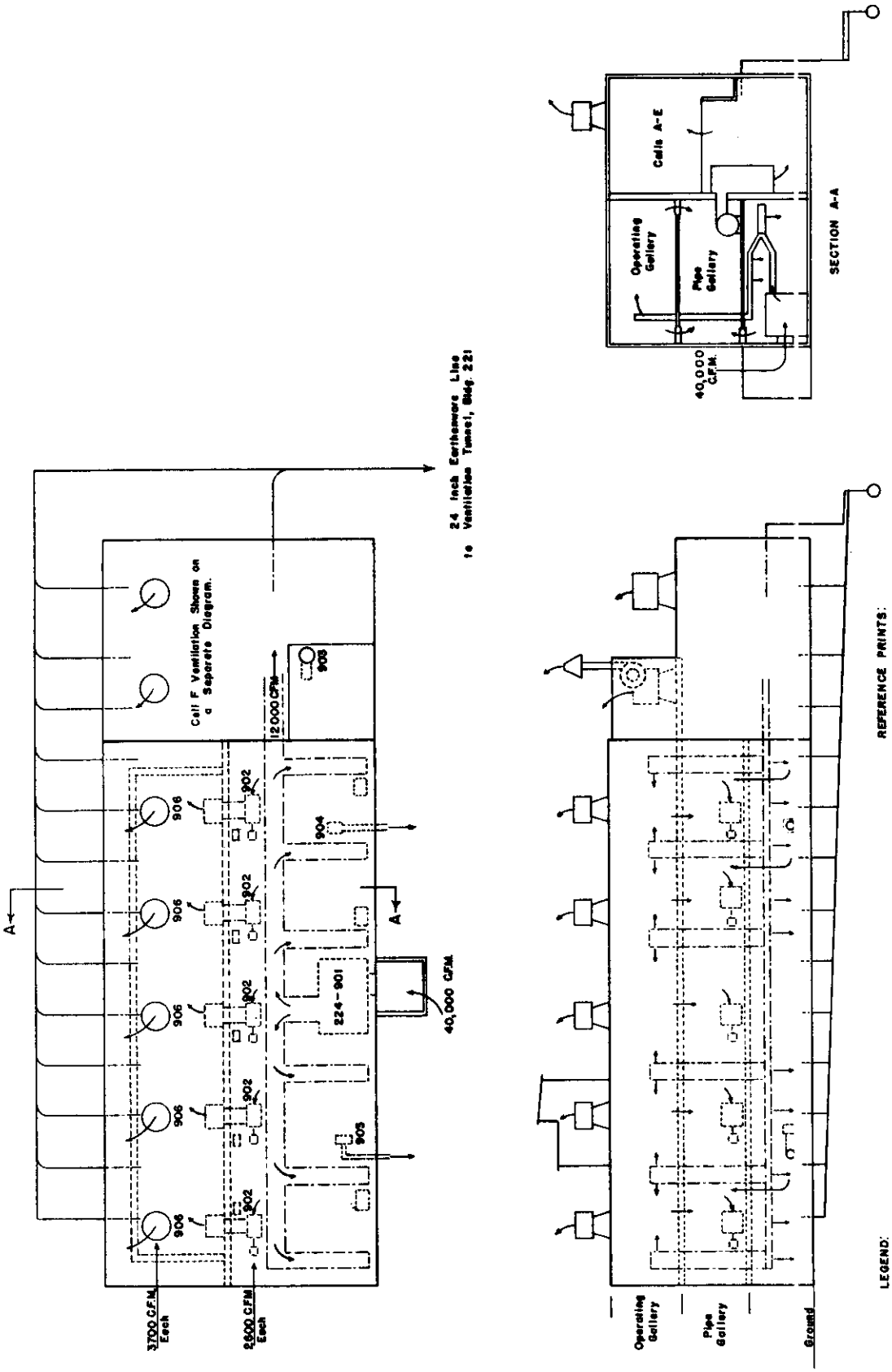
In the ventilation of the 271 Building fresh air from the outside is drawn in through a vertical duct reaching to the roof of the building (See Figure 70) into a central air filtering washing and conditioning unit. From this unit the air is fed through ducts to the four floors of the building and to the electrical gallery of 221 Building. Air is exhausted from the building through exhaust fans on each floor. The static pressure is maintained just slightly above that in the operating gallery of 221 Building, such that any flow of air would be from the 271 Building.

VENTILATION OF CONCENTRATION (224) BUILDING

Air is drawn into this building on the first floor through a filtering, washing and conditioning fan unit having a capacity of about 40,000 cu.ft./min. The air from this unit is fed through ducts to the first and third floor and flows through openings around the chemical headers to the second floor of the main section of this building. This unit also feeds air through ducts to Cell F which is separate from the main portion of this building. Air from the second floor (Pipe Gallery) of the main section of the building is fed by blowers into each cell (A to E inclusive) near the ground level and is exhausted to the roof in the cells proper by means of five ceiling fans each having a capacity of about 4,000 cu.ft./min. The static pressure is maintained at about 0.05 inches of water in the main operating areas of this building and about 0.2 inches of water below atmospheric in process portion of the Cells A to E. Hence, any leakage of air takes place from the operating areas to the cell areas. (See Figure 71 which indicates diagrammatically the air flow in the main portion of the 224 Building.)

Fresh air is fed to the F Cell through ducts from the main conditioning unit directly to the operating gallery, to one corner of the fee process equipment area, and to points in the vicinity of the F-10 cage. Air flows from the operating gallery and the area in the vicinity of F-10 cage through slots in the steel partitions separating them from the process equipment area and is exhausted from the process equipment section of the cell by means of two ceiling fans each having a capacity of about 8000 cu.ft./min. Air is also exhausted from the F-10 cage into a 24-inch earthenware line which is connected into the concrete ventilation tunnel of 224 Building. (See Figure 31) The process equipment portion of Cell F is maintained at approximately 0.2 inches of water vacuum. (See Figure 72 which indicates diagrammatically the air flow in Cell F.) All process vessels and centrifuges in the 224 Building are vented to the 24-inch earthenware line indicated above for venting F-10 cage. A vacuum of about 1 inch of water is maintained on this vent line resulting in a vacuum of about 0.5 inch of water on the individual equipment pieces.

FIGURE 71



VENTILATION DIAGRAM OF CONCENTRATION (224) BUILDING – EXCEPT CELL F

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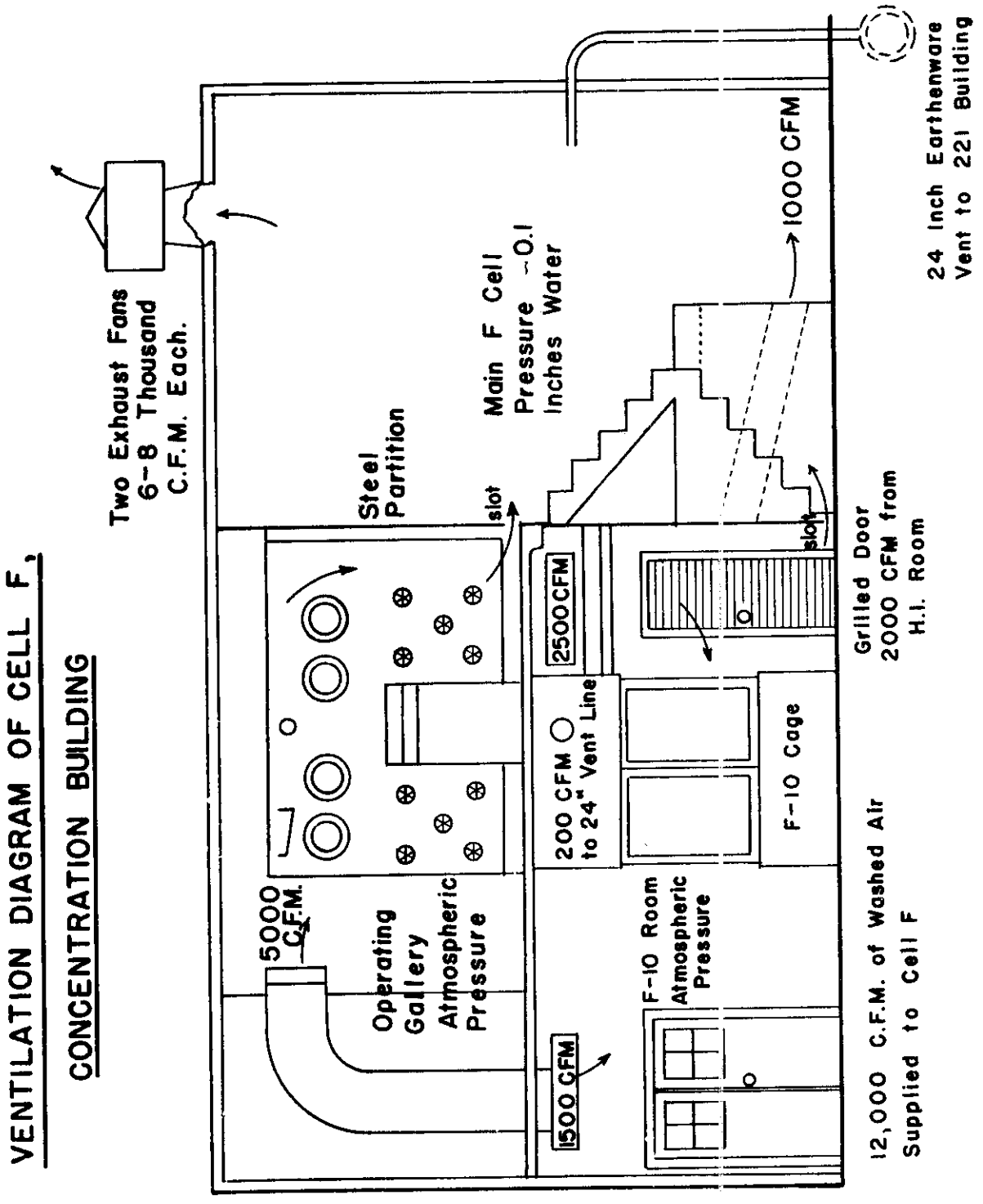
FIGURE 72



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BUILDING NO. 241 - WASTE DISPOSAL

Building No. 241 consists of a tank farm, retention pond and ditch, for the disposal of process waste solutions from the 221 Building, and a settling tank, and reverse flow well, and buried sump, for use in disposal of the process waste solutions from the 224 Building.

Tank Farm

The general layout of the tank farm is illustrated in Figure 73. This area as well as the other facilities are described in greater detail in Chapter IX.

Active process wastes from 221 Building are brought into the tank farm area in buried stainless steel lines terminating in diversion boxes. By altering the connections in a diversion box, the flow of waste is changed from one storage tank to another as desired. The diversion boxes contain pipe connections equipped with the same remote maintenance connectors and flanges as provided in the Canyon (221) Building. A portable impact wrench control assembly is provided for use in tightening or loosening the connectors. From the diversion boxes buried stainless steel lines run to the storage tank which are constructed of steel set in concrete. As shown in Figure 73 the tanks are buried in order to eliminate radiation hazards and are arranged in cascade in groups of three. The cascade arrangement allows the suspended solids containing the bulk of the radioactivity to collect in the first tank of each series. It may be possible with the second cycle waste solutions to eventually empty the tanks used for its storage when the activity has decayed to a safe level, thus increasing waste disposal capacity.

All process wastes from 221 Building are stored in these tanks. The most active wastes (metal solution and first by-product precipitate) eventually may heat up to boiling. Tanks for the metal wastes are equipped with air cooled reflux condensers so that they may not boil dry. A second series of tanks have outlets provided on which condensers may be installed. The remainder of the tanks have a number of outlets which could be connected to condensers if this should prove desirable. A series of 150-foot dry wells are provided in the vicinity of the storage tanks to permit detection of any leakage from the storage tanks.

Retention Pond and Ditch

Two retention ponds are provided which are in parallel and which overflow to a ditch. The innocuous cooling water from both 221 and 224 Buildings is piped to these ponds which are regularly monitored to prevent disposal of active wastes above ground.

[redacted]

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FIGURE 73

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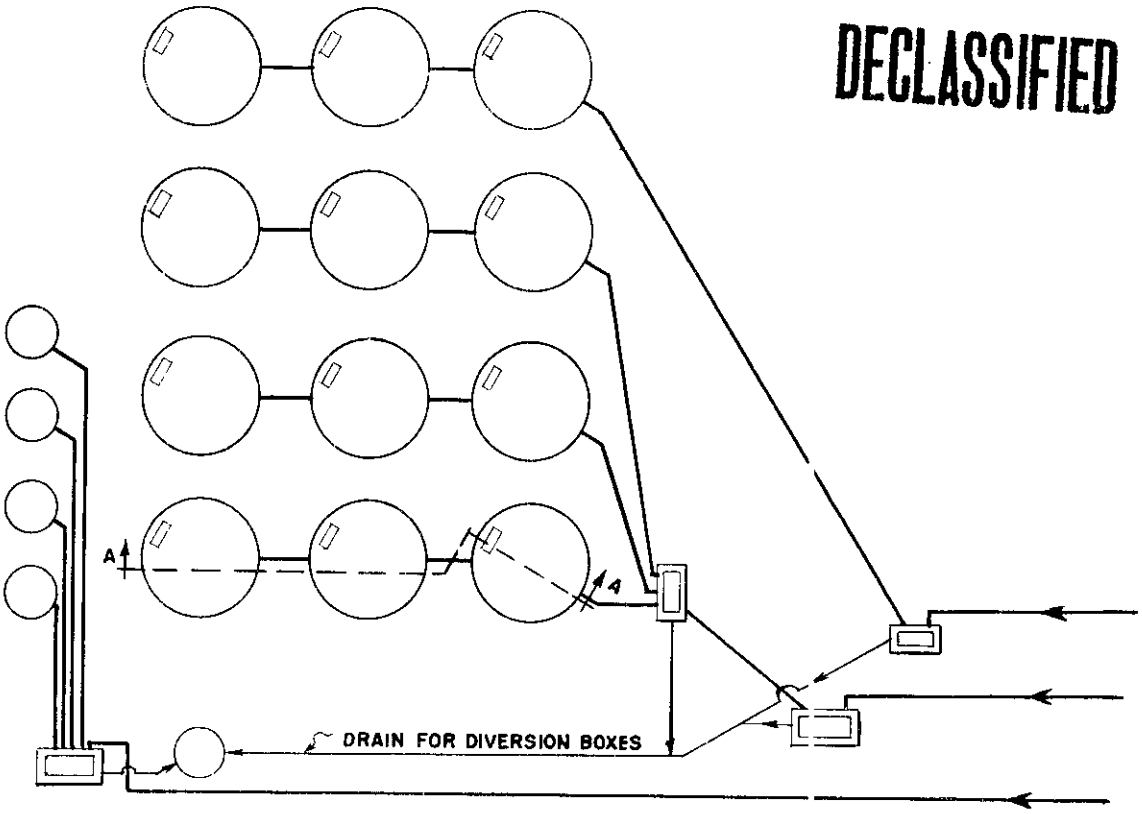

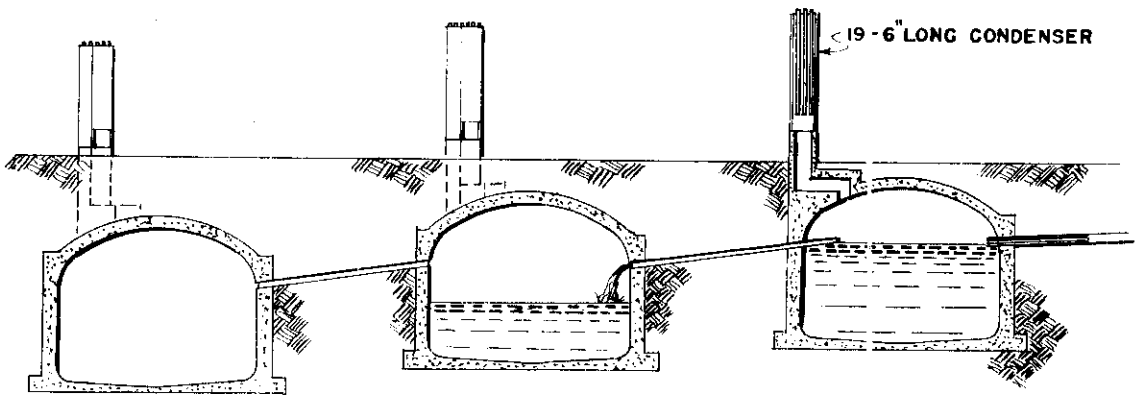

SECTION ON A-A OF 75'-0" DIA. STORAGE TANKS

241 BUILDING

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SETTLING TANK NO. 241-361, REVERSE FLOW WELL, AND BURIED SUMP

A settling tank and reverse flow well illustrated diagrammatically in Figure 74 is provided for disposal of low activity wastes from 224 Building and wastes containing only a trace of activity from Cell 5 of 221 Building. The settling tank is constructed from gunnite type concrete and overflows to the dry well. The major portion of the radioactivity in the wastes from 224 Building settles out in this settling tank and the supernatant overflows into the reverse flow well which ends above the water table. The Figure 75 illustrates the construction of a typical reverse flow well.

It was found in actual operation that the reverse flow wells were filling up with sand, which apparently flowed into the well casing through the perforations in its lower end. As a substitute for these wells buried sumps were constructed consisting of a reinforced open bottom 12-foot by 12-foot wooden crib 4 feet high buried in gravel 18 to 19 feet below the grade surface. These cribs are connected to the 241-361 settling tank by means of a 3-inch stainless steel pipe. Figure 76 illustrates diagrammatically the construction of a buried sump.

ISOLATION (231) BUILDING

General Facilities

This building is used for the isolation of the product as the nitrate from the carrier used in the Concentration (224) Building, and evaporation of its solutions to a viscous or semi-rigid cake for shipment.

A general layout of the area immediately adjacent to the 231 Building is illustrated in Figures 29 and 77. Figures 78 and 79 give the floor plan and Figure 80 shows the flow of ventilating air in this building. This building, as indicated by the floor plan, is self contained with regard to facilities for product isolation and for process chemical solution preparation, process control and research laboratories, materials storeroom, minor maintenance shop facilities, and equipment for ventilation, and waste disposal. Figure 83 is a view of one of the research laboratories.

Isolation of the product takes place in Cells 1 to 5 and the final evaporation of the solution in Cell 6-A. Figure 81 is a view of one of the isolation cells. The equipment in the isolation cells is described in more detail in Chapter VIII. Figure 82 illustrates diagrammatically the equipment for evaporation of the final product solution prior to shipment, and Figure 84 the Sample Can (container for product solution) in its shipping container.

Process solutions for recycle are returned to the 221 Building in either the transfer can (F-1) illustrated in Figure 67, or in a tank truck.

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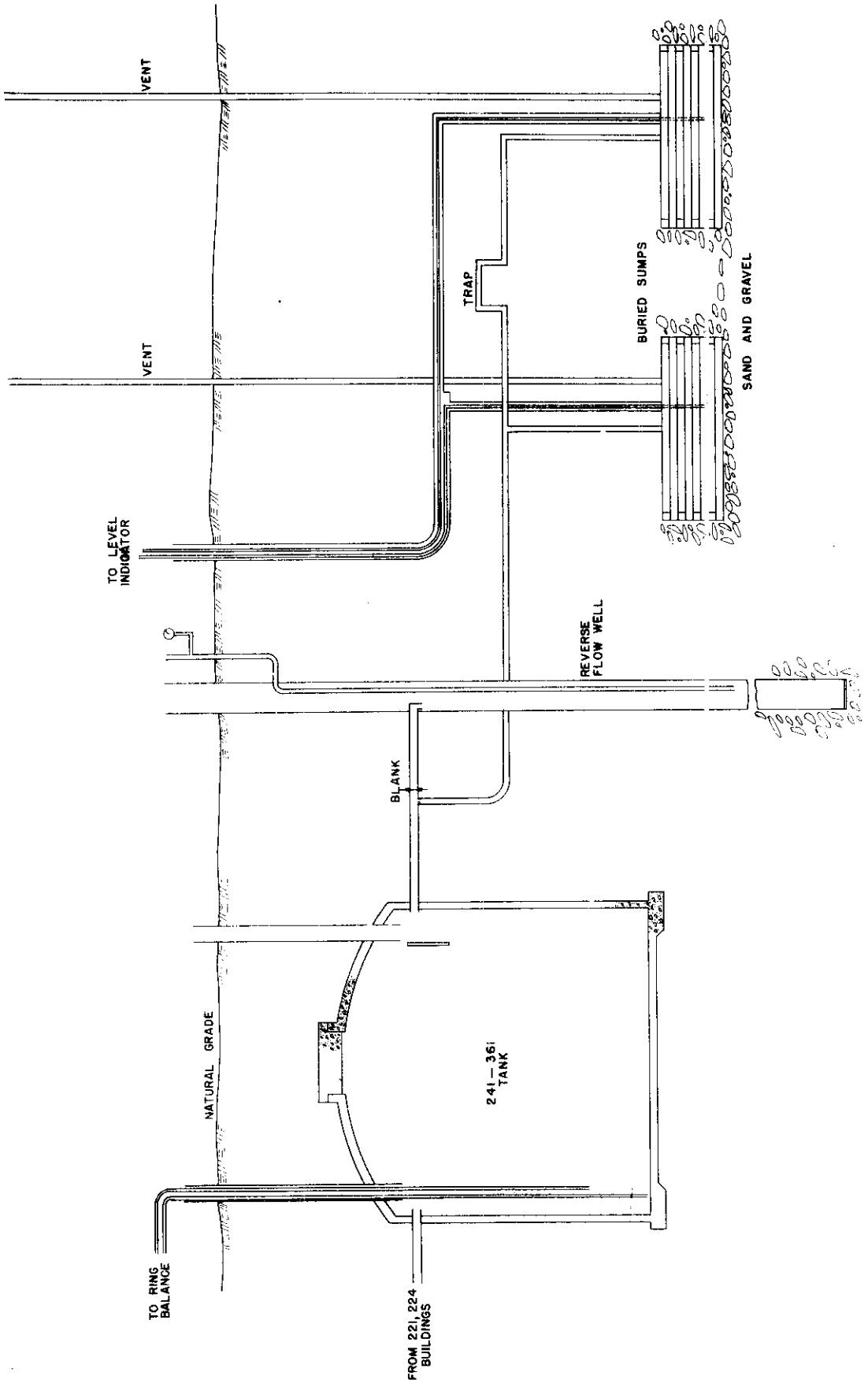
FIGURE 74



DIAGRAMMATIC REPRESENTATION OF 241-361 SETTLING TANK, REVERSE FLOW WELL, AND BURIED SUMPS

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FIGURE 75

241-T, 361-A REVERSE FLOW WELL.

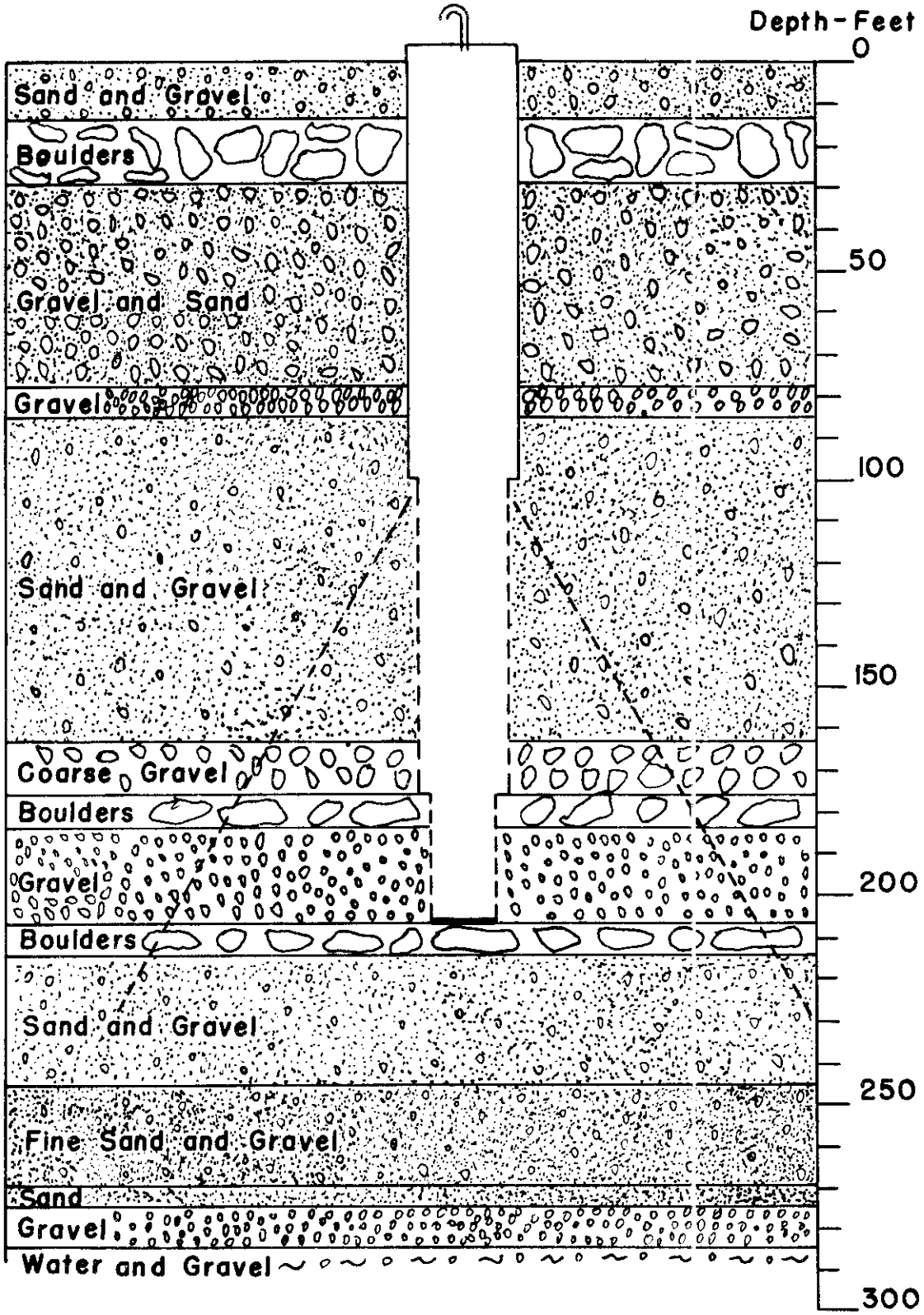

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FIGURE 76

DIAGRAMATIC REPRESENTATION OF BURIED SUMP

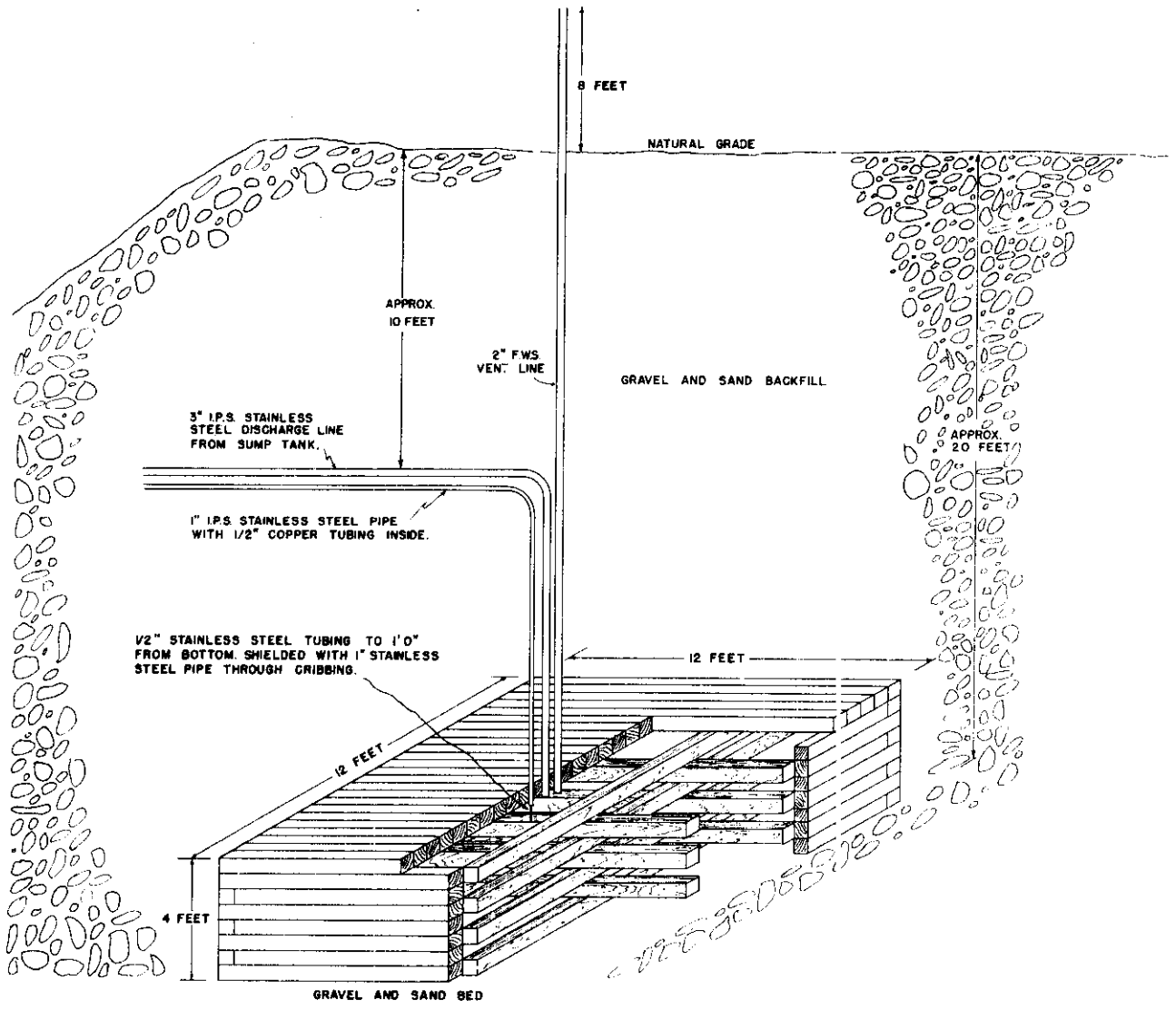

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FIGURE 77

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OUTSIDE WASTE LINE MAP FOR ISOLATION BUILDING

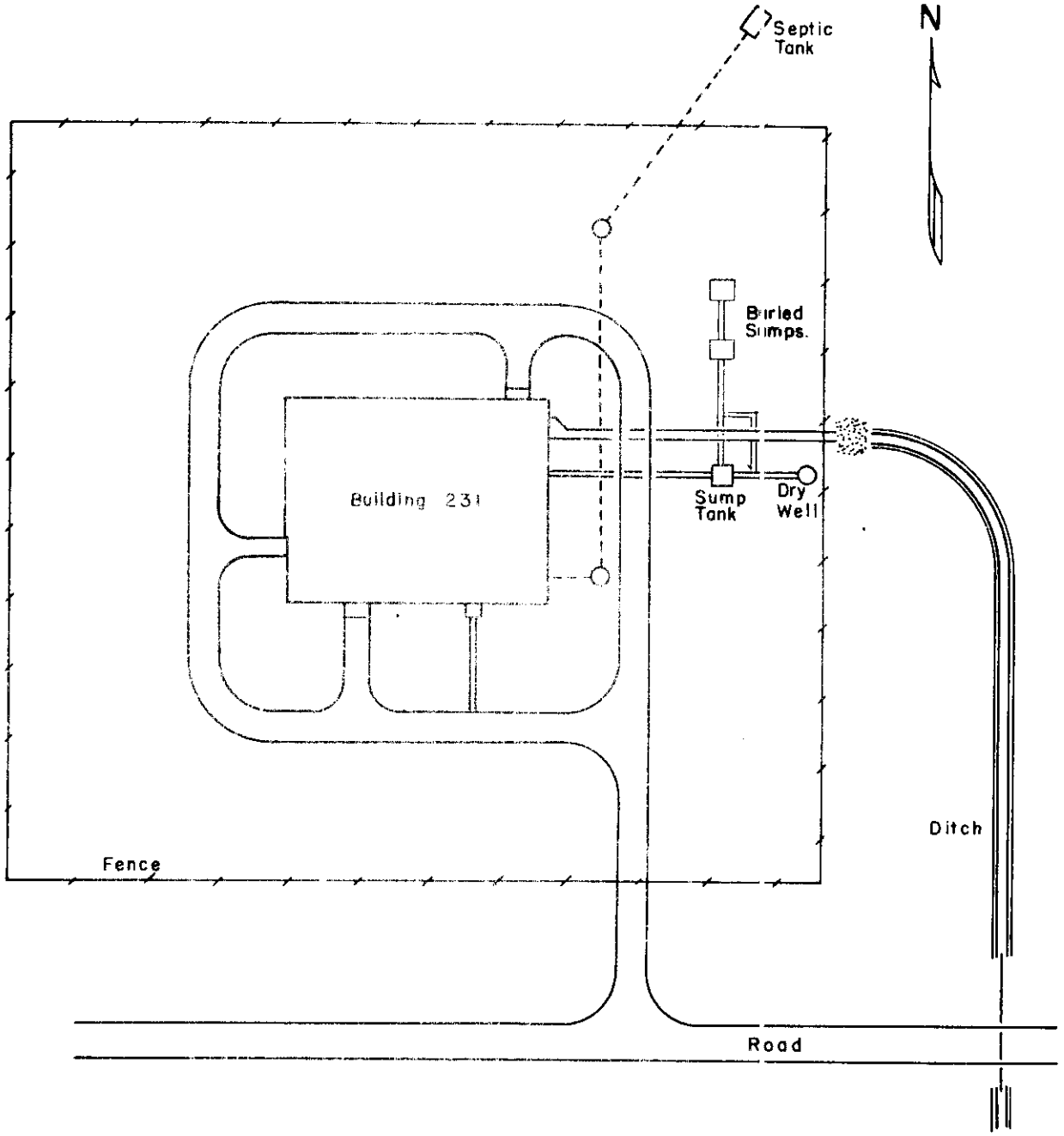

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FIGURE 78

FIRST FLOOR ISOLATION BUILDING

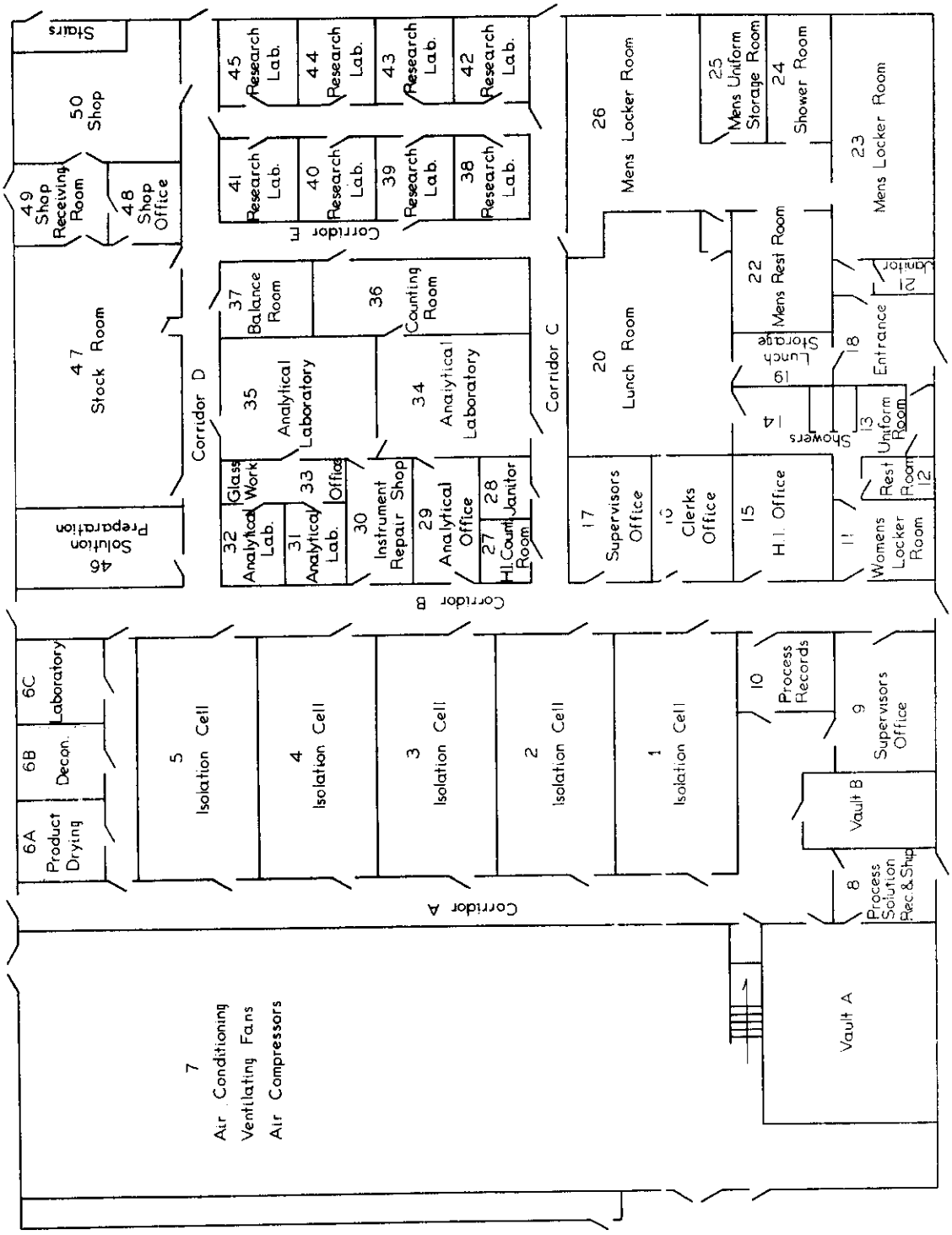
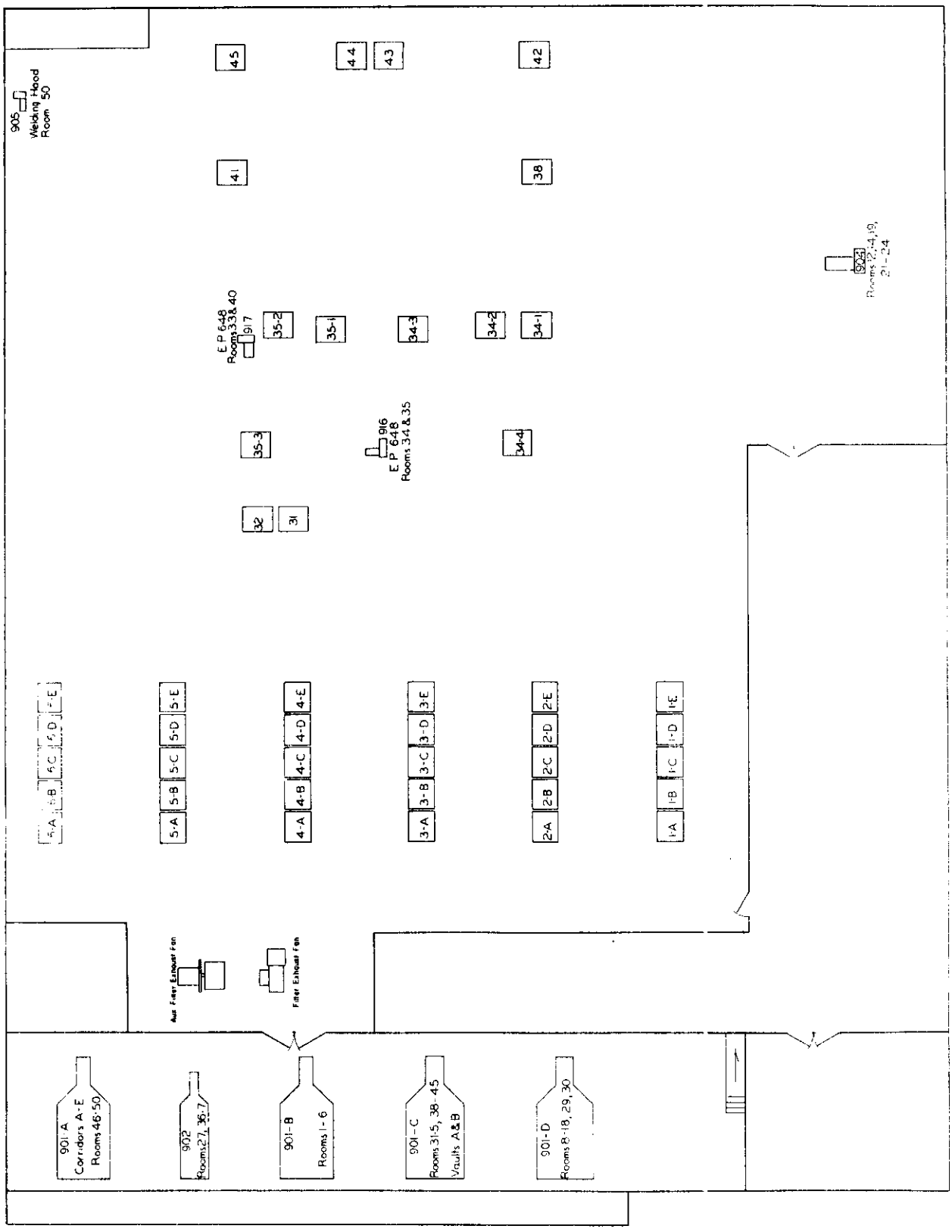


FIGURE 79



SECOND FLOOR ISOLATION (231) BUILDING

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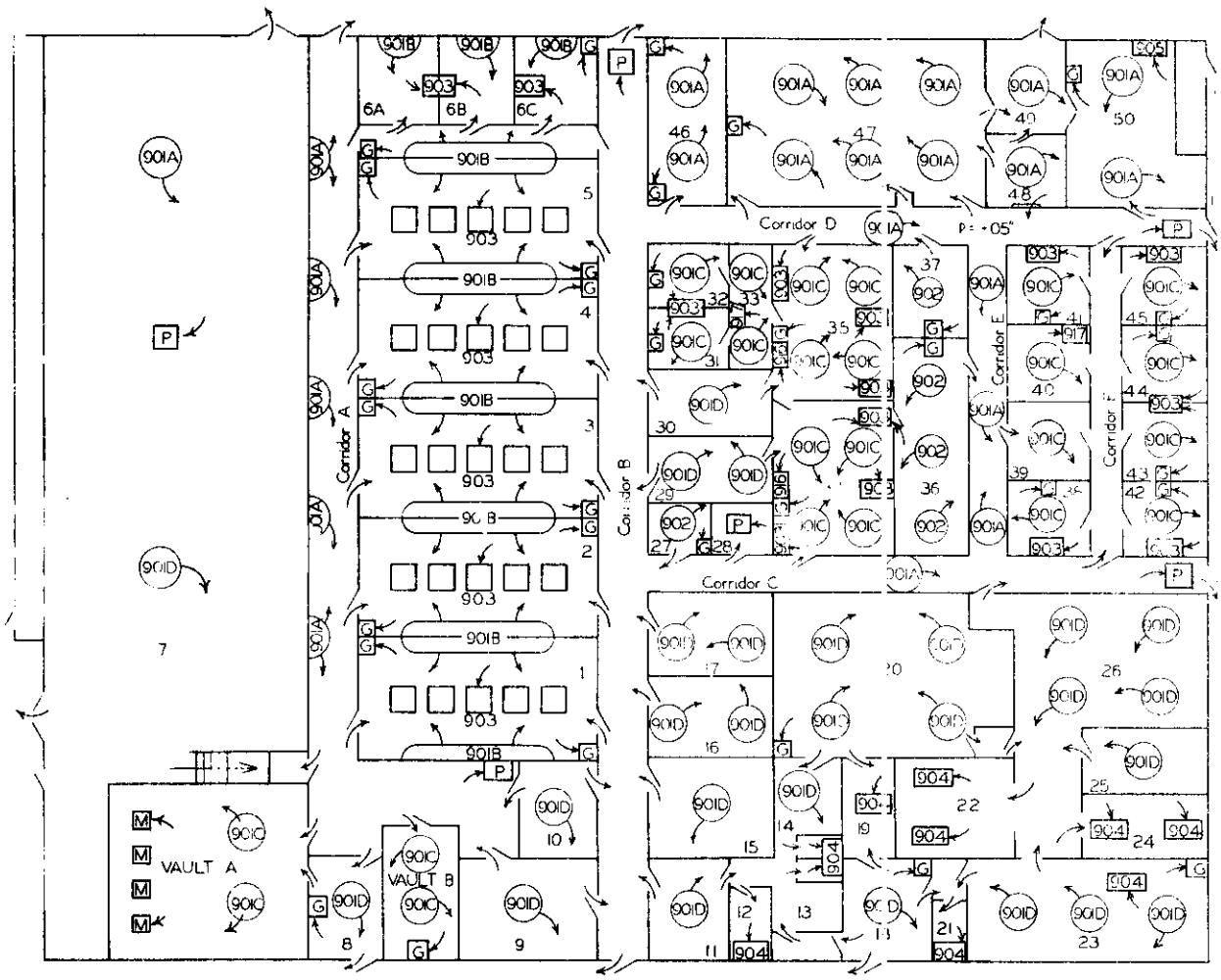
FIGURE 80

VENTILATING DIAGRAM OF OPERATING AREAS ISOLATION BUILDING



Supply Duct Numbers Indicate Fan Numbers

Exhaust Duct Numbers Indicate Fan Numbers

G – Gravity Operated Dampers

M – Manually Operated Dampers

P – Pneumatically Operated Dampers

FIGURE 81

CELL 1 - ISOLATION BUILDING

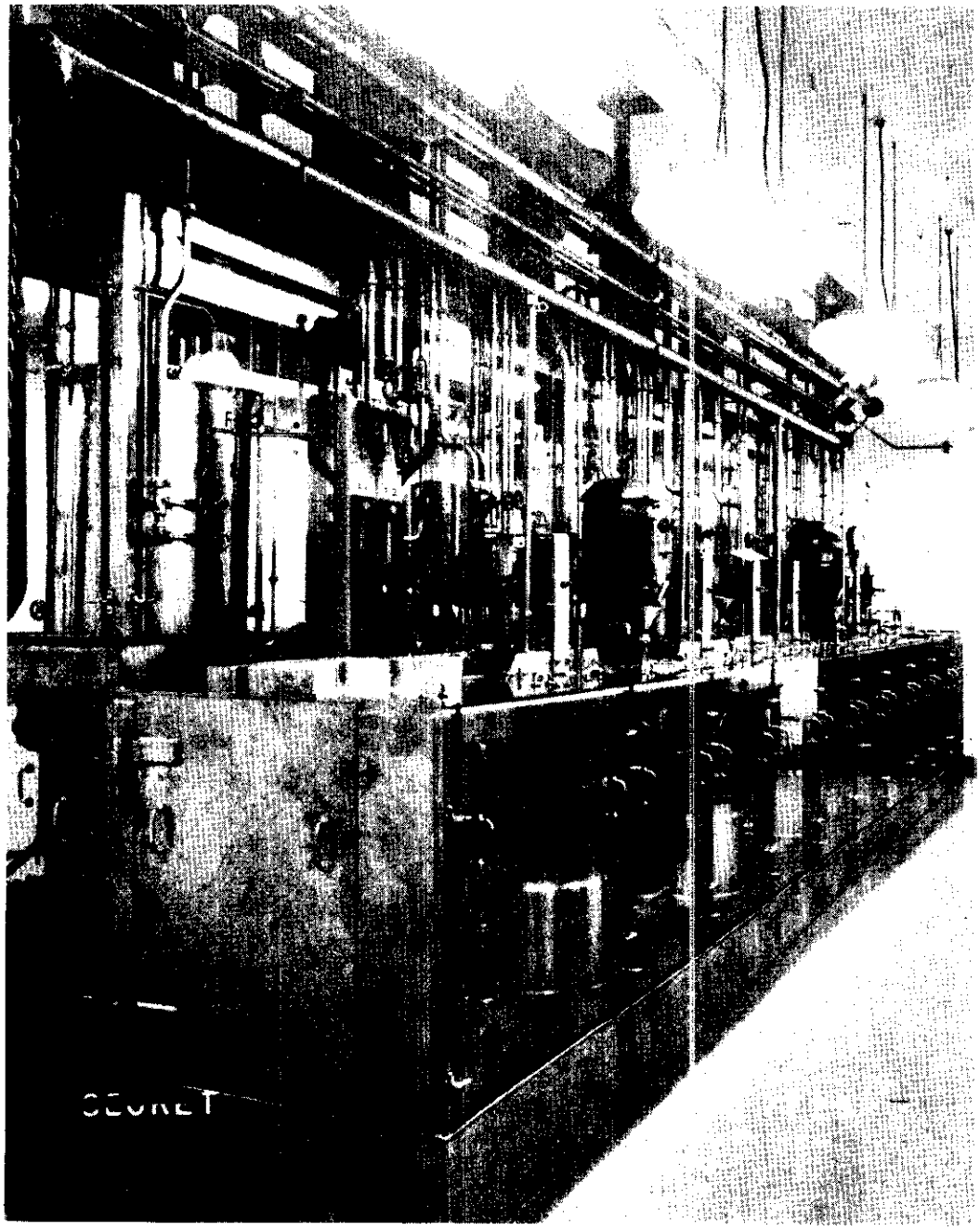


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FIGURE 82

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AGRAM OF EQUIPMENT FOR FINAL PRODUCT SOLUTION CONCENTRATION

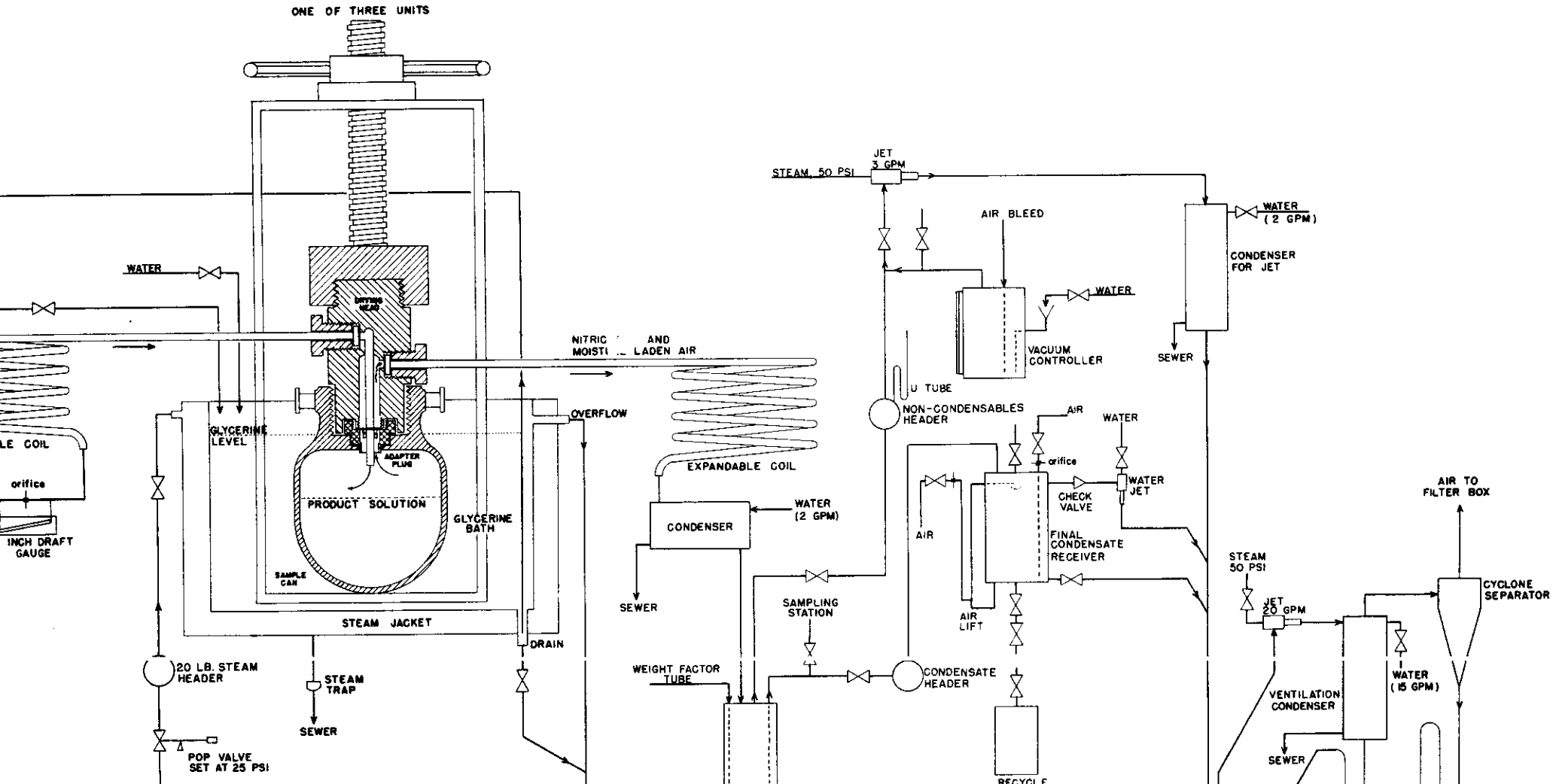

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FIGURE 83

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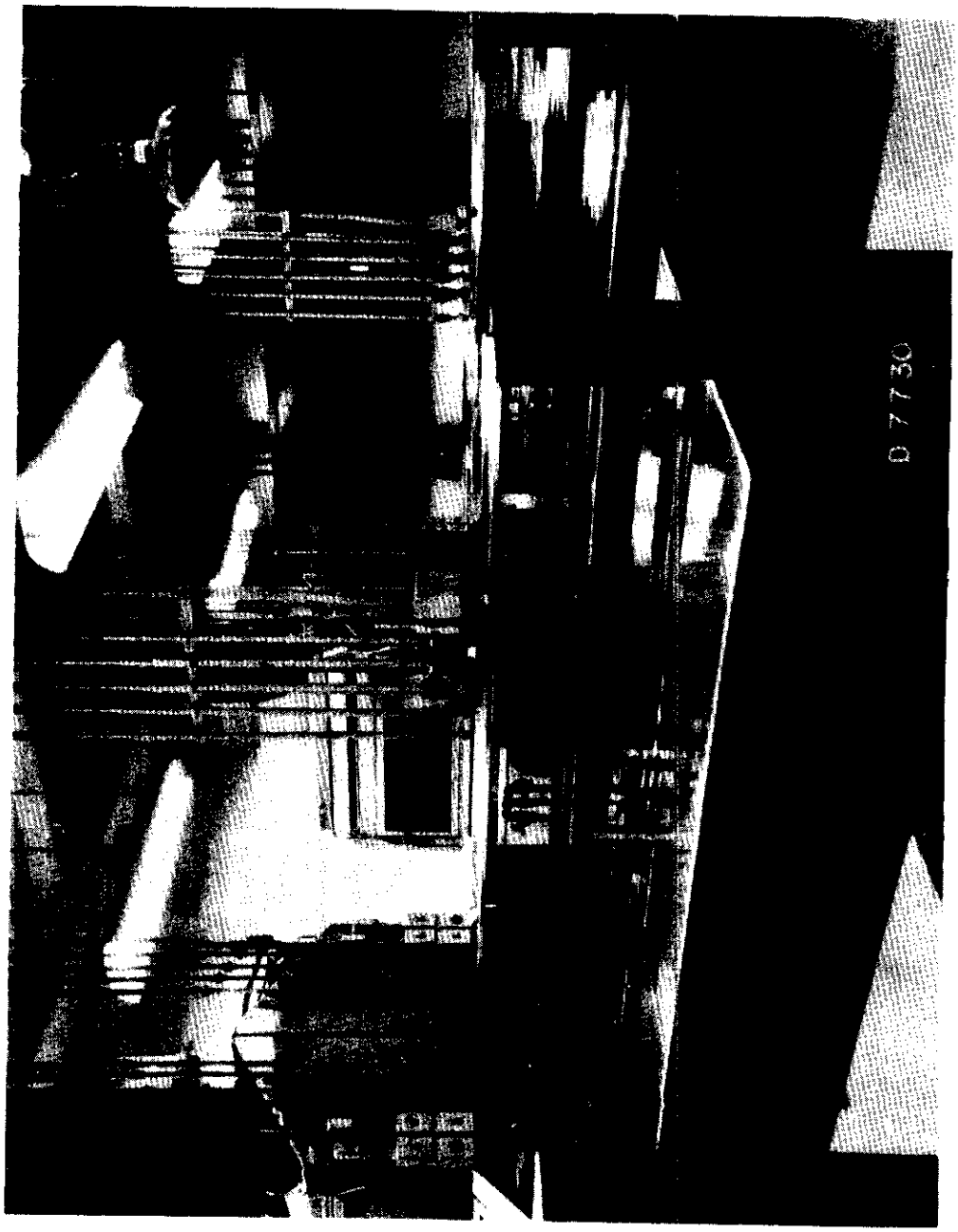

A RESEARCH LABORATORY IN THE ISOLATION BUILDING

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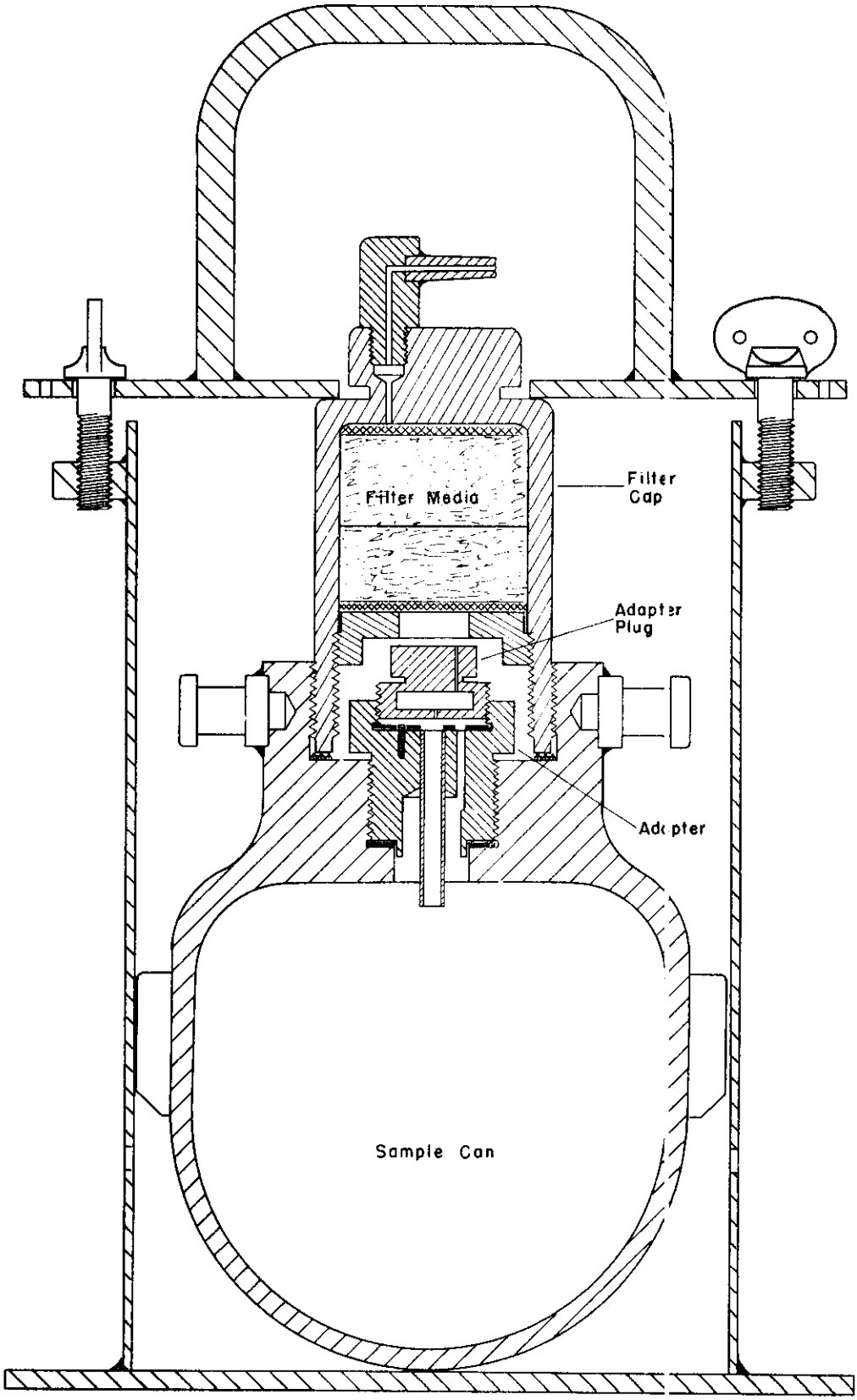
FIGURE 84

SAMPLE CAN AND CASE ASSEMBLY



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WASTE DISPOSAL FROM THE ISOLATION BUILDING

Waste solutions from this building are disposed of through one of three systems, a tile field for sanitary waste, an open ditch for the non-active process and laboratory wastes, and a stainless steel line and settling tank connected to a reverse flow well and buried sump for active process and laboratory wastes (illustrated in Figure 85). After several months of operation the reverse flow wells partially filled up with sand. For this reason buried sumps were constructed consisting of an inverted reinforced wooden box buried in gravel 18 to 20 feet below the grade surface. A feed line is connected to the settling tank and a vent provided to prevent air binding. (See Figure 86 for a diagram of waste lines in 231 Building and Figure 77 for a map of outside waste lines from this Building.)

VENTILATION OF 231 BUILDING

Because of the extremely toxic nature of the product and the fact that its solutions in high concentration are handled in this building special care has been given to the ventilation of this building and to the separation of product from the air exhausted from it. Figure 80 shows the flow of ventilating air throughout the 231 Building and indicates the fan feeding each room.

By means of solid doors, which are intended to be kept closed, this building is divided into four main separately ventilated areas itemized in Table II.

All laboratory and process hoods in this building are connected to a special filter box on the exhaust duct leading from each hood. These filter boxes illustrated diagrammatically in Figure 87 are packed with special fine filament rock wool pads which filter out any product or fine dust on which may be deposited product from the exhaust air. Four layers of this special rock wool filter medium, total thickness of 4 inches, are used in each filter box and each layer is sealed at its edges with a special adhesive to prevent the air being filtered from by-passing the filter medium. When the filter boxes were installed in this building (See Figure 88 for a view of the installation) each one was tested in order to insure that it would not by-pass more than 0.25-0.50% of a methylene blue smoke made up of particles within the range of 2-3 microns in diameter. Tests on actual process ventilating air showed all but 0.3% of the product content of air to be removed.

The above filter boxes are removable and extra filter boxes are on hand so that should one become contaminated to the point that the exit may be considered unsafe for breathing, it may be replaced by a new one, and the old one buried. The exhaust air from these filter boxes is discharged to the atmosphere through a 50-foot stack on the roof of the building from the 15,900 cu.ft./min. exhaust fan (Equipment piece No. 903). A standby

FIGURE 85

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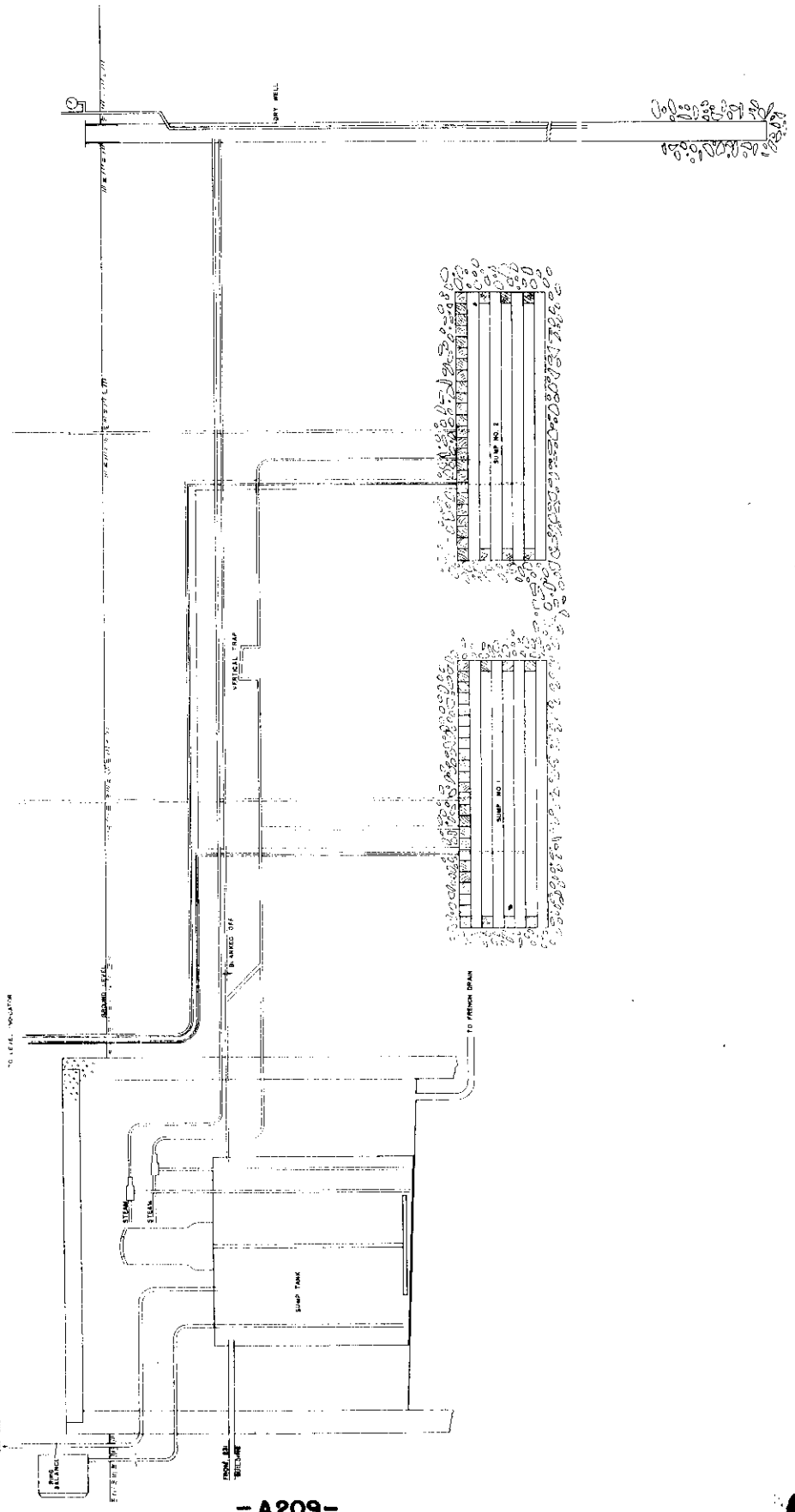

ACTIVE WASTE DISPOSAL SYSTEM

ISOLATION BUILDING

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FIGURE 86

SEWER LINE DIAGRAM OF ISOLATION BUILDING

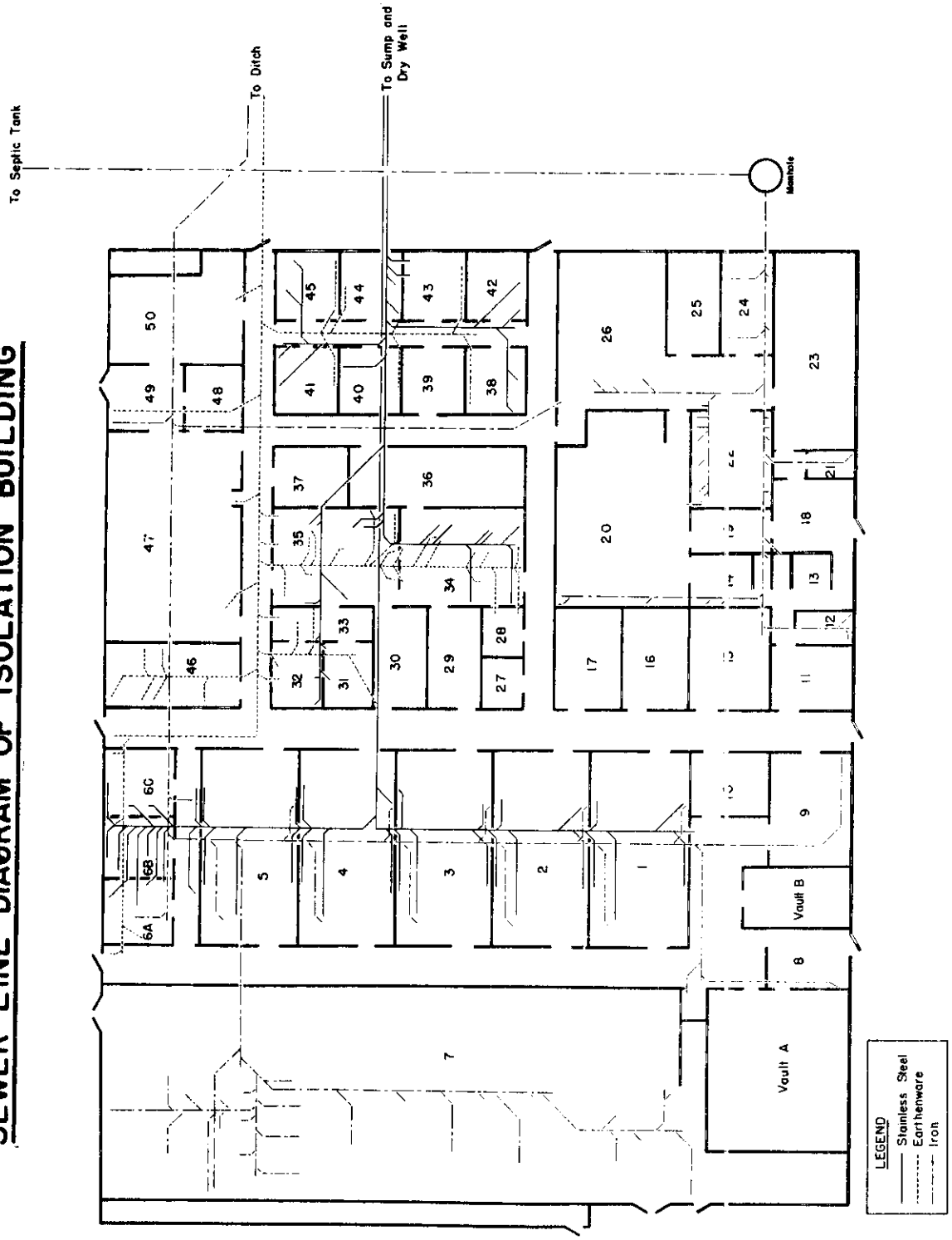

TABLE II

VENTILATING SYSTEM IN ISOLATION (231) BUILDING

Area	Rooms	Supply Fan - Capacity	Exhaust Method
Living Area	Corridors A - E inc.	901-A(17,700 cu.ft./min.)	Pneumatic roof damper opens at 0.01 inch water pressure
Living Area	Room 7 and Second Floor	901-A and D	" " " "
Living Area	Rooms 8-18 inc., 29 and 30	901-D(16,400 cu.ft./min.)	To corridor A or D etc. and fan 904
Living Area	Rooms 19, 20, 22 - 26 inc.	901-D	Fan 904 and pneumatic roof damper
Living Area	Rooms 46 - 50 inc.	901-A	Fan 905 and pneumatic roof damper
Plant Area	Cells No. 1 - 6 inc.	901-B(13,200 cu.ft./min.)	Automatic damper to roof and Fan 903 to hoods set for -0.10 inch water
Instrument Area	Rooms 27, 36 and 37	902-C(13,500 cu.ft./min.)	902 exhaust to air shaft, rooms set to be above + 0.12 inches water
Laboratory Area	Rooms 31 - 35 inc. Rooms 38 - 45 inc. Vaults A and B	901-C(14,700 cu.ft./min.)	Automatic dampers to 903 fan (capacity 15,900 cu.ft./min.) set to maintain suction of 0.10 inch water

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FIGURE 87

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ISOLATION (231) BUILDING EXHAUST AIR FILTER

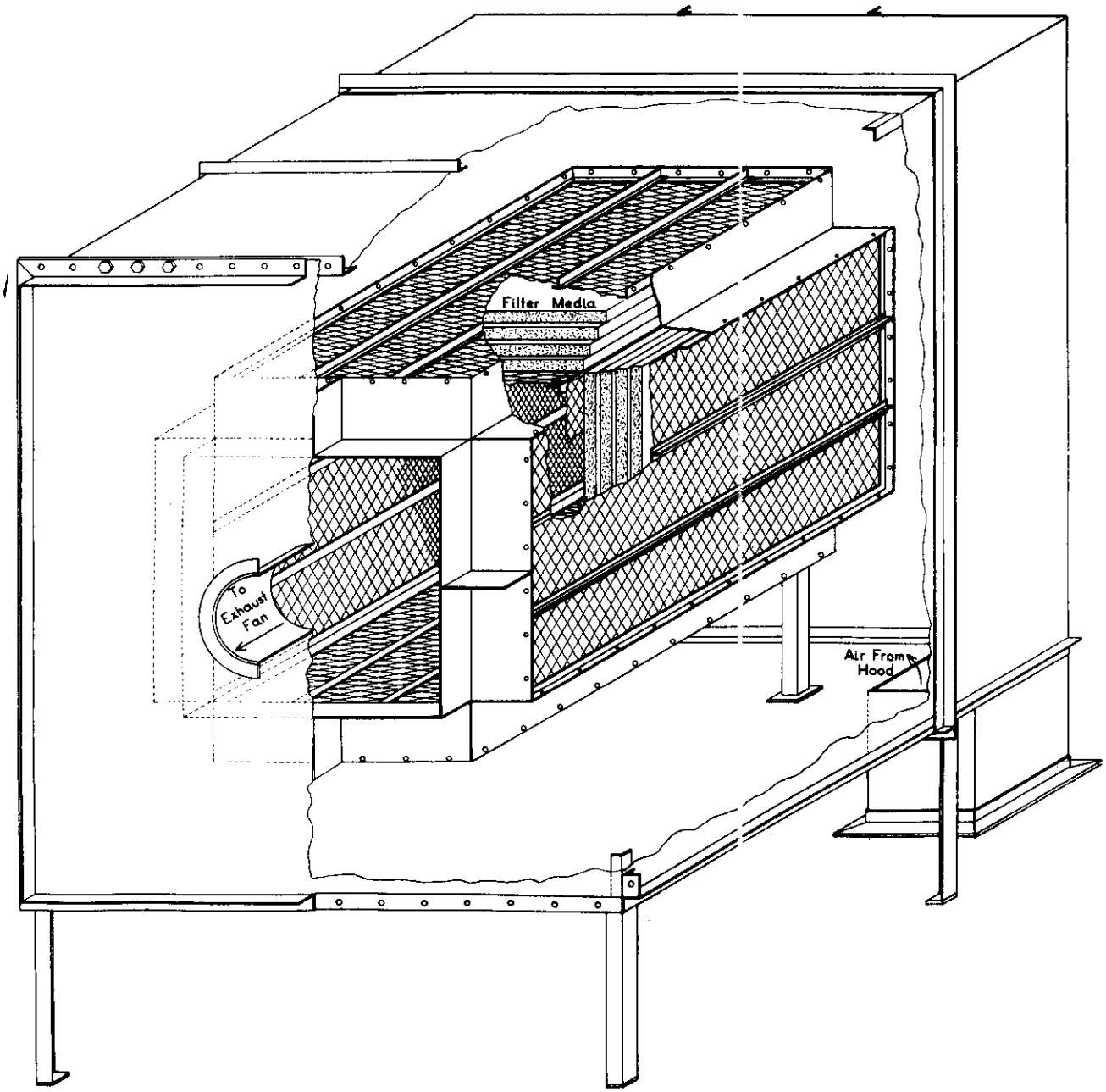

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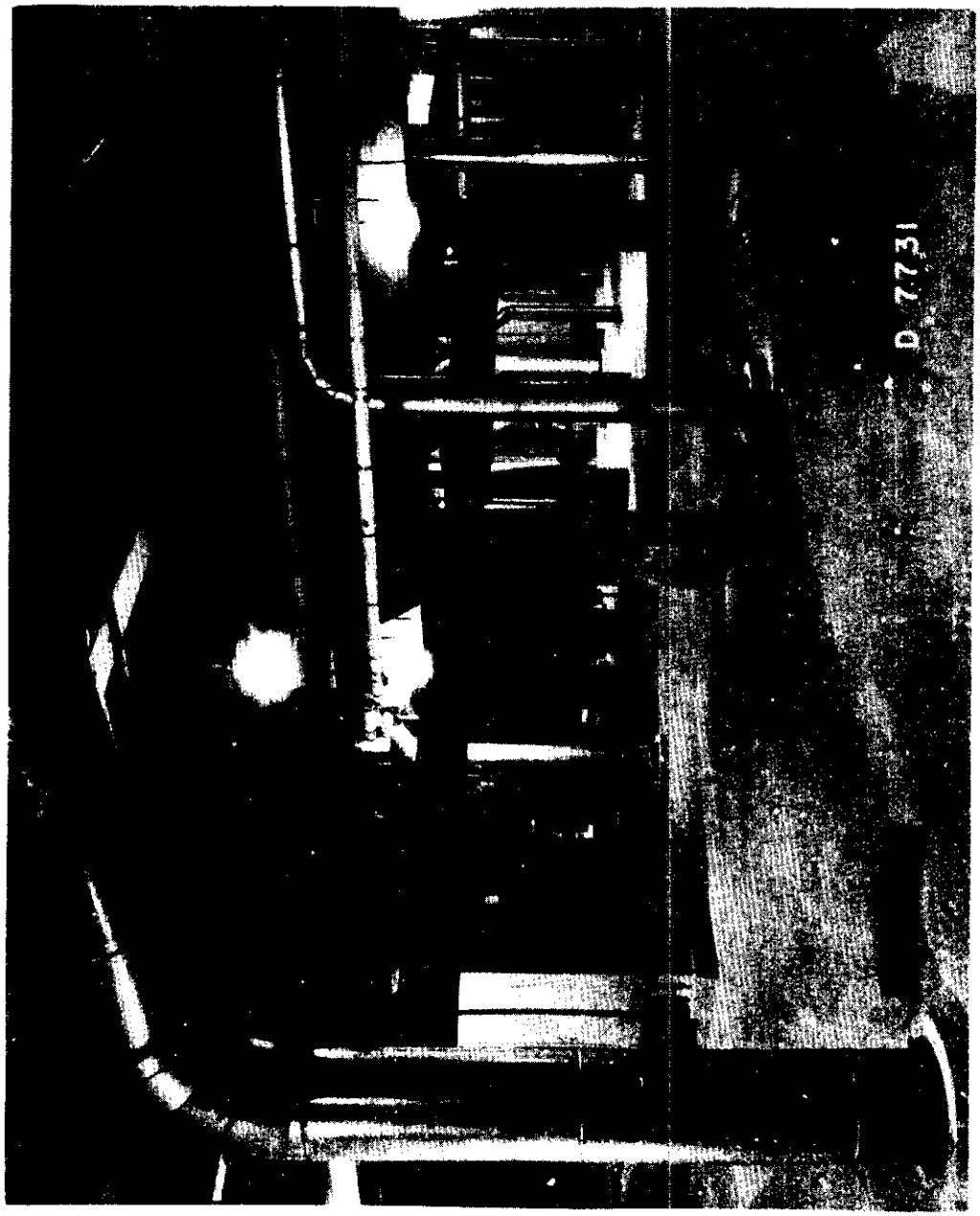
FIGURE 88

SECOND FLOOR - ISOLATION BUILDING



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steam engine driven exhaust fan is also provided in parallel with this exhaust fan for emergency use. Vacuum on this system is maintained at 6 inches of water by an automatic damper, and on each filter box at 1 inch of water by individual automatic dampers.

In order to avoid dust, all persons entering this building are required to wear clean canvas covers on their shoes or change to shoes which are always kept within this building. All persons entering processing areas are also required to change to clean coveralls or wear clean smocks in order to avoid contamination of their clothing by product.

METEOROLOGICAL OBSERVATION (622) BUILDING

General Facilities

Near the 200 West Area (See Figure 1) is located a 408-foot meteorological observation tower, together with an attendant building housing equipment for the automatic measurement and recording of wind direction, wind velocity, relative humidities and air temperatures. This equipment is known as 622 Building - 200 West Area. (For a view of this tower see Figure 89.)

This equipment is provided for the purpose of securing information (a) for the use of the operating personnel in the 200 areas in scheduling their dissolve operations and estimating the dilution factor to be expected on stack gases, (b) to provide the electrical department with forecasts regarding high winds (wind velocities more than 25 miles/hour at 50 feet above the surface), and (c) to be prepared at all times to issue a forecast of the wind direction and wind velocities over the northern half of the reservation in the event of an emergency disturbance at any of the 100 or 200 Areas.

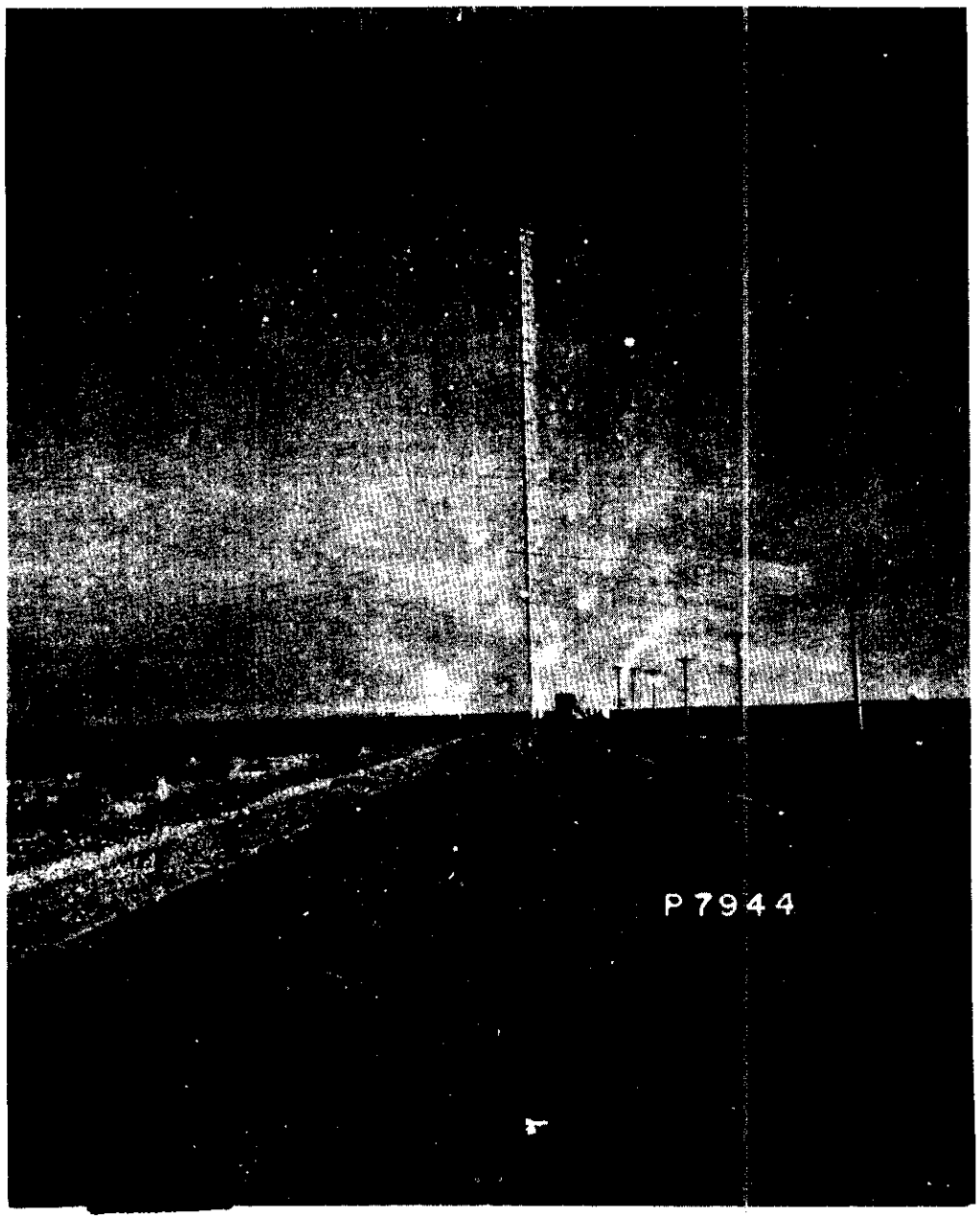
Since it was expected that under certain conditions the stack gases would be ejected to considerable heights above the stack top (200 feet high), a 408-foot tower was constructed to permit the various instruments to be exposed to a height which would approximate the elevation to which the stack gases would possibly be ejected. Furthermore, the tower, which is triangular in cross-section, was oriented so that the instrument booms, which extend perpendicular to the tower, are pointed northwest, that being the prevailing wind direction. The tower is constructed from painted structural steel angles bolted together. A steel safety ladder is provided, reaching to the top of the tower with landings approximately every 16-18 ft. apart. A small 75-lb. capacity elevator is provided for maintenance purposes.

Air Temperature Measuring Equipment

Air temperature measurements are made at every 50 foot elevation, using Leeds and Northrup nickel resistance thermometers, which are connected to an

FIGURE 89

METEOROLOGICAL TOWER (622 BUILDING



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eight-point Micromax recorder. Identical thermometers measure the air temperature at 4 feet above ground level, and the soil temperature 0.5 inch below ground level, which are connected to a separate two-point Micromax recorder. The temperature indicated by the soil thermometer is in proportion to the amount of solar radiation being received at the ground surface, which in turn affects the stability of the air, i.e. the "lapse rate". All of the resistance thermometers except the soil thermometer are shielded from the direct rays of the sun and from ground radiation of heat. In order to insure true air temperature measurements the thermometers used for measurement of air temperatures are aspirated by means of a 200 cubic foot per minute capacity exhauster. This rate of aspiration is considered rapid enough to nullify the effects of solar heating, heat reflection, or re-radiation from the ground.

Wind Direction and Velocity Measuring Instruments

Gurley electronic wind velocity anemometers and wind directive transmitters are located at 50-foot and 100-foot intervals on the tower, which are connected to Esterline-Angus recorders. The wind velocity transmitters and recorders are designed for an accuracy within 2% of full scale (100 miles/hour), and the direction transmitters and receivers are designed for an accuracy within 2° of the actual (true) direction. (In order to obtain information as to the wind direction and velocity conditions in the 100 Areas, wind direction and velocity units were erected on the patrol area headquarters buildings in each of the 100 Areas. The radio operators telephone the 622 Building each hour on the half-hour to report the current wind direction and velocity.)

Humidity Measuring Instruments

Bristol humidity measuring instruments and transmitters are located at 4 feet, 100 feet, 200 feet, 300 feet and 400 feet above the ground surface on the tower, which are connected to recorders in the adjacent building. These instruments are designed for an accuracy of 3%.

The above instruments provide facilities for the determination of:-

1) The vertical air temperature gradient (commonly called the "lapse rate").

2) The horizontal wind velocity at various levels, including that at the 291 Building "stack top" heights.

3) The vertical wind velocity gradient.

4) The wind direction at the 291 Building "stack top" levels.

5) The difference in wind directions between levels, called the "shear" in wind direction between layers.

6) The relative humidity at the various levels.

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STACK MONITORING 292 BUILDING

This is a small building located in close proximity to the Fan House and Process Stack - (291) Building and houses the instruments for monitoring the exhaust gases from the stack. The equipment is described in general under Chapter X.

GENERAL 200 AREA SERVICE FACILITIES

In addition to the foregoing described process buildings, general power and maintenance facilities are provided in each of the 200 Areas. The 200 West Area also includes the Central Shops (272-W) for the entire plant. The 200 East Area includes the Heat Treatment (273-E) Building, which is suitable for the heat treatment of large stainless equipment, and the Fabrication Shop (272-E), which is equipped for the fabrication of the remote maintenance equipment and piping used in the Canyon (221) Buildings.

A Laundry (2723-W Building) is located in the 200 West Area, which possesses facilities for the laundering of all of the coveralls and uniforms used by the operators in the process buildings. First Aid Buildings (2719, Storerooms (2713), Supervisor's Offices (2704) and various buildings for storage of equipment are also located in each area.

A complete list of buildings in the 200 Areas is given in Table III.

Table IV lists the steam water and compressed air services to the various buildings.

Table V gives the electrical services to the various buildings.

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TABLE III

LIST OF BUILDINGS IN THE 200 AREA

Note: When the building is located in the East Area the number is followed by the letter E and when in the West Area by the letter W. (See Figures 1, 5 - 18 inclusive)

Building No.	Building Name
211	Tank Farms
212	Metal Storage Basin
213	Product Storage-Magazine
216	Waste Disposal Trench
221	Canyon Building
221-Head End	Hot Semi-Works Building
222	Control Laboratory
224	Concentration Building
231	Isolation Building
241	Waste Disposal
251	Primary Substation
252	Secondary Substation
253	Distribution Substation
271	Service Building
272-E	Fabrication Shop
272-W	Central Shop
273-E	Heat Treatment Building
274	Machinery Storehouses
275	Chemical Storehouses
282	Reservoir and Pump House
283	Filter Plant
284	Power House
288	Ash Disposal Basin
291	Fan House; Process Stack
292	Stack Monitoring Building
2607	Septic Tanks
2701	Gate Houses; Area Badge House; H.I. Laboratory
2704	Supervisor's Office
2707	Change House
2709	Fire Headquarters
2713	Storerooms
2715	Oil and Paint Storage
2716	Automobile Repair Shop
2719	First Aid
2720	Patrol Headquarters
2722	Paint and Riggers Shop
2723-W	Laundry
2729	Extra Machinery Storage
2730	Salvage Yard
2731	Burning Pit
2734	Cylinder Storage
2743	Gate House and Guard Tower
2905	Wells and Pump House
622	Meteorological Observation Tower

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TABLE IV

STEAM, WATER, AND COMPRESSED AIR SERVICES IN VARIOUS BUILDINGS

221 Building

Unfiltered Raw Water:- 10-inch line, 110 lbs/sq.in. minimum pressure at pumping station.

Treated and Filtered Sanitary Water:- 8-inch line, 100 lbs/sq.in. pressure.

Steam:- The steam supply enters as an 8-inch line at 225 lbs/sq.in. pressure and is reduced in the building to 125 and 15 lbs/sq.in.

Process Compressed Air:- A 4-inch line at 110 lbs/sq.in. pressure is supplied from 271 Building.

Instrument Compressed Air:- A 4-inch line at 80 lbs/sq.in. pressure is supplied from 271 Building.

271 Building

Unfiltered Raw Water:- A 4-inch line at 110 lbs/sq.in. pressure is supplied from 221 Building.

Treated and Filtered Sanitary Water:- A 4-inch line at about 100 lbs/sq.in. pressure is supplied from 221 Building.

Steam:- Four 4-inch lines are supplied at 125 lbs/sq.in. pressure from 221 Building.

Process Compressed Air:- A 4-inch line at 100 lbs/sq.in. pressure is provided from compressors in the Building.

Instrument Compressed Air:- A 4-inch line at 80 lbs/sq.in. pressure is provided from compressors in the Building.

211 Building

Unfiltered Raw Water:- A 4-inch line is supplied from 271 Building at about 100 lbs/sq.in. pressure.

Treated and Filtered Sanitary Water:- A 3-inch line is supplied from 271 Building at about 100 lbs/sq.in. pressure

Steam:- A 2-inch line at 15 lbs/sq.in. pressure is supplied from 271 Building.

Compressed Air:- A 2-inch line at 15 lbs/sq.in. pressure is supplied from 271 Building.

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224 Building

Unfiltered Raw Water:- A 6-inch line at 110 lbs/sq.in. pressure is supplied from the pumping station.

Treated and Filtered Sanitary Water:- A 4-inch line is supplied at about 100 lbs/sq.in. pressure.

Distilled Water:- A still and storage tank is supplied.

Process Steam:- A 3-inch supply line is provided for each of 15 and 125 lbs/sq.in. pressure.

Heating Steam:- A 6-inch supply line is provided at 15 lbs/sq.in. pressure.

Instrument Air:- A 2-inch line is provided at 80 lbs/sq.in. pressure with a Bailey filter in the line.

Process Air:- A 3-inch line is provided at 100 lbs/sq.in. pressure with an Adam filter in the line.

292 Building

Steam:- A 3-inch 225 lbs/sq.in. line is supplied.

Treated and Filtered Sanitary Water:- A 2-inch line is supplied at about 100 lbs/sq.in. pressure.

291 Building

Steam:- A 3-inch line is supplied at 225 lbs/sq.in. pressure.

Compressed Air:- A 1½-inch line is supplied at 100 lbs/sq.in. pressure.

222 Building

Treated and Filtered Sanitary Water:- A 3-inch line at 100 lbs/sq.in. pressure is supplied, which is reduced to 30 lbs/sq.in. pressure. About 180 gal/hour of heated sanitary water is supplied at 160°F.

Distilled Water:- A still is supplied.

Steam:- A 3-inch line at 225 lbs/sq.in. pressure is supplied to the vacuum system and change rooms. A 1½-inch line at 30 lbs/sq.in. pressure is reduced from the 225 lbs/sq.in. line, and supplies the still and hot water heater. A 4-inch line at 10 lbs/sq.in. pressure supplies steam for heating.

[redacted]

Compressed Air:- A 1½-inch line at 100 lbs/sq.in. pressure is supplied to the inside wall, and is reduced to 20 lbs/sq.in. for use in the Building.

Vacuum:- A line at 24 inches mercury vacuum with a capacity of 15 lbs. air/hour is supplied from a steam jet.

Gas Supply:- Methane, propane, and oxygen is supplied in cylinders.

231 Building

Unfiltered Raw Water:- A 6-inch line is supplied at 110 lbs/sq.in. pressure from the pumping station.

Cold Treated and Filtered Sanitary Water:- An 8-inch line is supplied at about 100 lbs/sq.in. pressure, and is reduced to 50 lbs/sq.in. for use in the process areas.

Hot Treated and Filtered Sanitary Water:- A 3-inch line provides 25 gal/min of 160°F water.

Chilled Raw Water:- A 3-inch line is provided supplying 50 gal/min at 40°F.

Distilled Water:- A 2-inch line is provided from a 500 gallon storage tank, and still.

Process Steam:- A 6-inch line at 225 lbs/sq.in. pressure supplies the Building. A 4-inch line at 50 lbs/sq.in. pressure is reduced from the 225 lbs/sq.in. line (3600 lbs/hour).

Heating Steam:- A 10-inch line supplies 15 lbs/sq.in. steam (14,500 lbs/hour).

Compressed Air:- A 4-inch line supplies 300 cu.ft./min of free air at 100 lbs/sq.in. pressure from compressors. A 4-inch line supplies 75 cu.ft./min at 40 lbs/sq.in. pressure.

Vacuum:- A 3-inch line supplies 228 cu.ft./min free air capacity at 22 inches of mercury vacuum.

Gas Supply:- Oxygen, methane, hydrogen, and propane gases are supplied in cylinders and is piped to the various laboratories.

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TABLE V

ELECTRICAL SERVICES IN PROCESSING BUILDINGS

Building	Voltage	Phase		Total Capacity
221	440	3	Transformers	1800 KVA
	110/220	1	Transformers	350 KVA
	110/220	1	Em.Transformers	30 KVA
	2300	3	Direct	----
224	440	3	Local	420 H.P.
	110/220	1	Local	72 KW
	110/220	1	Local	2 KW
222	440	3	Local	12 H.P.
	110/220	1	Local	34 KW
291	440	3	Local	80 H.P.
	110/220	1	Local	5 KW
292	440	3	Local	0.75 H.P.
	110/220	1	Local	3 KW
241	440	3	Local	----
	110	1	Local	1 at 3 KW 1 at 32 KW
231	440	3	Transformers	300 KVA
	110/220	1	Transformers	100 KVA
	110/220	1	Em.Transformers	3 KVA
231 Sump	440	3	Local	15 H.P.
	110	1	Local	----

Electrical power is received in the 200 Areas at 13,800 Volts three phase current. It is distributed as 2300 Volts and transformed at the point of use to the voltages indicated.

A 750 KVA Generator in the power house is available for emergency use in the event of the failure of the main power source.

A 2300 Volt - 3 phase emergency line serves small emergency 110-220 Volt transformers at the 221 and 231 Buildings, in the event of failure of the main electrical power sources to these buildings.

All buildings have battery operated emergency lighting systems which turn on automatically in the event of the failure of the regular power services.

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REFERENCES

1) Complete List of Blueprints of Equipment and Buildings in the 200 Areas, Blueprint File B.P.F. 22

2) Report TNX - PG - 13, Parts I and II, "Start-Up Manual - Hanford Engineering Works 200 Area"

3) 200 Area Operating Manual, Part III - "Building 221 Dissolving"

4) Report CN-1879 and Supplements

5) "Equipment History Manual"

6) Purchase Requisitions for Equipment in 200 Areas

7) Design Division Engineering Department Specifications for Project 9536 - Hanford Engineer Works

8) Material and Equipment List - Hanford Engineer Works

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SECTION C - SEPARATIONS

CHAPTER III - PROCESSED METAL STORAGE AND HANDLING

The special considerations necessary in the storage and handling of processed metal are discussed in this chapter. Metal processed in the pile reaction must be stored for a period of time to allow decay of activity before it is processed.

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CHAPTER III - PROCESSED METAL STORAGE AND HANDLING

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The Presence of Radioactive Gases	303
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DECAY PROCESS

Pile metal is stored 35-60 days before processing. This storage period is necessary to allow the decay of activities which are due to the presence of Np^{239}, radioactive gases in the processed metal, and various short-lived elements. The decontamination required and obtained during the separation, concentration and isolation processes is in addition to that obtained by decay during the storage period.

The Presence of Np^{239} in the Fresh Pile Metal

The product is formed by the reactions:-

$$_{92}U^{238} + {}_{0}n^{1} \longrightarrow {}_{92}U^{239} \xrightarrow{\beta^-} {}_{93}Np^{239} \xrightarrow{\beta^- + \text{a soft gamma ray}} {}_{94}Pu^{239}$$

The Np^{239}, with a half-life of 2.3 days, is present in significant amounts, for example, 3% of the weight of product at shutdown after 100 days pile operation. This element is similar to product in chemistry and tends to follow it through the separation process although it is eliminated to a considerable extent during the extraction and decontamination process. Elimination of the Np^{239} in decontamination tends to give a loss of product yield, since it decays to product on aging. However, after 35-60 days cooling this effect is negligible. On the other hand, presence of the soft gamma emitting Np^{239} in product solutions in the later stages of the concentration and isolation operations would upset any plan for bringing the decontaminated product out from behind shielding.

Figure 1 indicates, as a function of the decay time the activity due to Np^{239} in 1 metric ton of metal containing about 100 g. product/ton of metal. If the assumption is made that none of this activity is eliminated in the separation process, it is evident that process solution cannot be brought out from behind shielding before approximately 60 days, although the heavily shielded canyon operations can be started earlier. Actually, however, the Np^{239} is reduced by a factor of approximately 100 during processing in the Canyon and Concentration Buildings, thereby reducing the time from 60 days to approximately 45 days.

The Presence of Radioactive Gases

Among the radio-elements in the processed metal are xenon, iodine, and ruthenium. All of the xenon and part of the other two elements are volatilized during the dissolving operation and discharged to the atmosphere through the 291 Building stack. These gases require dilution with an enormous volume of air to make the atmosphere in the vicinity of the stack safe. This feature is discussed in Chapter IV of this section of the manual.

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FIGURE 1

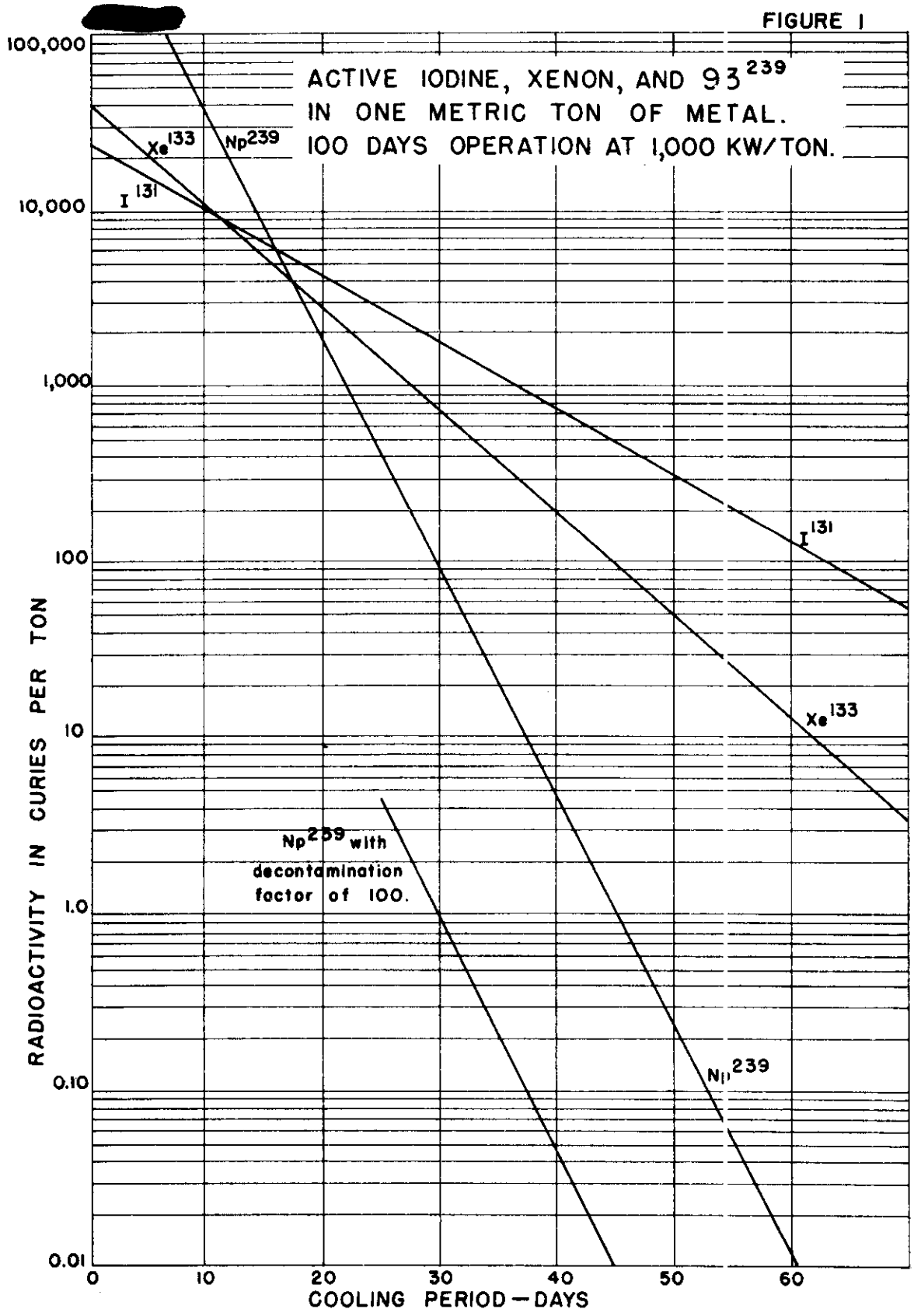

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Figure 1 shows how the 8-day iodine and 5.4-day xenon contents of pile metal decrease during the decay period. Safe disposal of the dissolver off-gases is made less difficult by aging the metal before processing.

The decrease of the gross activity in a bucket of metal after 5-day and 30-day lag storage periods is indicated in Table I. This activity does not fall off as rapidly as that associated with iodine, xenon or Np^{239} because the total activity is largely due to longer-lived radio-elements and their daughters.

TABLE I

Shielding Required by Bucket (825 lb) of Metal Processed 100 Days at 1000 KW/Ton

Stage in Process	Heat Evolved, BTU/Min.	Safe Distance, Ft.	Minimum Shield, In.		
			Lead	Concrete	Water
Pile Bldg. (1 hour)	426	1200	12	53	112
212 Bldg. (5 days)	58	800	11	48	98
Canyon Bldg. (30 days)	21	600	10	45	92

Table II gives the more important sources of radiation in the metal after 100 days pile operation at a power level of 1000 kilowatts/long ton, followed by 30 and 45-day cooling periods. These calculated results may be in error by as much as a factor of two but serve to illustrate the chief fission elements which must be eliminated during the decontamination process. (See Chapter I for a complete list of the fission elements present after various cooling periods and their radioactive properties.)

The Presence of Short-Lived Fission Elements

Whereas the decay of Np^{239} and radioactive gases is the principal reason for storing the metal before dissolving, the decay of the short-lived (half-life of 30 days or less) fission elements is also important. In general, the process decontamination factor is independent of the cooling time (30-60 days), which means that with shorter cooling times, more fission activity occurs with the final product. A useful approximate rule for estimating the gross mixed fission activity at any time is that the half-life of mixed fission activities at any time is equal to the total cooling at that time.

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TABLE II

Radioactive Energy Released by Metal Processed for 100 Days at 1000 KW/Ton

		Power Generated (Watts)/Long Ton Metal			
		30 Days Cooling		45 Days Cooling	
Element	Half-Life	Beta	Gamma	Beta	Gamma
Sr	55 days	75	--	62	--
	30 years	0.4	--	0.4	--
Y	57 days	68	--	56	--
	60 hours*	1.5	--	1.5	--
Zr	65 days	18	110	21	92
Cb	35 days*	18	120	23	120
Ru	330 days	7.6	--	10	--
	40 days	6.8	29	5.2	22
Te	32 days	3.6	1.0	2.5	0.7
I	8 days	2.6	3.7	0.7	1.0
Cs	36 years	0.5	0.8	0.5	0.8
Ba	12.5 days	26.0	--	11	--
La	40 hours*	48.0	140	21	54
Ce	28 days	32.0	28	21	19
Pr	17 mins.*	59	--	56	--
	13 days	22	--	10	--
Totals		390	432.5	301.8	309.5

*Effective half-life is longer than value indicates because of long-lived parent.

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METAL HANDLING

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Metal is received in the 200 Areas in buckets containing 105 slugs each, approximately 825 pounds, and is not normally removed from the buckets until it is dumped into the dissolvers. It is received and stored in the 212 Building Storage Basin until ready for the dissolving operation in the Canyon Building.

For transportation purposes, a bucket is placed in a cask (Figure 34 of Chapter II) which has 12.5-inch thick lead-filled walls to provide shielding so that the metal can be safely moved about the plant. (With this amount of shielding, the normal radiation level at a distance of about 2 feet from the casks entering the 212 Buildings from the 100 Areas was found to be approximately 0.5 mr./hour.) The decay process in a 1-ton batch of metal (100 days, 250 g. product/ton of metal) generates heat at the rate of about 23,000 BTU/hour as received in 212 Building at 5 days cooling and 7,200 BTU/hour as received at the Canyon Building after 35 days storage. (A calorimeter test, to measure the rate of heat liberation, is described in the Appendix.) This heat can cause enough temperature rise to damage the slug coating unless means of heat removal are provided. For this reason, the flat cars on which the casks of metal are transported are provided with water tanks. One cask can be loaded in each of the two tanks on a car. The water is circulated by convection through tubes leading into the casks. In order to minimize escape of radiation, the tubes are led through the cask walls on an S curve, and the cask cover and seat have stepped edges. To facilitate water circulation, and prevent the development of hot spots the bucket has a perforated skirt and bottom. Casks containing active metal are immersed in water at all times except during crane operations in 212 and 221 Building.

The procedures by which metal is moved into 212 Building, stored, and taken out are described in Chapter II of this section of the manual.

The metal, in buckets (but not in casks) is stored in 212 Building under 19 to 20 feet of water, which is supplied from two wells, one of 600 gal/min. and one of 1000 gal/min. capacity. This depth of water is fully adequate for shielding purposes. A normal water flow of 100 gal/min. prevents development of high localized activity at points where slight corrosion occurs. This mixing and dilution action is the chief purpose of the water circulation, as only a small flow is needed to prevent an excessive temperature rise. (See Figures 2 to 5 inclusive which summarize the calculated heat evolution from metal in 212 Building and the cooling water required for bombarded metal from various levels of operation in the piles.) In the first 8 months of operation when using the above 100 gal/min. water flow, a 1.0°C temperature rise of the cooling water was the maximum observed. The fresh well water temperatures remained at 17° - 19°C.

The 100 gal/min. water flow is not adequate, however, if fractured slugs are present. The metal corrodes readily and if coatings are broken radioactive matter becomes dissolved and dispersed in the water. In this case the flow can be increased to 1000 gal/min. to reduce the specific activity of the water until the "hot" buckets are located and removed.

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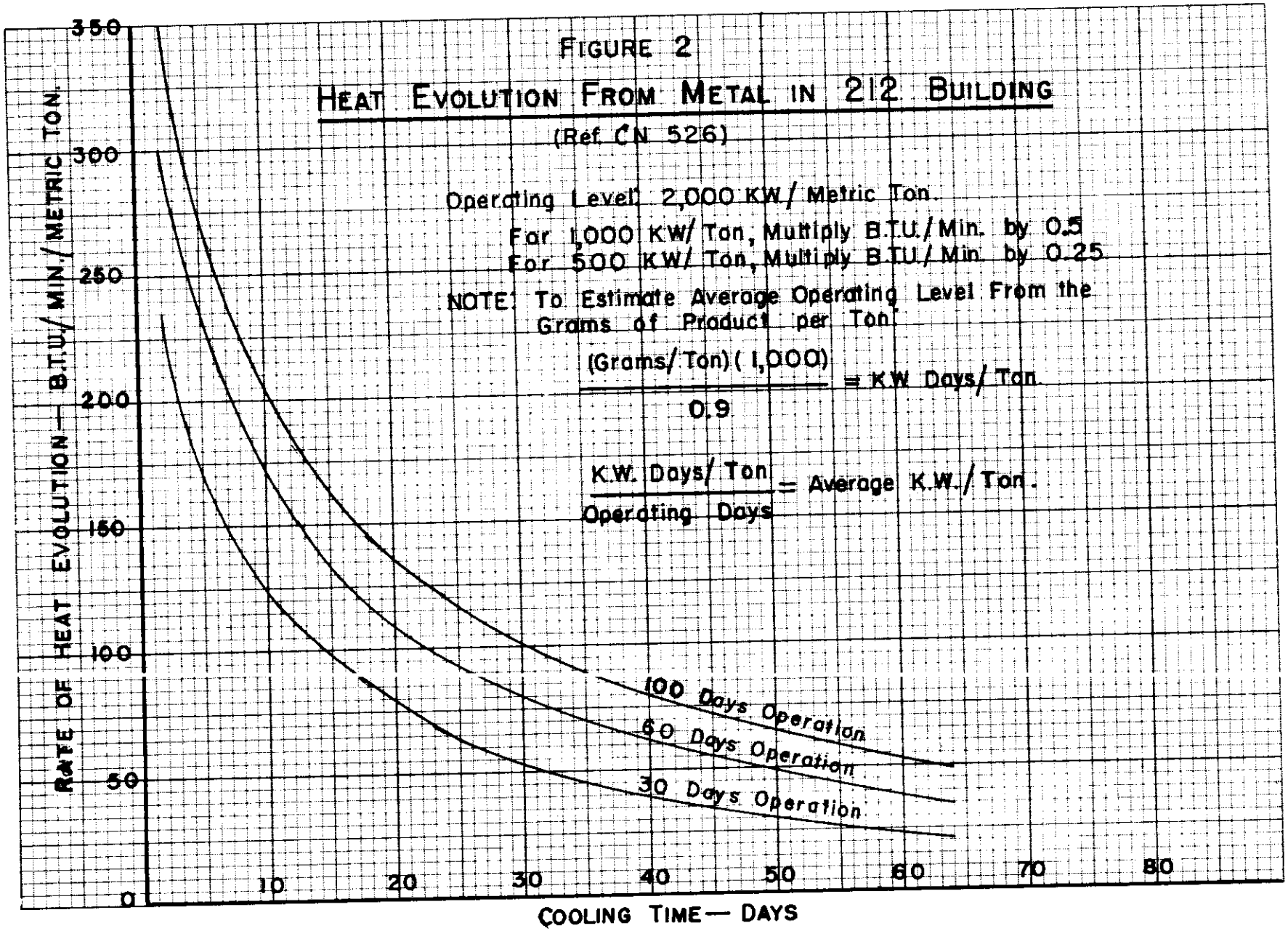

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FIGURE 3

COOLING WATER FLOW REQUIRED IN 212 BUILDING

(Ref. CN-526)

Assume Temperature Rise of 25°C (45°F).

Operating Level: 2,000 K.W./Metric Ton.

For 1,000 K.W./Ton, Multiply Gal./Min. by 0.5

For 500 K.W./Ton, Multiply Gal./Min. by 0.25

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FIGURE 4

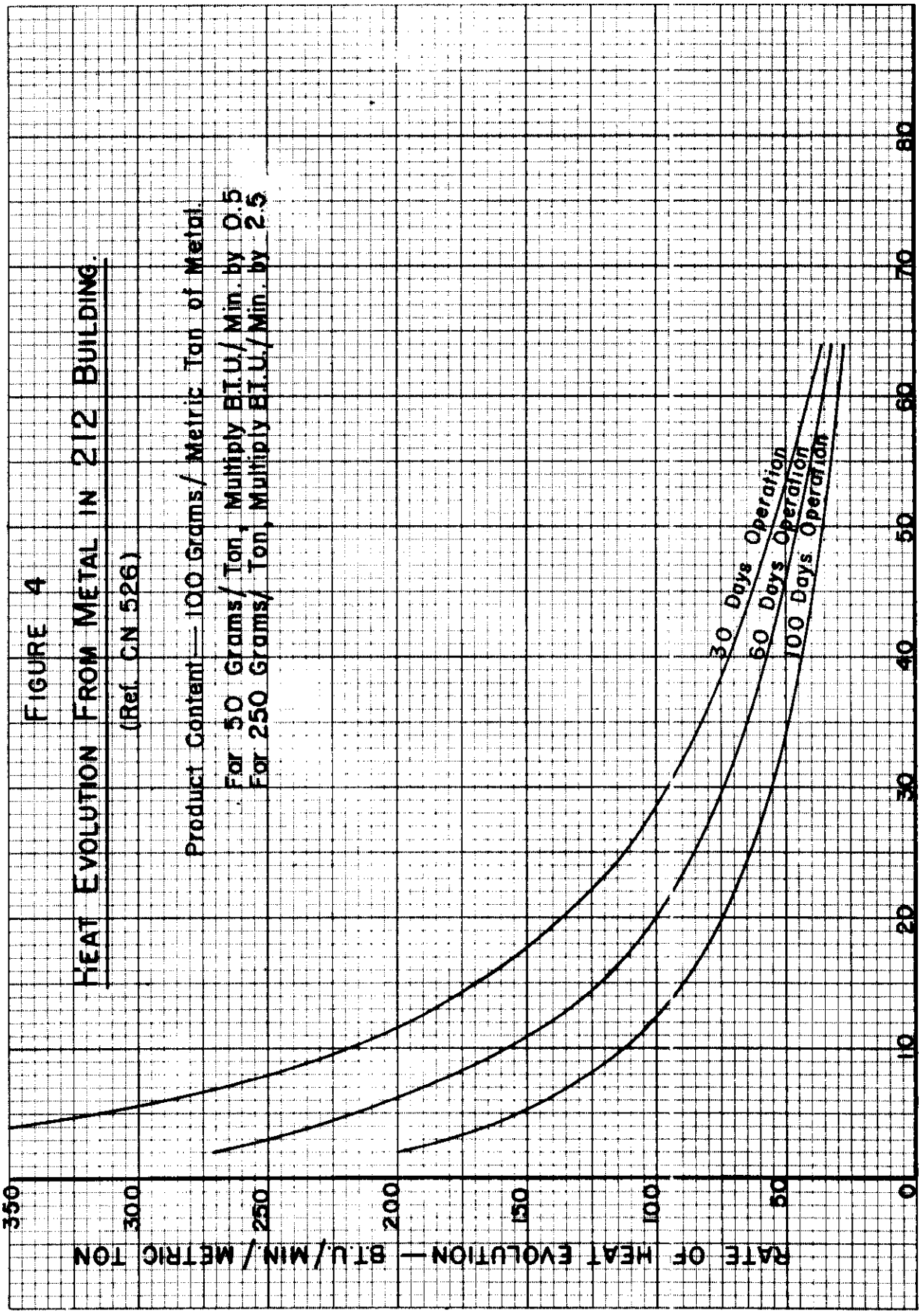
HEAT EVOLUTION FROM METAL IN 212 BUILDING

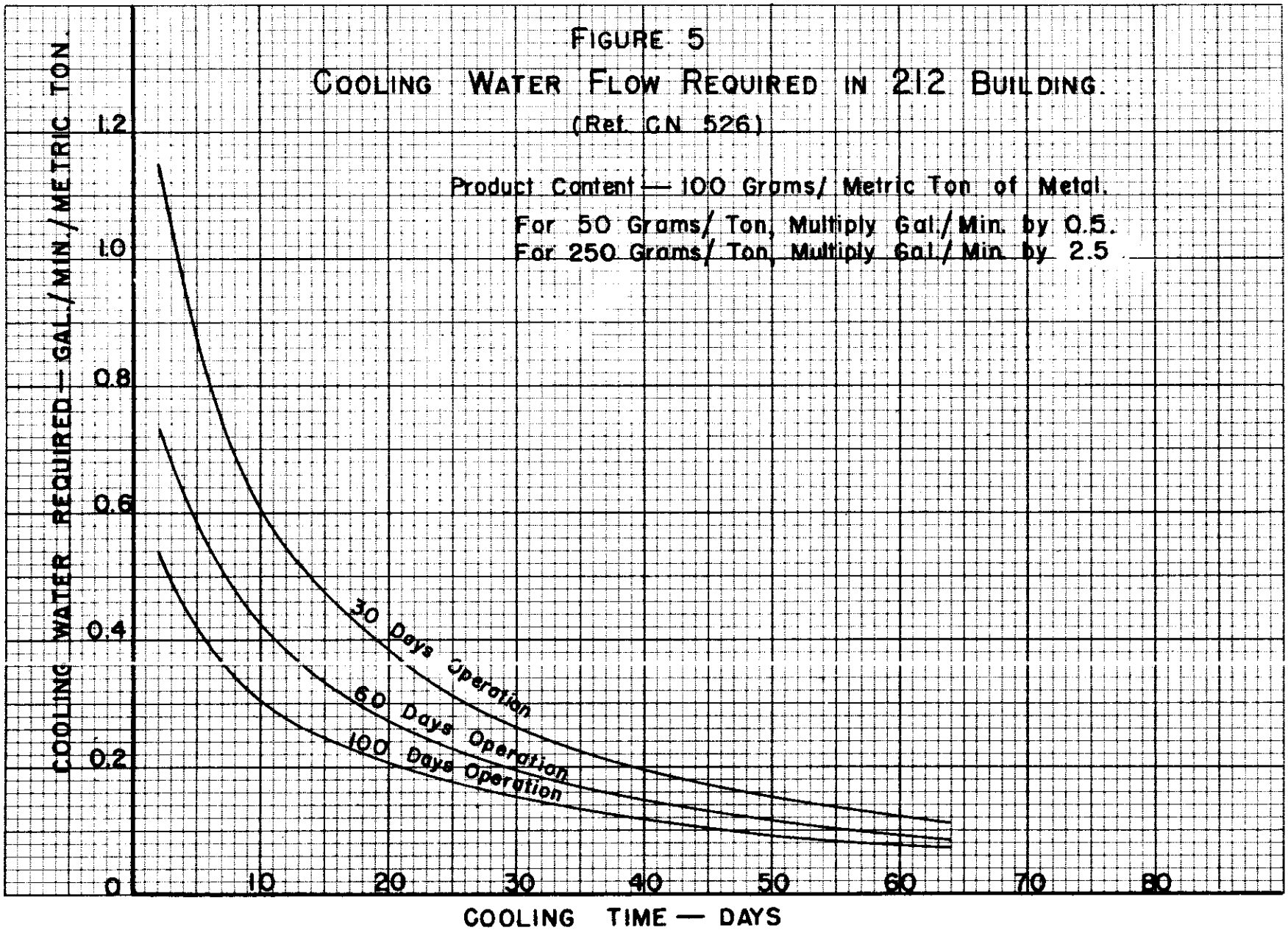
(Ref. CN 526)

Product Content — 100 Grams/ Metric Ton of Metal.

For 50 Grams/ Ton, Multiply B.T.U./ Min. by 0.5

For 250 Grams/ Ton, Multiply B.T.U./Min. by 2.5





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Laboratory monitoring of the storage basin cooling water over the first 8 months of operation showed 20 to 40 beta disintegrations/(min)(ml), which is considered innocuous.

The buckets containing slugs with damaged coatings are stored in Section 2-R (Cell 4) of the Canyon Building until they are ready for processing. This cell is provided with a waterproof lining and is normally filled with water. The overflow from Section 2-R (Cell 4) discharges into the "haunch" sewer along with the Canyon Building cooling water wastes so that there is adequate dilution of the active effluent. In the first 8 months of operation no damaged slugs were found which required storing in the above special cell in the Canyon Buildings.

Metal is brought into the Canyon Buildings in the tank cars, three-fourths of a metric ton/car. Two cars are brought in at one time with four cars being required to charge one dissolver.

These cars enter the building through a 150-foot railroad tunnel which goes through the front wall at cell level into Section 2-L. The purpose of the tunnel is to provide a long and well shielded entrance so that the accidental spilling of a bucket of slugs on the cell floor cannot cause dangerous radiation hazards outside the building.

The metal coming into the Canyon Building is charged directly from cask car to dissolvers, except in the case of buckets containing damaged slugs coming in for storage. The charging operation is carried out with the overhead crane. To prepare for receiving the metal, two cover blocks are removed from Section 2-R and from one of the dissolver cells (3-L, 3-R, and 4-L) and the lid on the dissolver itself removed. (See Chapter IV)

The unloading and charging operations create the maximum radiation hazard existing in 200 Area operations. The canyon is closed to all personnel and the locomotive bringing cars into the building must withdraw beyond the tunnel entrance before bucket handling begins. This procedure is controlled by the dispatcher through phone connections to the crane operator and building entrances and the electrically locked doors of the tunnel and canyon deck.

The crane operator lifts the cover off of the car, unlocks the cask with an impact wrench, picks up the cask lid with a yoke and places it on one end of the car. The bucket is then lifted out of the cask, brought over the dissolver and lowered into the charging opening. As shown in Figure 6, the bucket is lowered onto the hooks in the dissolver opening, being guided to them by the centering guides. As the crane operator continues to lower the bucket yoke, the lower lugs of the bucket catch on the hooks causing the bucket to tip forward and to dump the slugs into the dissolver. When this operation has been completed, the bucket is lifted up, rinsed in water in Section 2-R to remove any active dissolver solution and replaced in the cask.

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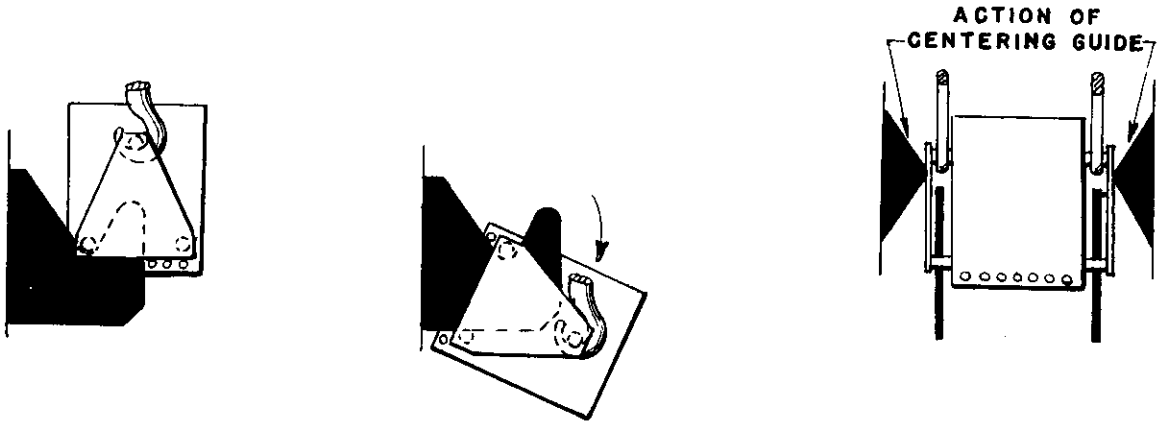
FIGURE 6

CHARGING THE DISSOLVER



The canyon deck is unsafe for personnel during the charging operation while buckets are in the air. No personnel is permitted in the canyon or tunnel when either a full or empty slug bucket is being transported. It may also be unsafe in the tunnel or canyon when a cask lid is removed. Radiation from hot metal in an uncovered dissolver cell can, by means of "sky shine", render the canyon or the car tunnel unsafe for personnel. However, at radiation levels encountered during the first 8 months of operation, it has been considered safe to take samples from Section 8 and beyond while the dissolver cell covers are removed, even with 3 tons of metal in the dissolver.

Laboratory measurement on the beta activity present in the cooling water surrounding the casks on the railroad cars returning to the 212 Buildings from the 200 Areas have shown 20 to 100 beta disintegrations/(min)(ml), while the cooling water in those returning from the 100 Areas have shown an average of 50 disintegrations/(min)(ml).

After eight buckets have been loaded, the dissolver lid and cell covers are replaced; the coating removal procedure as described in Chapter IV may then be started.

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APPENDIX

MEASUREMENT OF RATE OF HEAT EVOLUTION FROM ACTIVE METAL

Using the dissolver as a calorimeter, it is possible to measure experimentally the rate of energy release during the decay prccess. Data have been obtained in B Canyon Building on the standard 3-ton charges of metal plus a metal heel of about 1 ton; the sodium nitrate solution employed in the coating removal step was used to absorb the radiations from the metal. The procedure is as follows:-

1) Load 3 tons (8 buckets) of metal on top of metal heel to make approx. 4 tons total.

2) Add the $NaNO_3$ solution to cover the slugs.

3) Turn off condenser water and jacket water. With the air sparger on, turn on the coil water and cool the dissclver to 3°-4° below cell temperature. Maintain this condition for about 6 hours.

4) Shut off air sparger and coil water and reduce the vent jet pressure to give only about 1 inch of water vacuum in the dissolver.

5) Allow the dissolver temperature to increase and record the time required to rise from 2.5°C below cell temperature to 2.5°C above cell temperature.

In calculating the heat capacity of the dissolver, the following items are included; metal weight, dissolver weight, $NaNO_3$ solution weight, jacket and coil water weights, and the weight of the UNH heel from the previous run. In the cases studied, the heat capacity of the system has been estimated to be 5575 BTU/°F. The temperature of the column, condenser, and packing material does not change appreciably during such a test, and the heat lost from the system via the vent gas is less than 1 BTU/min.

In a typical test, 3 tons of metal containing about 214 g./ton metal and cooled 28 days were charged on top of an approximately 1 ton heel containing about 176 g. product/ton and cooled 35 days. Heat was evolved at an average rate of about 100 BTU/(min)(ton). Possible sources of error in this procedure are (1) agitation during heating period is not positive, and (2) some of the radiation escapes the vessel without being converted into heat.

[redacted]

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SECTION C - SEPARATIONS

CHAPTER IV - PREPARATION OF METAL SOLUTION

The first steps in the separation process are concerned with the preparation of uranyl nitrate solution from the active metal. This chapter describes the operations of coating removal, metal dissolving, sulfuric acid addition, and solution storage; these are the necessary operations prior to extraction.

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CHAPTER IV - PREPARATION OF METAL SOLUTION

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PROCESS

General

The operations of coating removal and metal dissolving are carried out consecutively in the dissolvers. These are vertical tanks equipped to receive charges of the active slugs and conduct operations at the boiling point under slightly less than atmospheric pressure. There are three dissolvers in each Canyon Building; located in Sections 3-L, 3-R, and 4-L, and designated as 3-5L, 3-5R, and 4-5L respectively.

The metal is charged into the dissolvers in batches of 3 long tons (8 buckets of 105 slugs each). In order to increase the rate of solution by use of a large amount of reacting surface, a metal heel of approximately 1 long ton is carried in the dissolver. Thus the dissolver contains approximately 4 long tons of metal just after charging and approximately 1 ton after three 1-ton batches of metal have been dissolved.

The aluminum jackets are removed from the slugs by reaction with sodium hydroxide in the presence of sodium nitrate solution. The sodium nitrate prevents the evolution of hydrogen which might reach an explosive concentration in the dissolver. Part of the silicon bonding alloys is dissolved, and part forms scale or sludge which is flushed out with the coating removal solution or with the water and acid washes which follow the coating removal step.

The metal is dissolved in batches of 2200 lbs. each by adding 5000-5400 lbs. of 60-62% nitric acid and digesting until a specific gravity of 1.80-1.82 at the boiling temperature is reached. Each batch of metal solution is diluted in the dissolver to approximately 55% UNH to prevent crystallization, cooled, and jetted to Section 4R where previously accumulated wash water dilutes it to approximately 40% UNH. Sulfuric acid is then added to give a concentration of 0.338 lb. H_2SO_4/lb. metal. After agitation the solution is ready for transfer to the extraction section.

During metal dissolving the off-gases, which include air, oxides of nitrogen, and small quantities of radioactive elements, are removed from the vessel via a stainless steel line and vent jet to the 291 Building stack for dilution and disposal to the atmosphere.

Under unfavorable weather conditions, e.g., no wind, down draft, or fog, the 291 Building facilities do not dilute the radioactive gases to a safe level and the dissolvers can not be operated. However, two dissolvers can produce metal solution faster than it can be processed. This excess capacity permits a 4-day stock of metal solution to be accumulated; the third dissolver is held as a spare.

Chemistry of Coating Removal

Each metal slug of 7.85 lbs. average weight is enclosed in an aluminum jacket when received in the 200 North Area. The jacket wall thickness is

0.035 inches and the heavier and pieces are approximately 0.375 inches thick, one being integral with the aluminum tube and the other welded on after the slug is placed in the jacket. The aluminum and uranium surfaces are bonded together with molten aluminum-silicon, which produces aluminum - silicon - uranium alloys on the surface of the slug. The total coating material on a finished jacketed slug weighs 115-120 grams (0.265 lb.) of which 80-90 grams are aluminum.

Two methods have been developed for removing aluminum jackets: the mercury catalyzed 10% nitric acid procedure, and the 10% sodium hydroxide - 20% sodium nitrate procedure. The HNO_3 - Hg procedure was used extensively in the pilot plant where there was no problem of silicon bonding material. It has not been used at Hanford because control of the reaction with multiple batch charges is questionable and a caustic treatment is necessary to remove the bonding alloys.

Two versions of the 10% NaOH - 20% $NaNO_3$ procedure have been used: with the metal charge in the vessel, (1) all the caustic is added to the cold $NaNO_3$ solution, the temperature allowed to rise, and the heat of reaction removed with coil cooling water, or (2) the caustic is added slowly to the boiling $NaNO_3$ solution, and the heat of reaction removed by the column condenser water. The latter method is now being used; the chemistry of the two variations is essentially the same.

In the absence of sodium nitrate the theoretical mol ratio of caustic to aluminum is 1 (Equation 1 below), but in the presence $NaNO_3$ it may be either 5/8 to 1 (Equation 2 below) if ammonia is formed or 1 (Equation 3 below) if sodium nitrite is formed. Several plant scale tests with inactive materials have indicated that 40-70% of the reaction proceeds according to the nitrite reaction.

1) $2\ Al + 2\ NaOH + 2H_2O \longrightarrow 2Na\ AlO_2 + 3\ H_2$

2) $8\ Al + 5\ NaOH + 3NaNO_3 + 2H_2O \longrightarrow 8NaAlO_2 + 3\ NH_3$

3) $2\ Al + 2NaOH + 3NaNO_3 \longrightarrow 2NaAlO_2 + 3NaNO_2 + H_2O$

Laboratory experiments have shown that a NaOH/Al mol ratio of 1.25 provides an adequate excess for solution of the aluminum. However, this ratio is not great enough to insure that Al_2O_3 will not precipitate if the solution is allowed to stand for a day. Experimental results have shown that a NaOH/Al mol ratio of 1.65 prevents precipitation even if the solution is held for long periods of time. In the plant this ratio is approximated by the use of 1070 lbs. of 50% NaOH to dissolve about 220 lbs. of coating material from a 3-ton metal charge.

Aluminum is vigorously attacked by sodium hydroxide over a tested concentration range of 5-50%; the reaction rate increases with increased caustic strength. Uranium metal loss by caustic attack is very slight in solutions up to 30% NaOH, but the rate of solution becomes appreciable in 50% NaOH. In the coating removal step, dilute caustic is desirable to limit the rate of reaction and to obtain sufficient solution volume to cover the slugs in the dissolver without using an unreasonably large excess of caustic. Precipitation of

alumina may occur in the coating waste if the caustic concentration is as low as 8% based on final solution weight. At 10% NaOH and a NaOH/Al mol ratio of 1.65 or above, alumina does not precipitate even after a long storage period.

The concentration of sodium nitrate used is less critical than that of the caustic. Concentrations as low as 10% are satisfactory for supression of hydrogen formation, but there is some evidence that solid residues are more completely removed from the dissolver with the coating waste solution if 20% sodium nitrate is used.

Caustic dissolves aluminum-silicon alloy, but the aluminum-silicon-uranium scale may be removed by undercutting and the disintegrating action of the alkali. A large fraction of this suspended material is removed with the coating waste. The dissolver is given a water flush and a 5% nitric acid wash to remove any residual material. The coating solution, water wash, and acid wash are combined in Section 15 for disposal to the waste storage area. The resultant solution is sufficiently alkaline for storage; the specific gravity is 1.5 and the freezing point is below -10°C. Pilot plant data show a maximum product loss of 0.1% in this solution.

Chemistry of Metal Dissolving

Uranium metal is dissolved and oxidized by nitric acid to uranyl nitrate hexahydrate ($UO_2(NO_3)_26H_2O$). The rate of this reaction increases with increased temperature and increased nitric acid concentrations. The acid consumed in the reaction can vary between the extremes indicated in the following equations:

$$U + 4\ HNO_3 \longrightarrow UO_2(NO_3)_2 + 2NO + 2H_2O$$

$$U + 8\ HNO_3 \longrightarrow UO_2(NO_3)_2 + 6NO_2 + 4H_2O$$

Actual acid consumption lies between these two extremes and has averaged approximately 5.5 mols acid/mol metal in pilot plant and plant operation. Acid utilization may vary with dissolver column design, gas velocity in the column, (which in turn is affected by the amount of air leakage into the vessel) and condenser water temperature and flow. Essentially no acid is regenerated until the contents of the dissolver reach the boiling point and the flow of reflux (containing water, acid, and dissolved oxides) begins in the column.

The heat of reaction of metal with nitric acid is 1472 to 2713 BTU/lb. With nitric acid of 60% or higher concentration and adequate metal surface, this heat is sufficient to keep the reaction mixture at the boiling point.

The product, contained in the slugs of metal, is oxidized by nitric acid to either the (III) or (IV) state and forms soluble nitrates. There has been no indication in pilot plant or plant operation of the oxidation of any appreciable amount of product to the (VI) state. The wide variety of fission-formed radioactive elements, or by-products, include nitric acid soluble, insoluble, and volatile materials. The nitric acid insoluble materials are present in very small quantities and do not seriously affect the handling of metal solution. The volatile materials of greatest importance are xenon and iodine with a small quantity of argon. These two principal elements appear in the dissolver

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off-gas and must be diluted with very large volumes of air before being discharged to the atmosphere from the 200-ft. stack. Thus, the ventilation and dilution requirements are based on the small quantities of radio-gases rather than the much larger amounts of nitrogen oxides.

During a dissolving operation, approximately 850 lbs. of nitrogen oxides are evolved. Facilities provided for the disposal of the radioactive gases dilute the nitrogen oxides well below the limits adopted for ordinary industrial practice. A curve showing the relative xenon and iodine concentrations in the dissolver off-gas from three consecutive dissolvings is shown in Fig. 1. The rate of xenon evolution is approximately proportional to the rate of the metal-acid reaction. The curve for iodine indicates that it is evolved until the dissolver reaches the boiling point, when the column reflux then scrubs the iodine out of the off-gas. At a later time in the cycle when most of the acid has been consumed, the acidity of the reflux falls off and iodine again leaks through the column. Evolution of active gases essentially stops when the charge is diluted and cooled. This explanation of the iodine curve has been checked by laboratory experiments where it was found that iodine was lost from a refluxing solution of dilute nitric acid but not from a refluxing solution of strong acid.

The unjacketed metal slugs are 8 inches long, 1.36 inches in diameter, and weigh 7.85 lbs. each. A total of 840 slugs are included in each dissolver loading of 3 long tons; this metal is dissolved in three 1-ton charges. It is necessary to carry a metal heel of approximately 1 ton in the dissolver in order to provide adequate surface for dissolving the third charge of a series in the desired time of approximately 8 hours. It has been found that the peak reaction during the dissolving of the second charge may be as vigorous as the peak reaction on the first charge. This is probably due to the large pitted and porous surface area of the metal at the start of the second charge as compared to the relatively smooth slug surface (except for the heel) at the start of the first charge. The reaction rate on the third of a series of three reflects the smaller metal surface area and is slower than the first two.

The product in processed metal is not equally distributed throughout the slug, but is more concentrated near the surface. In pilot plant operation with 1x4-inch slugs it was found that the ratio of product in three successive batches of metal (outside, middle, and core material) was 1.00/0.87/0.73. Carrying a 1-ton heel of core material in the dissolver thus tends to equalize what might otherwise be an even greater difference in level. The blending of three successive dissolver charges in storage tends toward a constant product concentration for extraction.

The use of starting nitric acid strengths below 60% is not efficient from the time cycle standpoint because of slow reaction rate. Acid strengths up to 70% have been used in semi-works on single-batch charges; higher acid concentrations increase the reaction rate but also increase the corrosion rate of the vessel. From the standpoint of both corrosion and reaction rate, 70% HNO_3 is the maximum acid strength permissible and even this concentration is not recommended for multiple batch dissolving.

It is necessary to stop the reaction between the nitric acid and the metal when the free acid content is 0.2% to 1.5% HNO_3 (20% UNH basis) in order to

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FIGURE 1

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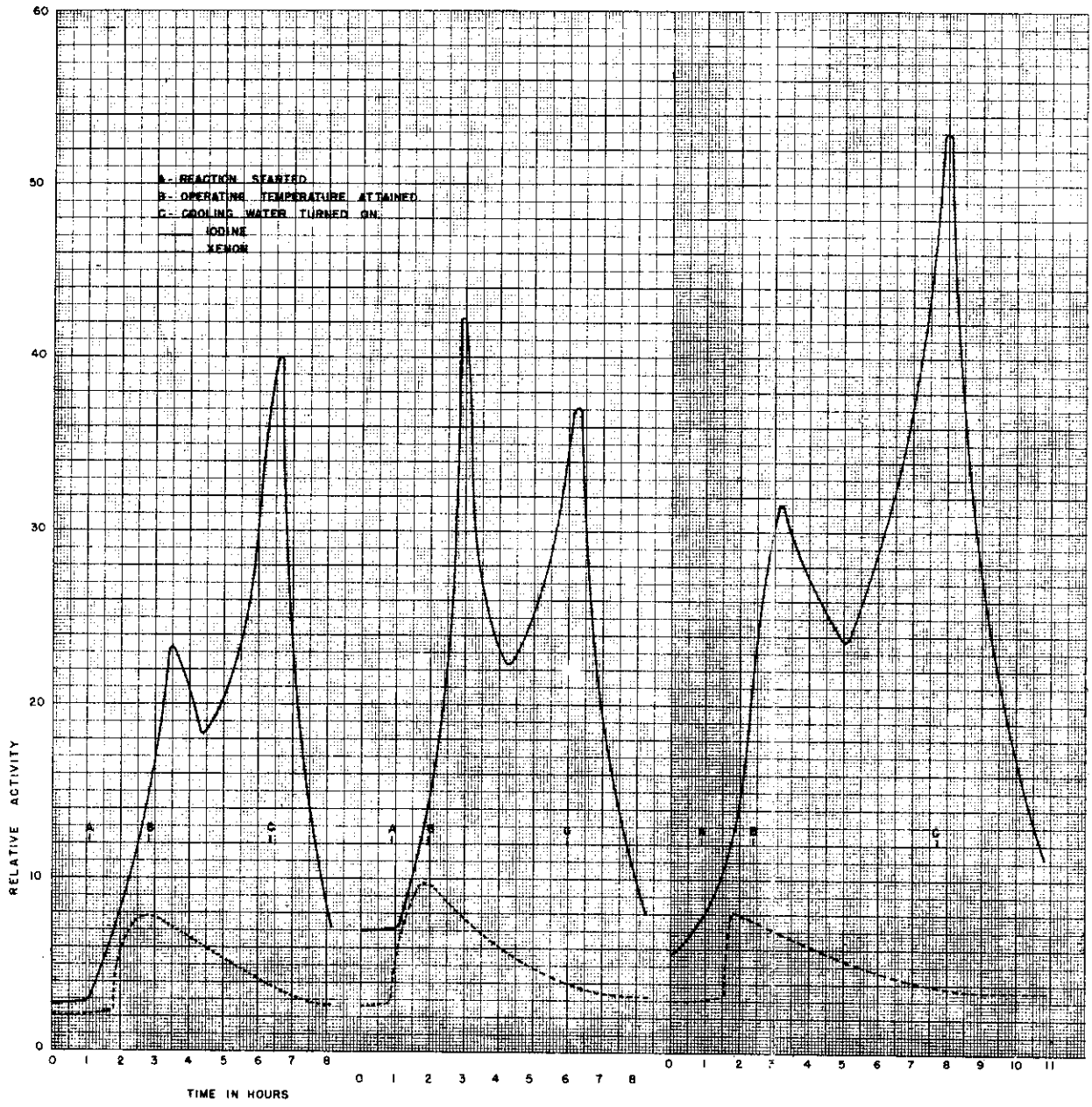
XENON & IODINE EVOLUTION DURING DISSOLVING



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assure satisfactory yields on the subsequent extraction step (See Chapter V). Sampling and analysis of the 70% - 80% UNH solution in the dissolver at the end point is not practical since the material freezes when cooled to room temperature. It has been found that the proper free acid concentration exists when the solution has been digested to a specific gravity of 1.80-1.82 (at the boiling temperature). It is usually not possible to predict the composition of a three-component mixture (HNO_3 - UNH - H_2O) by fixing one variable; in this case, the effect of the residual nitric acid on the specific gravity is small. The boiling point of the solution at the end point is approximately 105°C with a 30-inch H_2O vacuum on the dissolver.

Metal Solution Storage

The addition of sulfuric acid to the UNH solution in storage is not critical. The acid may or may not be added at this time, depending upon operating convenience. An advantage to be gained by the addition of the sulfuric acid at this point, based on laboratory data, is the "stabilization" of plutonium in the lower states of oxidation, but this is not important due to the subsequent nitrite treatment.

EQUIPMENT

Dissolver

The dissolver (D-62064), shown in Figures 2 and 3, is a 25-12 S-Cb tank of rugged construction designed to withstand the mechanical shocks of the metal charging procedure and severe corrosion conditions. Because of their exceptional bottom thickness and complicated construction, the dissolvers do not receive the heat treatment given all other process vessels and, hence, are less corrosion resistant, particularly to solutions containing hydrogen fluoride.

The tank is 7 feet in diameter by 6 feet 10 3/8 inches high with a maximum working capacity 6 inches from top of 1700 gallons. A jacket and coil are provided and both connect to water and steam at the gauge board for heating or cooling as necessary. An air and steam sparger ring is placed on the bottom of the vessel, between the coil and wall. Inlet and outlet pipes for jacket and coil and the sparger inlet terminate in vertical connector flanges on top of the vessel, from which connections are made to appropriate cell wall connectors.

At the top of the vessel is the 2-foot 10-inch diameter charging opening through which metal is loaded. Beneath this opening is a 5-foot 3-inch diameter crib of stainless steel staves which receives the metal and protects the coils. The charging opening is covered with a heavy stainless steel lid which rests on a grooved machined surface but is not otherwise fastened in place. Since a satisfactory vacuum must be maintained in the dissolver during operation, it has been necessary on several vessels to regrind the lid and flange surfaces to limit the air leakage under the lid. Beveling of the edges of the flange and groove makes easier the proper seating of the lid on the dissolver.

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FIGURE 2

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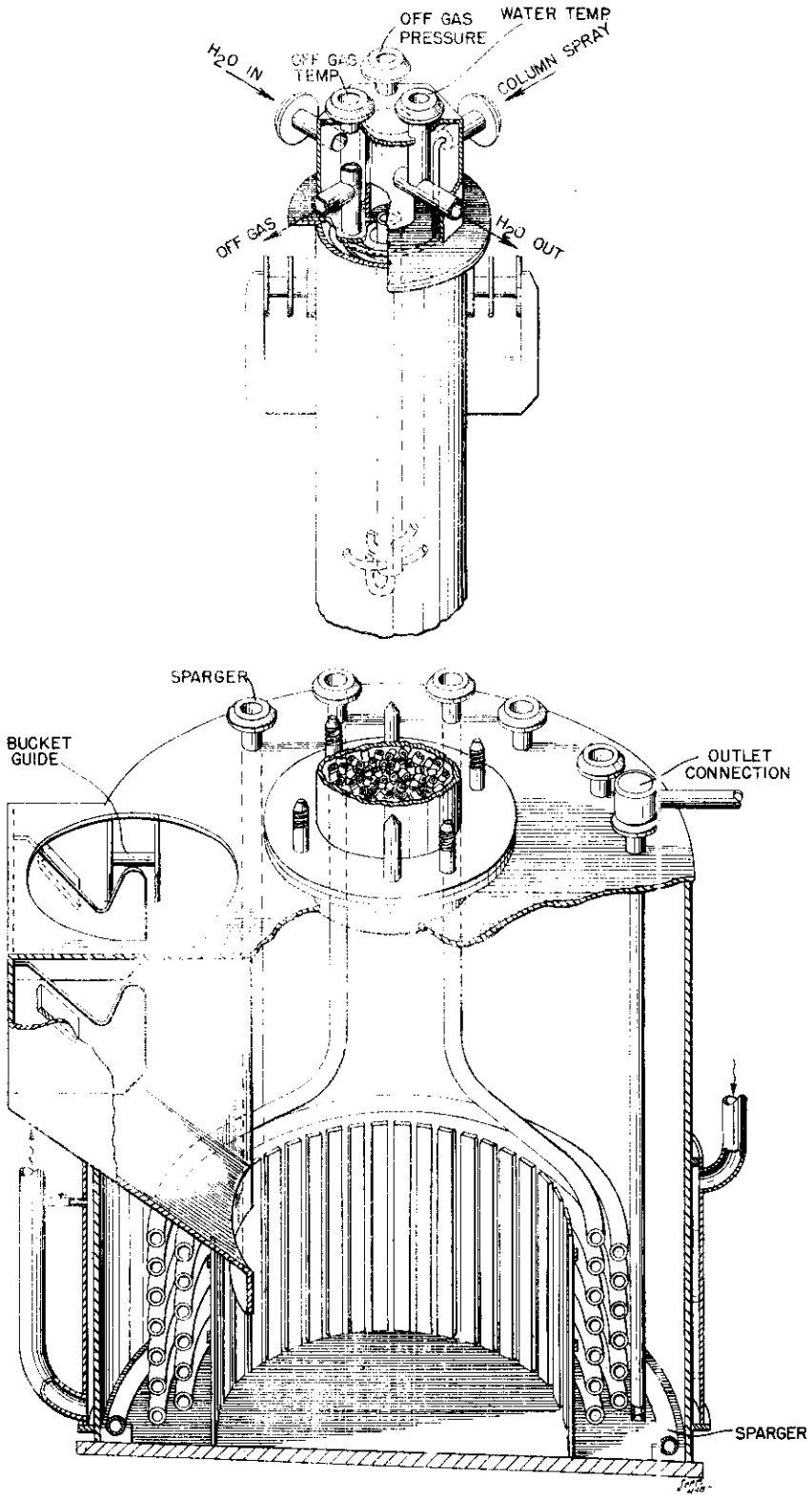

DISSOLVER

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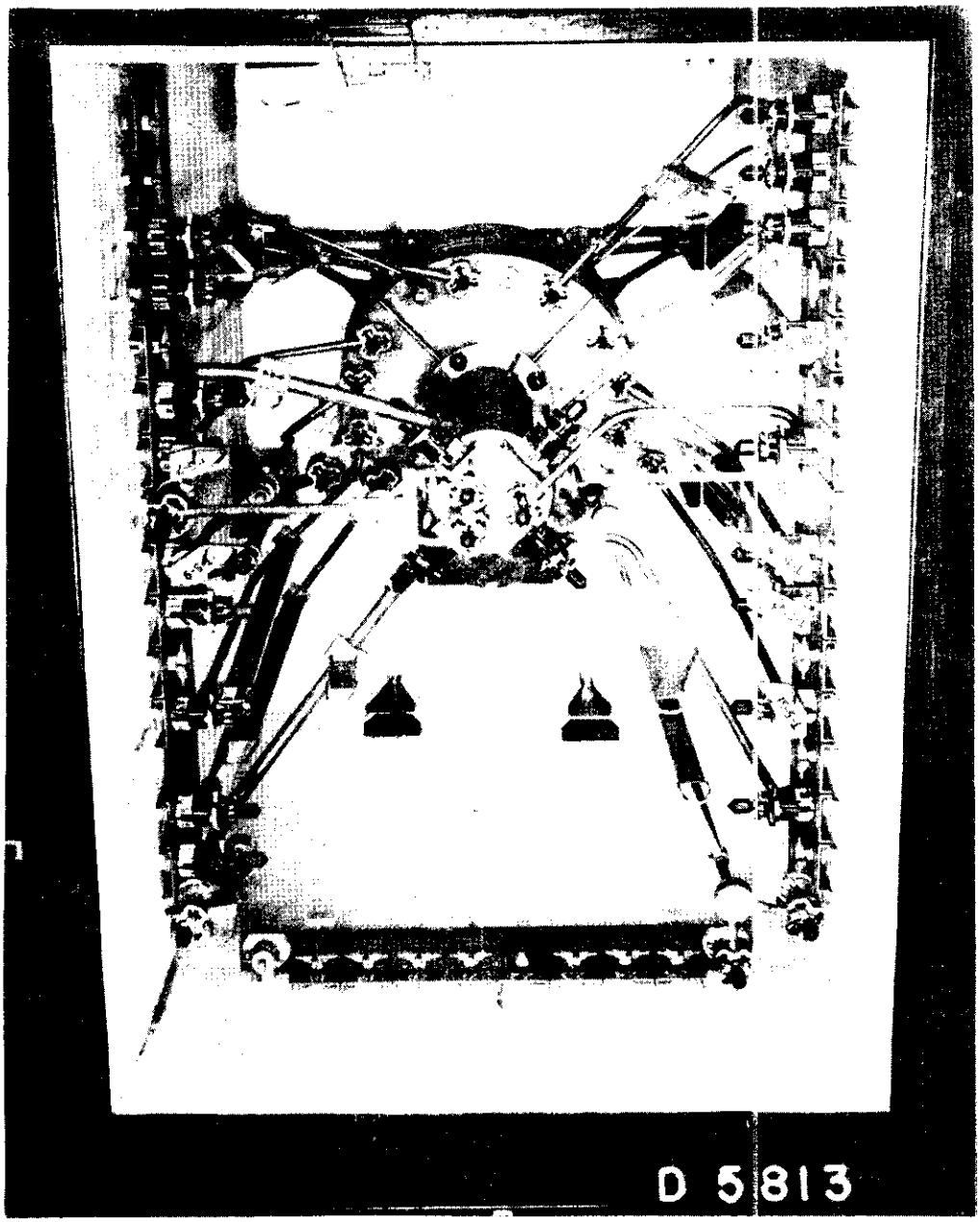
FIGURE 3

UNCLASSIFIED

DISSOLVER 3-5-L



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In addition to the jacket, coil and sparger connections, there are nine openings through the top of the dissolver, terminating in horizontal flanges. Connections are made through these for process transfer lines, chemical feed, sampler, thermometer and a liquid level and density recorder.

Piping such as thermometer wells or transfer outlets, which must reach into the vessel, are screwed or welded to the face of automatic connectors attached to prefabricated cell piping assemblies. Using the impact wrench and hooks or "pipe grabber", the crane operator can unfasten an assembly at the cell wall and vessel connectors, and take it out for repair or replacement. An outlet pipe is shown in position on the right side of Figure 2.

In addition to the lines going to the dissolver itself, the cell wall connectors furnish outlets for a space microphone and for measuring cell pressure and temperature.

Figures 4 and 5 are diagrams of the connections in Sections 3 and 4. The cell and pipe gallery connections are identified with the standard numbering system. In addition, cell connections not blanked off at the gallery wall carry a note indicating their function or the letter of the panel board to which they run. Where two letters are used, a double line runs to the board indicated, e.g., Connector 8 carries two thermometer leads from Panel M and is labeled MM.

Column

On top of the dissolver is a column 2 feet in diameter by 12 feet high (D-62063). (The performance characteristics of the dissolver and column are described in the appendix.) This is guided into place by dowel pins on the dissolver and bolted down with the impact wrench, so that it can be taken out for repair or replacement without moving the dissolver itself.

The lower portion of the column contains about 5 feet of Duriron Raschig ring packing for fractionating vapor from the dissolver. Above this is a reflux condenser (D-62063) consisting of six concentric cooling coils 5 feet high. The column is estimated to develop a pressure drop of 20 to 25 inches of water before loading and is normally operated at a 10-inch maximum. At the top of the column is a spray ring for washing down the condenser coils.

Four horizontal connector flanges are placed around the head of the column and three vertical flanges are located on the top. The horizontal connectors are the spray and column inlets, the coil and off-gas outlets. The off-gas outlet is attached via the cell wall, to a line terminating in the breach of the 291 Building stack. At 291 Building the line contains a steam jet by which the dissolver can be maintained under a partial vacuum during operation to prevent leakage of gas into the cell. (See Figure 68, Chapter II, for the arrangement at the base of the stack.) Two of the flanges on the top of the column are connected into the off-gas and cooling water outlets, respectively, for thermometer connections, while the third opens into the coil space and provides for measurement of the off-gas pressure.

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FIGURE 4

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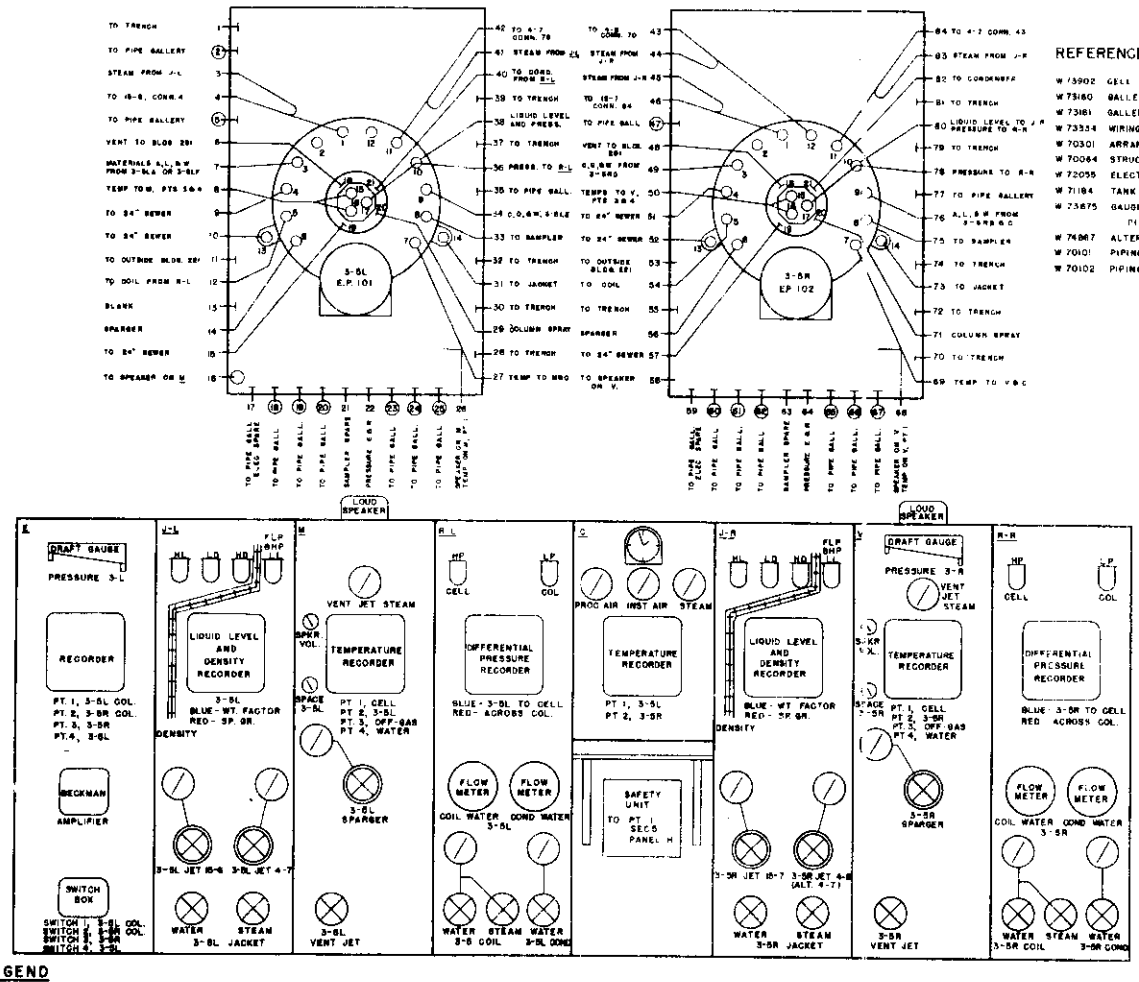
CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 3



REFERENCE DRAWINGS

W 73902	CELL PIPING
W 73180	GALLERY PIPING AND ARRANGEMENT
W 73181	GALLERY PIPING AND ARRANGEMENT
W 73334	WIRING DIAGRAM, ELECTRICAL
W 70301	ARRANGEMENT KEY
W 70064	STRUCTURAL KEY
W 72055	ELECTRICAL KEY
W 71184	TANK ARRANGEMENT
W 73675	GAUGE BOARD AND SERVICE PIPING HEADERS
W 74867	ALTERNATE CELL PIPING
W 70101	PIPING IN CONCRETE
W 70102	PIPING IN CONCRETE

LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WALL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM
- HP HIGH PRESSURE LEG
- LP LOW PRESSURE LEG
- BHP HIGH PRESSURE LEG, BACK RING, CROSS COLUMN
- FLP LOW PRESSURE LEG, FRONT RING, TO CELL

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FIGURE 5

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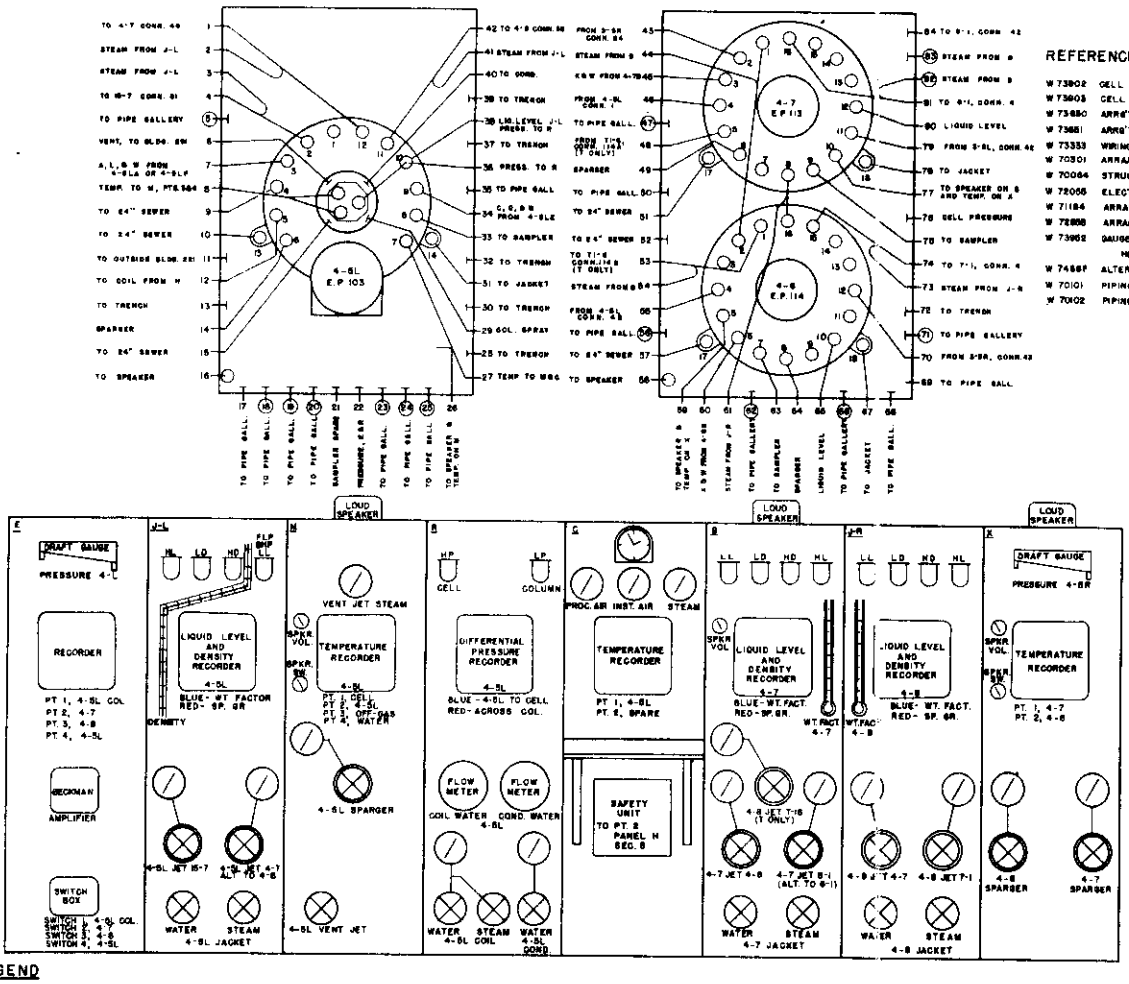
CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 4.



LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WALL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM
- HP HIGH PRESSURE LEG
- LP LOW PRESSURE LEG
- BHP HIGH PRESSURE LEG, BACK RING, ACROSS COLUMN
- FLP LOW PRESSURE LEG, FRONT RING, TO CELL

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Gauge Board

Figure 5 shows the Section 4 gauge board, controlling Dissolver 4-5L, and the two solution storage tanks. The various instruments shown on the board are described in Chapter X of this section of the manual.

The four-point temperature recorder reads off-gas, condenser outlet water, dissolver and cell temperatures. A second thermometer in the dissolver is connected to the two-point recorder in the center of the gauge board. All dissolvers and precipitators are equipped with two temperature recorders. Process steps conducted in these vessels require close temperature control; the double thermometers serve to check each other as well as to give more frequent readings.

The dual differential pressure recorder measures the pressure drop across the column and the drop between cell and dissolver produced by the off-gas steam jet. For measuring the latter, it is connected into the draft gauge cell pressure line and the liquid level and density recorder vessel pressure line. Column pressure drop is measured by balancing the same vessel pressure line against column pressure, measured through a line leading into the top of the condenser space. An inclined liquid manometer has been installed in parallel with the density recorder which allows accurate reading of the specific gravity in the range of 1.75 - 1.85.

Flowmeters and pressure gauges are provided to aid in controlling the flow of steam or water to the coil and water to the condenser. The jacket line is valved for water and steam but has no indicators; heating or cooling with the jacket is a slow process. Close and speedy control is best obtained by operation of the coil. Pressure on the jacket and dissolver coil is limited to 15-20 lb./sq.in. by pop valves placed in the discharge lines.

All jet and sparger lines are equipped with gang valves. As the steam flow through these valves is turned off, they automatically pass through a position where compressed air is blown through the vessel line. This expels the steam and prevents condensation in the line, which might otherwise cause a suckback of radioactive process solution into the gallery piping. In the case of spargers, this arrangement also permits either air or steam to be used.

The sensitivity recorder is connected to the two ionization chambers looking at the dissolver and the two chambers looking at the two respective storage tanks. This instrument gives some indication of the progressive steps in metal dissolving and can be used to follow the effectiveness of metal heel dissolution and preliminary cell cleanup operations.

Gallery Tanks and Chemical Headers

At the left of the gauge board is a 400-gallon weigh tank with connections to the nitric acid, sodium nitrate solution, and water headers; it is used for addition of the solutions required in coating removal and metal dissolving. Opposite this weigh tank is a 600-gallon fixed tank used to store water for an emergency drowning of the dissolving reaction. The outlet of this tank is

[redacted]

connected to the outlet line of the large weigh tank. A small 75-gallon scale tank, piped to the caustic and water headers, drains to the dissolver through a separate line.

Solution Storage Facilities

Section 4-R contains two metal solution storage tanks (4-7 and 4-8) (See Fig. 6) 8 ft. in diameter and 14 ft. high, with a capacity of 5100 gallons each. These tanks are not mechanically agitated, but are provided with a sparger consisting of a straight inlet tube with a slotted cap on the end. They are jacketed for water and steam.

Transfer lines to and from these tanks are shown in Figure 51 of Chapter II. Usually these tanks receive metal solution from the dissolvers and deliver it to Section 8 for extraction. A connection to Section 6 allows for the possible adoption of a pre-extraction decontamination step. A single weigh tank in the operating gallery is piped to both vessels and connected to the sulfuric acid header. Thus the proper concentration of sulfuric acid for complexing the metal can be added to each dissolver charge as it is received in storage.

Instrumentation of the storage tanks includes a temperature recorder, a liquid level and density recorder, and the usual contact and space microphones for following the operation of transfer jets. Each tank is connected to a sampler in the right-hand wall. Ionization chambers "looking" at each tank record on the Section 4 gauge board.

OPERATIONS

Coating Removal

Three metric tons (6600 lbs.) of metal are normally charged into the dissolver for coating removal. Handling multiple batches reduces the unit charging time and reduces the time for subsequent dissolving operations by providing a large amount of metal surface. The amount of metal charged is limited by the capacity of the dissolver for coating removal solution and the ability of the coils, jacket, and condenser to control the vigorous dissolving reaction.

During coating removal and dissolving operations the 291 Bldg. steam jet on the column off-gas line is operated to produce a minimum pressure difference of 10 inches of water between the cell and vessel at the time of the most vigorous reactions. When no reaction is occurring, the pressure difference is 20 to 30 inches of water.

The jacketed slugs in the dissolver are covered with 3850 lbs. of 26% $NaNO_3$ solution. The vent jet steam pressure is set at 100-105 lbs./sq.in. gauge and the column condenser water is adjusted to 80-120 gal./min. If the dissolver vacuum exceeds 20 inches of water, the seating of the dissolver lid

FIGURE 6

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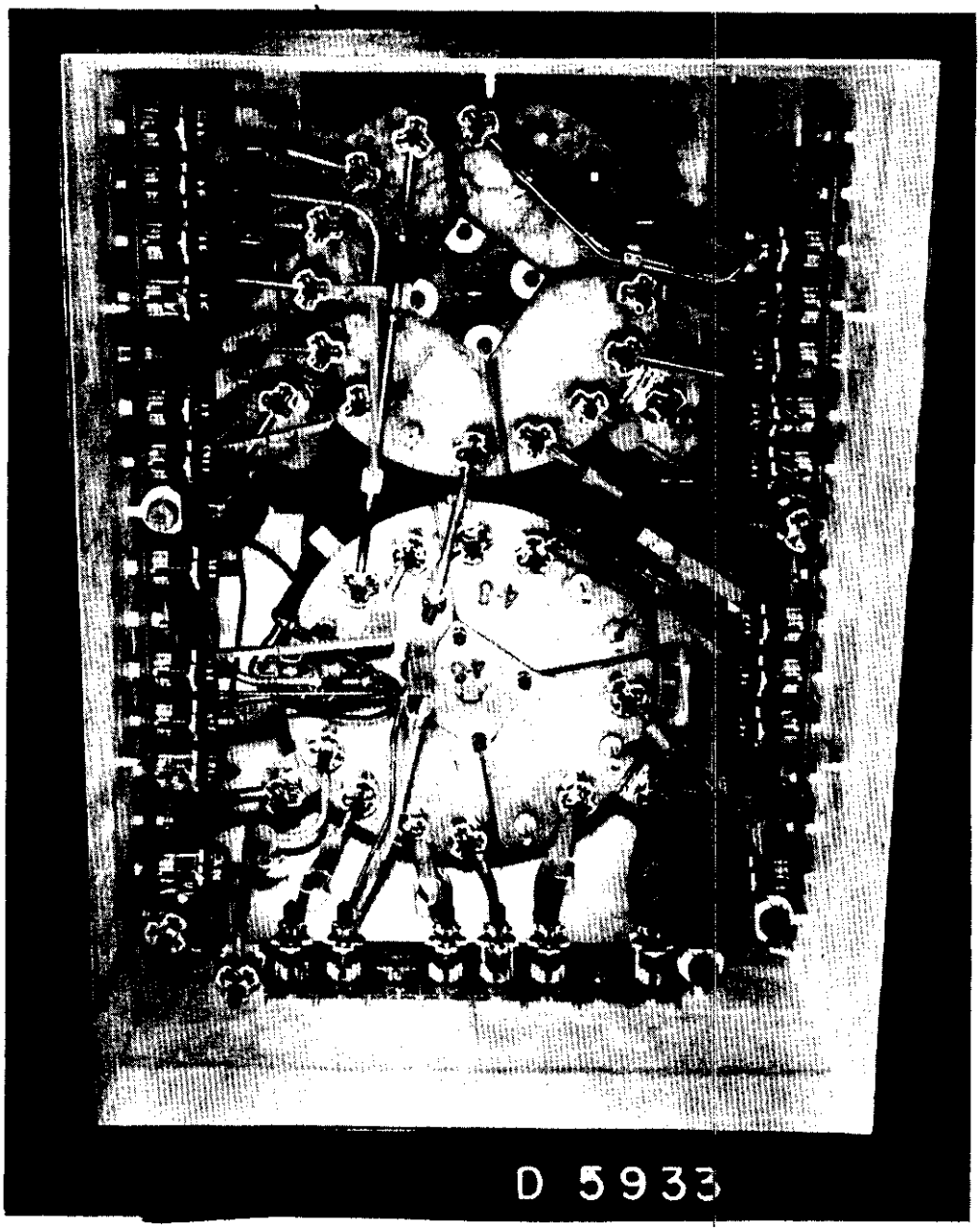
METAL SOLUTION STORAGE CELL



is considered satisfactory and the vessel is brought to boiling with steam on the coil and sparger. The sparger is then turned off but gentle boiling is continued with the coil to provide agitation during the addition of the caustic. A total of 1070 lbs. of 50% NaOH is added at a rate of 10 lbs./minute through an orificed line. During the addition of the first half of the caustic the heat of reaction appears as additional load on the condenser; the exit water and gas temperatures rise and the additional reflux causes a small pressure drop across the column. At this time boiling and agitation can be effected with only 2-10 lb. steam on the coil.

After the caustic addition is complete the contents of the vessel are refluxed for 2 hours to dissolve the thick end caps. The solution is cooled to 35°C and jetted to Section 15. An 800-lb. water wash is sparged for 10 minutes to flush out the alkaline heel and is then jetted to Section 15. This is followed by a 4300-lb. 5% HNO_3 wash which is sparged for 1 hour. Following removal of this acid wash, the metal dissolving step may be started.

Dissolver Operation

The possibility of contamination of the plant area with volatile by-products requires that satisfactory wind direction and velocity exist before starting to dissolve a charge of metal. Although the off-gas is diluted with 60,000 cu.ft./min. ventilation air at the base of the stack, a further dilution factor of 500 from stack to ground is necessary. Hence meteorological data are checked and a satisfactory forecast obtained before dissolving starts.

The vent jet is run at approximately 100 lb./sq.in. steam pressure during dissolver operation. This will draw a vacuum of 25-30 inches of water in the pot when no reaction is taking place. The condenser water is turned on to prevent acid and water vapor loss in the off-gas. Metal is dissolved in batches of approximately 2200 lb., using 5200 lbs. of 60-62% nitric acid. The amount of acid charged is not particularly critical, since errors affect only the amount of final solution. The amount of acid used is adjusted according to the length of time required to dissolve the third of three charges. If this period is greater than 8 hours, (start of heating until end point) the dissolver heel is increased by decreasing the acid charged; a period of less than 6 hours indicates an excessive heel. Dissolving times for the first two charges of a series are 5-6 hours.

Some heating with the steam coil is usually employed to start the reaction. In the early stages of a dissolving cycle, cooling with coil water may be necessary to keep the pot vacuum above 10 inches of water. As the reaction proceeds it becomes less violent due to dilution of the acid and therefore increased heating is necessary to keep the dissolver contents at the boiling point. The operation is stopped when the specific gravity of the solution as indicated on the inclined manometer reaches 1.82. To prevent further reaction which may lead to precipitation of basic salts in the dissolver, the solution is diluted to 50-55% UNH, cooled to 35°C, and jetted to storage where previously accumulated dissolver wash water dilutes it to approximately 40% UNH.

If unfavorable weather conditions develop during a run, the charge is cooled to 40°C with coil water and held until suitable off-gas dilution and disposal can be affected. The drowning charge in the gallery water tank can be used to stop the reaction quickly if emergency conditions arise.

A summary of data obtained during the first test dissolving with a starting charge of 3 tons of inactive metal in the 45-L vessel (T Canyon Building) is shown in Table I.

During the time between the completion of dissolving a three charge series and loading another, or during the time between dissolving charges of the same series, the 2500-lb. dissolver water wash is allowed to stand on the metal heel. This allows the use of coil cooling water for removing the heat of radioactive decay. The wash, which normally runs 5% UNH, is moved into the solution storage tank before the next loading or dissolving step.

No serious equipment corrosion problems are encountered under regular process conditions. The use of coil steam pressures over 30 lbs./sq.in. is not desirable, since laboratory tests have shown that at 50 lbs./sq.in. the heated metal surface is attacked quite seriously by the UNH-nitric mixture.

Metal Solution Storage

The metal solution from either 4-5L or 3-5R Dissolver is usually transferred to Section 4-R, Tank 4-8. The order of receipt of materials is (1) dissolver water wash from previous run, (2) metal solution, and (3) sulfuric acid from gallery tank. Under present procedure 800 lbs. of 93% sulfuric acid is added to each batch of solution equivalent to 1 long ton of metal. This sulfuric acid forms a uranyl sulfate complex with the metal and prevents its precipitation as a phosphate in the extraction step (Chapter V).

A series of three dissolver batches are usually blended in 4-7 Tank from which the desired quantities are furnished to Section 8 for extraction. Solution equivalent to 1 ton of metal and containing the dissolver wash, sulfuric acid, and jet dilutions from Dissolver 4-8 to 4-7 to 8-1 weigh approximately 13,000 pounds.

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TABLE I

SUMMARY OF DISSOLVER RUN DM-4
Dissolver 4-5L - Building 221 T

	Starting Period	Intermediate Running Conditions	Final Period
Time, Hours (approx)	0 to 1	1 to 5	5 to End (7)
Pot, °C	25 to 60	60 to 105	105
Sp. Gr. of Solution	1.35	1.35 to 1.65	1.65 to 1.81
Pot Vacuum, In. H_2O	28	28 to 12 to 18	18 to 23
Drop Across Column, In. H_2O	0	0 to 2.5	1.0
Temp Off-Gas, °C	Below 20	20 to 35	35
Off-Gas Flow, Cu.ft./min.	85	85 to 130	90
NO_2 in Off-Gas, %	3.5	35 to 45	10 to 8
HNO_3 in Stack Condensate, N	---	7.5	5.0
HNO_3 Loss in Stack Condensate, lbs./hr.	---	15	4
Dilution Factor NO_2 (vent to stack		1850 *	

3 Tons Inactive Slugs at Start (No Heel)

Vent Jet Steam Pressure 105 lb./sq.in.

Condenser Water Flow 75 gal./min.

*Stack sample taken via 292 Building line, 50 feet up stack; may not be representative.

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REFERENCES

Report CN 2021	Recommendations for Process Conditions at HEW, and Review of Process Chemistry
Report CN 1879	Progress Report on First Nine Months Operation of Separation Plant (Pilot Plant)
Report CN 566	Solution of Uranium Metal in Nitric Acid
Report CN 1871	Metal Solution
File No. 3-811	Required Wind Dilution for Reduction of Xe and I to Tolerance Levels
File No. 3-2278	(SE-PC-14) Some Density Values and Temperatures of UNH - HNO_3 - H_2SO_4 Mixtures
File No. 3-3003	(SE-PC-74) The Evolution of Iodine During Metal Dissolution

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APPENDIX

DISSOLVER PERFORMANCE CHARACTERISTICS

Prior to active operation of the T Canyon Building, a study was made of the operating characteristics of the dissolvers and off-gas system. A summary of the data obtained is presented below.

Water boil-up tests showed that with maximum vent jet steam pressure and condenser water flow, 50 lb./sq.in. steam on the coil would effect sufficient boil-up to produce excessive column pressure drop. Summaries of water and acid boil-up data are given in Tables II and III. The rate of heating in the dissolver with the steam sparger is much greater than with the coil. As mentioned before, the jacket is not very effective for heating or cooling.

The capacity of the dissolver vent jet is approximately 175 cu.ft./min. (air) when operated at 105-115 lbs/sq.in. steam pressure, assuming a dry column and no vacuum in the vessel. If the dissolver lid is in place and sufficient liquid is charged to seal the ends of the dip tubes, a vacuum of 25 to 30 inches of water will develop and the air leakage of only 60-90 cu.ft./min. will be removed by the vent jet. The relation between vent jet steam pressure, pot vacuum, and air leakage into the dissolver for the T Canyon Building vessels is given in Tables IV, V, and VI and Figures 7, 8, and 9. Figure 10 shows the off-gas line orifice characteristics.

The liquid level in the dissolver determines to a considerable extent the maximum vacuum obtainable, since air can enter via the dip tubes. Table VII and Figure 11 show the relation between pot vacuum and liquid level at maximum vent jet steam pressure. In general, a high vacuum indicates a close fitting lid and low air leakage into the vessel.

FLOW SHEET

The process flow sheet, based on removing the jackets from three metric tons of slugs and dissolving one metric ton of metal, is given as Table VIII.

OPERATING LOG FORMS

The following operating log forms given at the end of the appendix constitute the detailed operating instructions by which the operators perform the various steps of charging the dissolver, removing the coating, dissolving the metal and adding the sulfuric acid in metal solution storage.

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APPENDIX

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TABLE II

Water Boil-Up Tests - 221-T Dissolvers

Equilibrium Conditions After 2 Hours Operation

	3-5L*	3-5R	4-5L
Wt. Water Charged, lb.	5080	4150	4150
Steam Coil Pressure, lb./sq.in.	50	50	40
Pot Temperature, °C	99	99.5	99.5
Condenser Water Flow, gal./min.	80	76	82
Exit Water Temperature, °C	51	48	53
Off-Gas Temperature, °C	29	33	44
Vent Jet Pressure, lbs./sq.in.	108	108	104
Pot Vacuum, In. Water	23.6	9.5	15.7
Column Pressure Drop, In. Water	12.5	16.5	10

*Lid Repaired

Notes: Time to heat Dissolver 3-5R from 25 to 100°C, 20 min. with 32 lb./sq.in. steam on coil

Time to cool Dissolver 3-5R from 100 to 50°C, 22 min. with 40-60 gal./min. water rate on coil

Time to heat 3-5L Dissolver from 25 to 100°C, 20 min. with 50 lb./sq.in steam on coil.

Time to cool 3-5L Dissolver from 100 to 35°C, 20 min. with 60 gal./min. water rate on coil

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APPENDIX

TABLE III (SUMMARY)

Nitric Acid Boil-Up Tests - 221-T Dissolvers

Equilibrium Conditions After 1 1/2 Hours Boiling

	3-5L	3-5R	4-5L
Wt. 60% HNO_3 Charged, lbs.	5400	5400	5400
Steam Coil Pressure, lbs./sq.in.	23	18	17
Pot Temperature, °C	115	117	115
Condenser Water Flow, gal./min.	67	67	67
Exit Water Temperature, °C	22	< 20	21
Off-Gas Temperature, °C	22	< 20	<21
Vent Jet Pressure, lbs./sq.in.	107	104	105
Pot Vacuum, In. Water	24.5	20.8	24
Column Pressure Drop, In. Water	0.5	1.0	1
Off-Gas Flow, cu.ft./min.	85	93	90
Max. Off-Gas Loss HNO_3, lb./hr.	0.6	0.2	0.4

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TABLE IV

Pot Vacuum vs. Air Leakage into 3-5L Dissolver

After Grinding-in Lid

4730 lb. Water (Equivalent to Coating Removal Level)

Steam Pressure, lbs./sq.in.	Vacuum at Jet, Inches Water	Pot Vacuum, Inches Water	Air Leakage, cu.ft./min.
101	37	27.5	70
94	31.5	26.5	61
74	26	23.6	43
58	21.5	20.4	30
52	20	18.2	27
43	15.5	14.4	24
34	12	11.2	20
25	9.8	8.7	17
19	7.0	6.1	12
9	4.3	3.4	10

5610 lb. Water (Equvalent to Metal Solution Level)

102	35.0	30	63
91.5	32.8	29.6	48
78	29.5	26.9	41
67	24.5	23.4	32
56	21.0	19.6	28
48	18.0	16.9	24
44	15.5	14.8	22
34	12.5	11.8	20
24	9.0	8.6	17
16	6.0	5.9	12

[redacted]

[redacted]

APPENDIX

DECLASSIFIED

TABLE V

Pot Vacuum vs Air Leakage into 3-5R Dissolver

4630 lb. Water (Coating Removal Level)

Steam Pressure lbs/sq.in. Gauge	Vacuum at Jet, Inches Water	Pot Vacuum, Inches Water	Air Leakage, cu.ft./min.
112	34	24.4	93
94	29	21	86
79	25.3	18	80
64	21	15	69
53	16.8	12	63
40	14	9	53
28	9.5	6	42
17	5	3	22

5840 lb. Water (Metal Solution Level)

106	33.3	25.5	26
99	31.5	23.7	84
83	27.8	20.5	79
69.5	23	17.6	72
58	16	14.8	64
44	14.5	11.7	56
32	10.5	8.7	47
22	7.5	5.9	34
9	4.3	3.0	22

[redacted]

[redacted]

APPENDIX

TABLE VI

Pot Vacuum vs Air Leakage into 4-5L Dissolver

DECLASSIFIED

Steam Pressure, lbs/sq.in. Gauge	Vacuum at Jet, Inches Water	Pot Vacuum, Inches Water	Air Leakage, cu.ft./min.
4535 lb. Water (Coating Removal Level)			
112	32.5	25.5	110
79	25.5	21	75
68	21.0	18	69
58	17.5	15	62
47	14.5	12	56
36	11.5	9	47
21	7.0	6	37
11	3.8	3	22
5910 lb. Water (Metal Solution Level)			
112	36	30	85
96	32	27	76
85	28	24	71
74	24.5	21	66
64	20.3	18	62
54	17.0	15	56
44	13.8	12	48
34	11.0	9	42
23	6.8	6	33
11	4.0	3	20
5810 lb. Water (Metal Solution Level)			
112	35.5	29.6	90
96	31	27.0	76
85.5	28.25	24	72
76	24.5	21	66
65.5	20.75	18	63
54.5	18.5	15	57
44	13.75	12	47
34	10.5	9	42
24	7.5	6	32
11.0	4.0	3	20

[redacted]

FIGURE 7

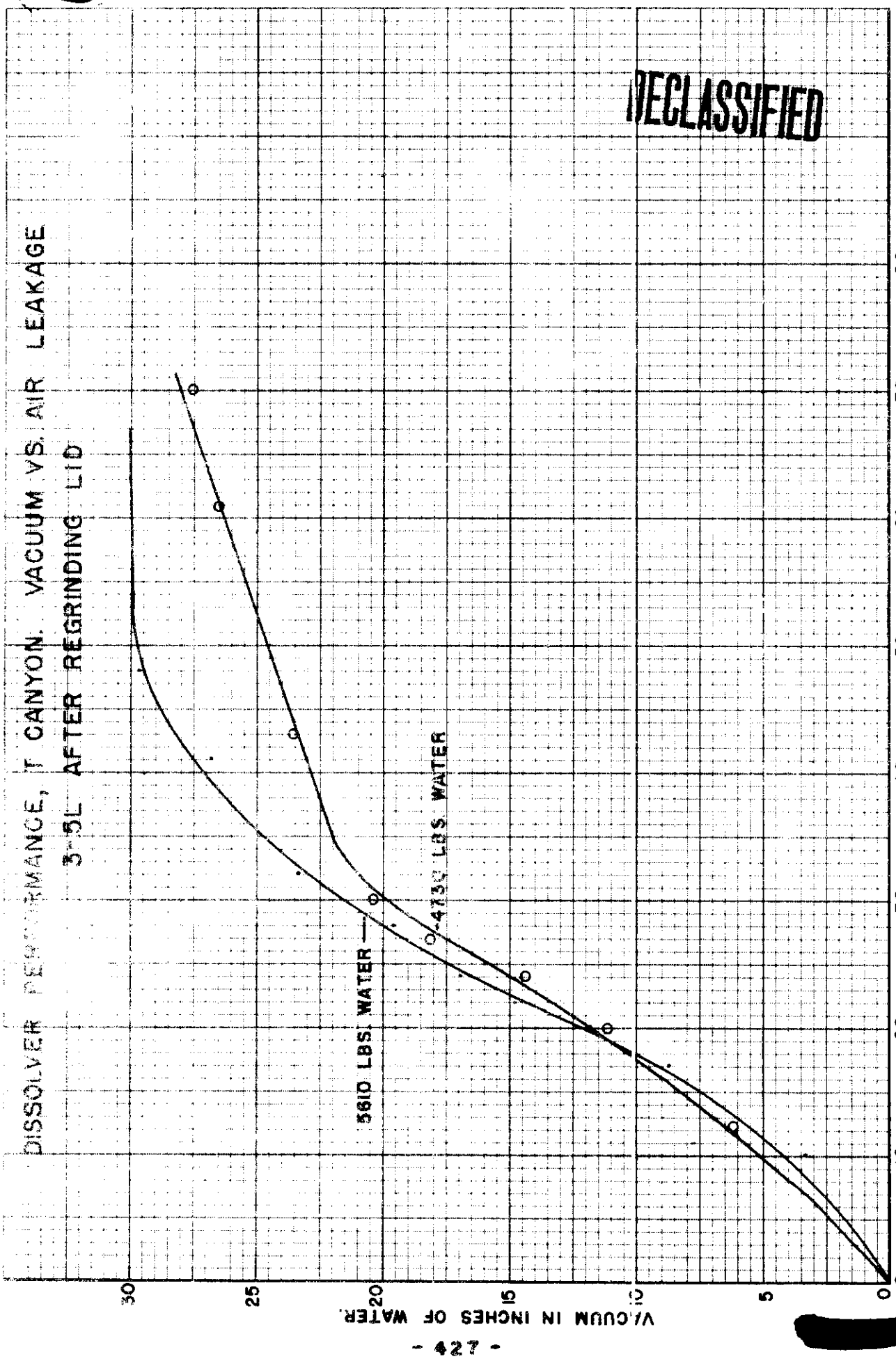

DECLASSIFIED
DISSOLVER PERFORMANCE, T CANYON. VACUUM VS. AIR LEAKAGE
3-5L AFTER REGRINDING LID
5810 LBS. WATER —
0-4134 LBS WATER
VACUUM IN INCHES OF WATER
AIR LEAKAGE INTO DISSOLVER — CU. FT./ MIN.
0
5
10
15
20
25
30
0
10
20
30
40
50
60
70
80

FIGURE 8

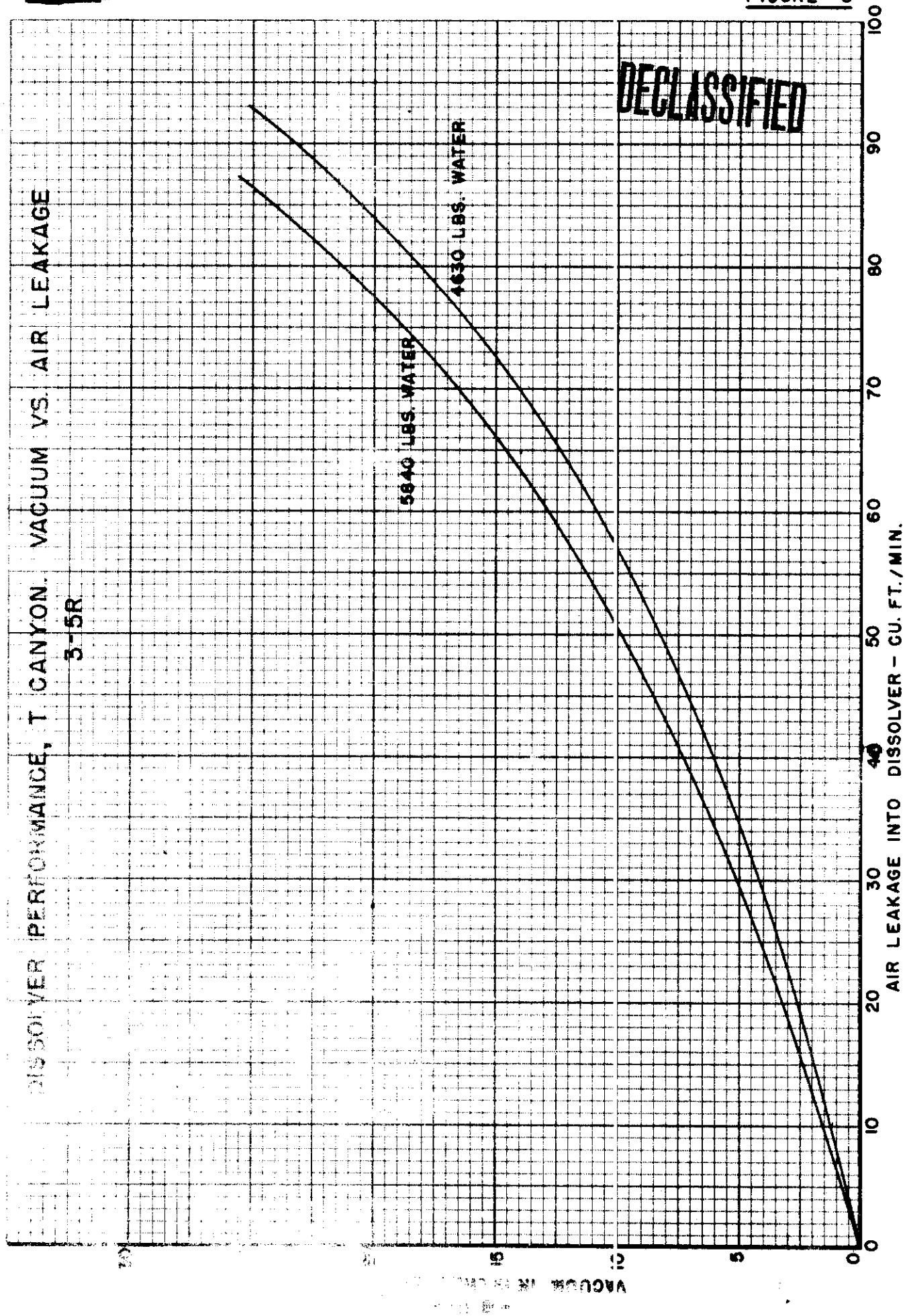
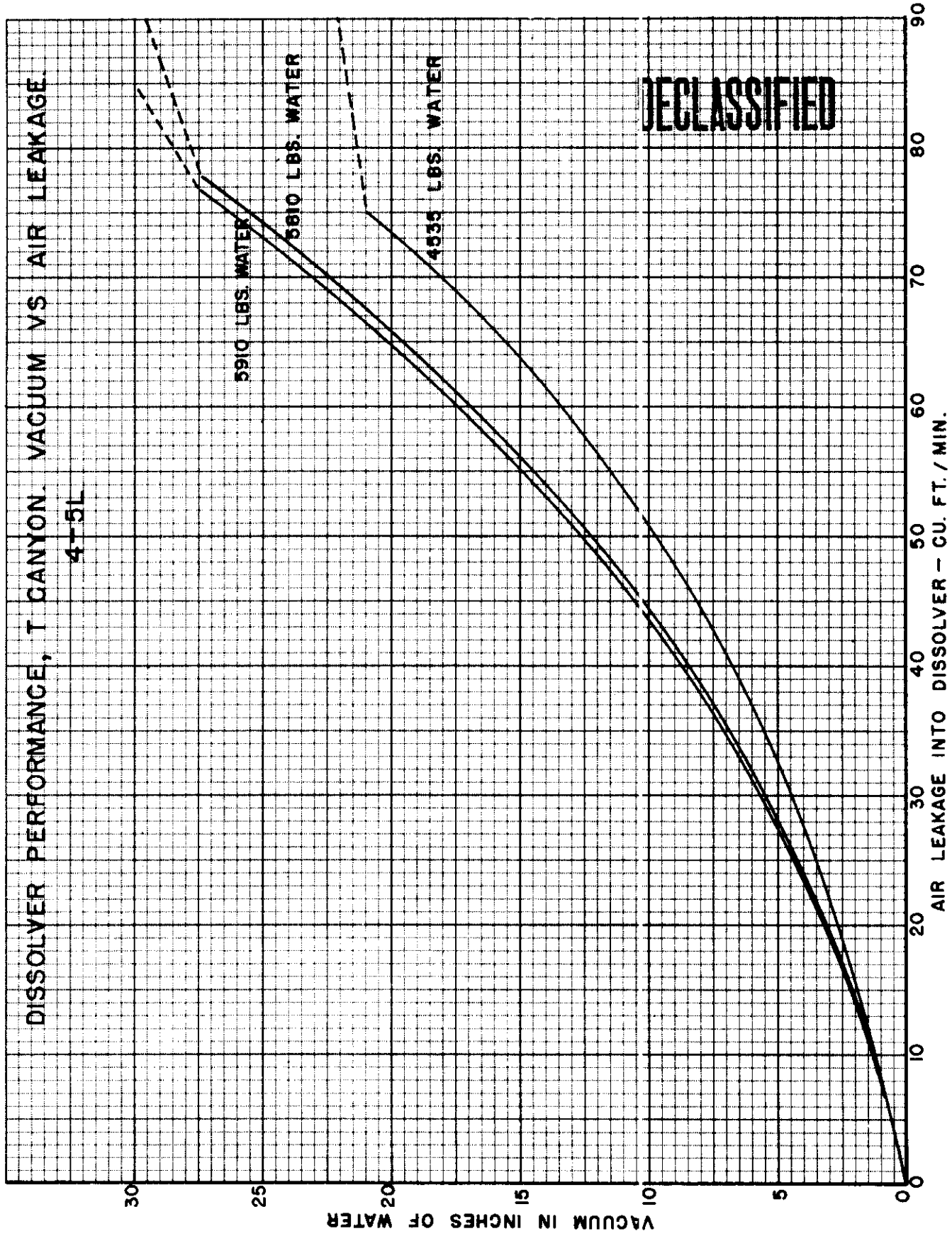

DECLASSIFIED
DISSOLVER PERFORMANCE, T CANYON. VACUUM VS. AIR LEAKAGE
3-5R
5840 LBS. WATER
4630 LBS. WATER
VACUUM IN INCHES OF MERCURY
AIR LEAKAGE INTO DISSOLVER - CU. FT./MIN.
0
5
10
15
20
30
40
50
60
70
80
90
100

FIGURE 9



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[redacted]

FIGURE 10

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ORIFICE CALIBRATION

291-T DISSOLVER OFF-GAS LINE

(PIPE DIAMETER 3.260 IN., ORIFICE DIAMETER 2.232 IN., FLANGE TAPS, TEMP. 68°F, PRESSURE 14.5 LBS./SQ.IN.)

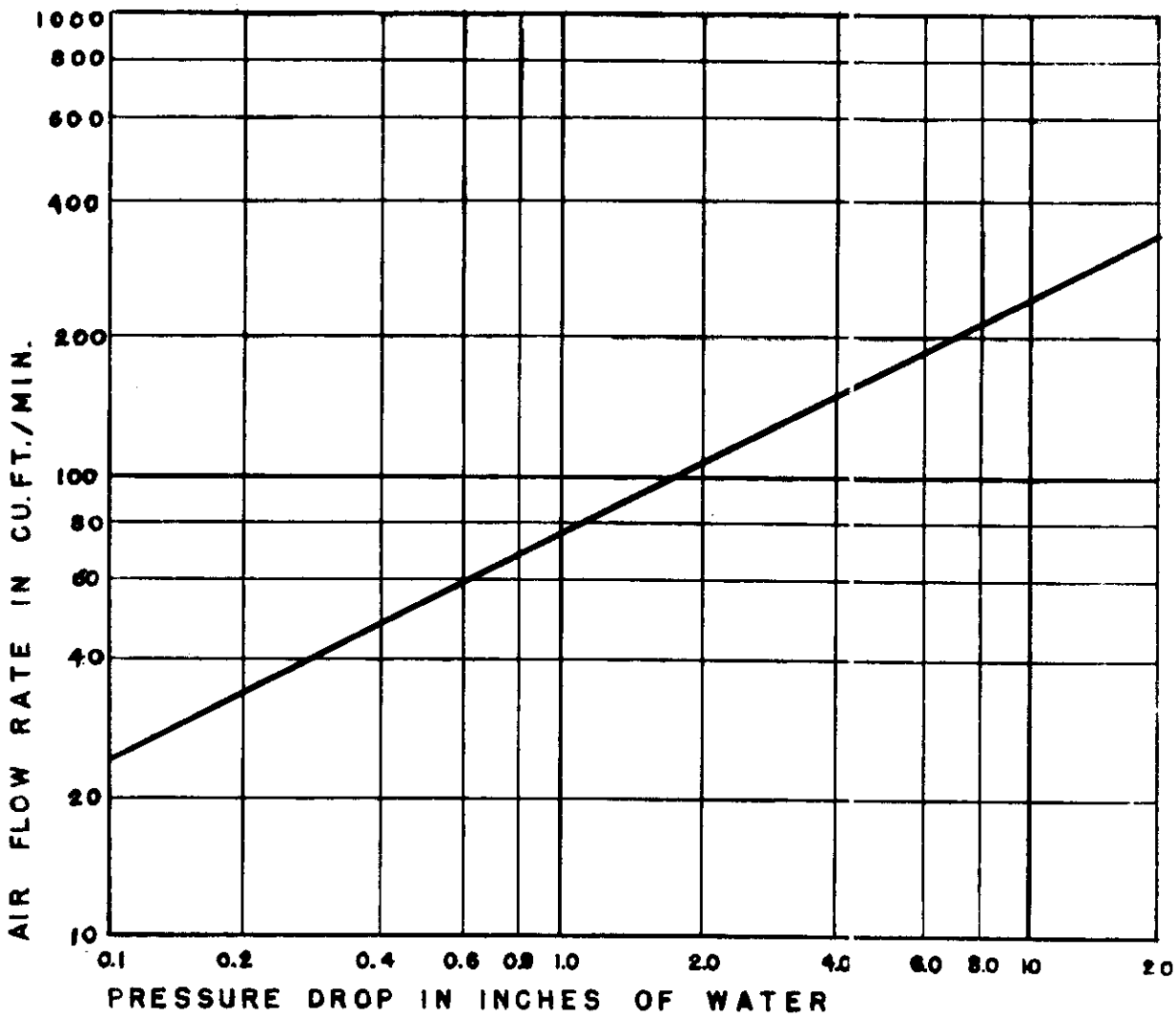

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TABLE VII

Effect of Liquid Depth on Pot Vacuum

Water in Vessel (lbs.)	Steam Pr. on Vent Jet, lbs/ sq.in. Gauge	Max. Vacuum, Inches Water	Air Leakage into Vessel, cu.ft./min.
	Dissolver 3-5L (Before Grinding Lid)		
300	108	5.2	---
2030	108	7.2	---
3960	108	7.0	---
4880	109	7.5	164
5960	108	7.1	---
8240	108	7.5	---
	Dissolver 3-5L (After Grinding Lid)		
353	108	14	---
2100	108	20.5	---
4730	101	27.5	70
5610	102	30	63
7432	108	30	---
	Dissolver 3-5R		
300	108	7.5	---
2030	108	14.4	---
4050	108	21.6	---
4630	112	24.4	93
5840	106	25.5	86
6000	108	24.2	---
8100	108	24.8	---
	Dissolver 4-5L		
900	112	9.0	---
2676	112	15.5	---
4535	112	23.5	110
4565	112	23.1	---
5810	112	29.6	90
5910	112	30	85
7430	109	29.0	---

[redacted]

FIGURE 11

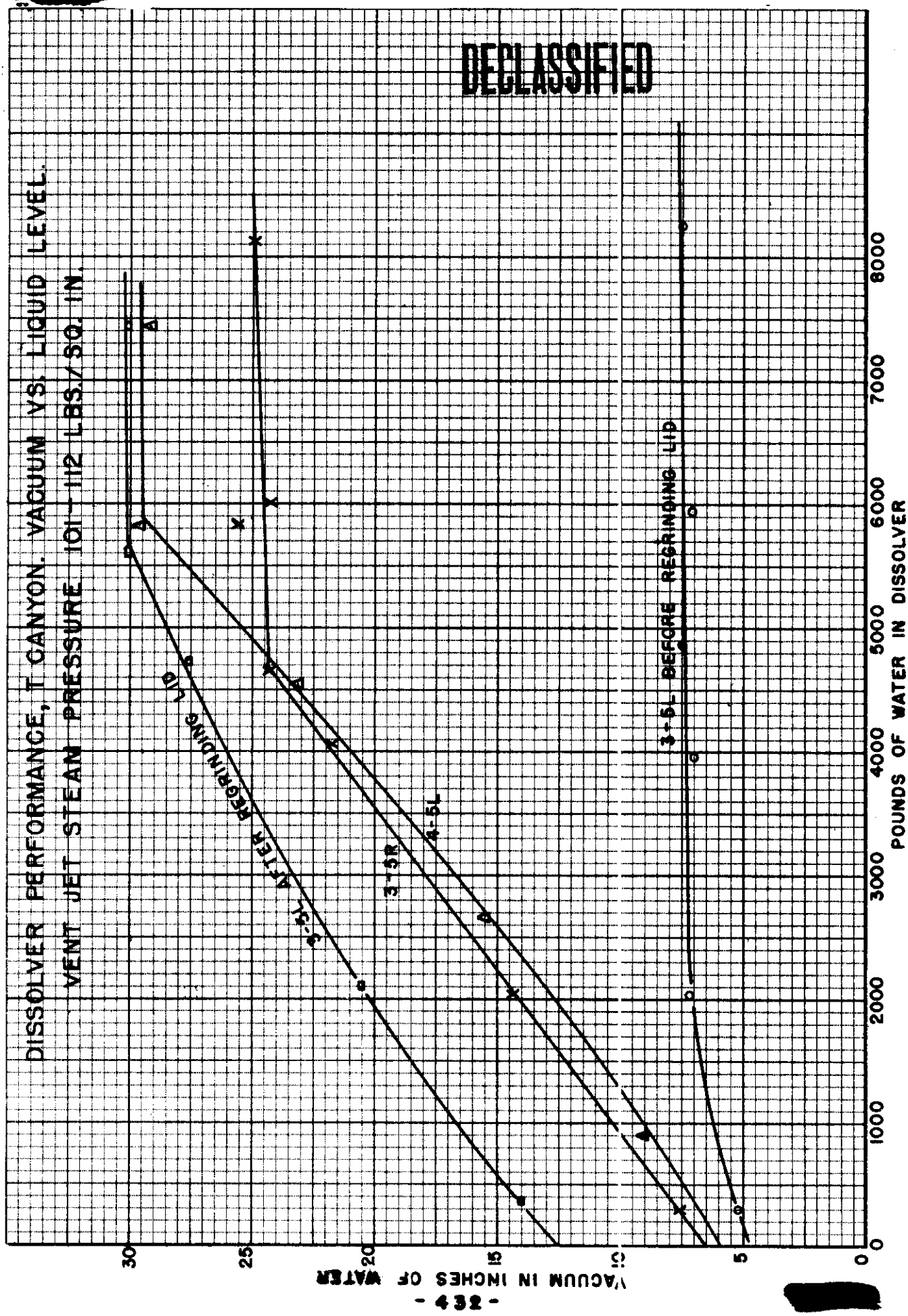
DECLASSIFIED
DISSOLVER PERFORMANCE, T CANYON. VACUUM VS. LIQUID LEVEL.
VENT JET STEAM PRESSURE 101 - 1/2 LBS./SQ. IN.
3-5L BEFORE REGRINDING LID
3-5R
4-5L
AFTER REGRINDING LID
VACUUM IN INCHES OF WATER
POUNDS OF WATER IN DISSOLVER
0
5
10
15
20
25
30
1000
2000
3000
4000
5000
6000
7000
8000

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TABLE VIII

Preparation of Metal Solution

Sections 3 and 4

A. Coating Removal (Basis - 3 charges of 2200 lb. each)

1. Charge 6600 lb. active jacketed slugs (8 buckets of 105 pieces each).
2. Add 3850 lb. (389 gal.) of 26% $NaNO_3$ to Dissolver 3-5R or 4-5L.
3. Heat dissolver to boiling and add 1070 lb. (85 gal.) of 50% NaOH solution at 10 lb./minute.
4. Digest for two hours at boiling temperature.
5. Cool to 35°C and jet to waste receiver 15-7.
6. Flush dissolver with 835 lb. (100 gal.) water. Air sparge 10 min.
7. Jet water to 15-7 Tank.
8. Add 4300 lb. (500 gal.) of 5% HNO_3 solution and sparge with air for one hour.
9. Jet acid wash to 15-7 Tank.

Solution Composition in Dissolver

(Assume 210 lb Al, 50% to NH_3 reaction 50% to $NaNO_2$ reaction)

	Lbs.	%
NaOH	282	5.5
$NaNO_3$	380	7.4
$NaAlO_2$	638	12.5
Na_2SiO_3	6	0.1
$NaNO_2$	402	7.9
H_2O	3402	66.5
	5110 (532 gal)	

TABLE VIII
(Cont.)

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B. Metal Dissolution (Basis 2200 lb. Active Metal)

1. Add 5400 lb. (480 gal.) of 60% HNO_3 solution to the dissolver.
2. Bring dissolver to boiling temperature (100-110°C).
3. Hold dissolver at boiling temperature until specific gravity of solution reaches 1.82.
4. Add 2500 lb. (300 gal.) water to dissolver.
5. Sparge with air and cool to 40°C.
6. Jet metal solutions to storage Tank 4-8.
7. Add 2500 lb. (300 gal.) water to dissolver.
8. Hold water on metal heel until ready to dissolve the next charge, then jet it to 4-8.

Note: Off-Gas No NO present in off gas at vent jet.
1420 lb. NO_2 evolved
(11,100 C.F. S.T.P.)

Solution Composition in Dissolver

	Lbs.	%
HNO_3	134	1.5
UNH	4640	52.0
H_2O	4156	46.5
	8930 (750 gal.)	

C. Metal Solution Storage

1. Receive 2575 lbs. (309 gal.) wash water from dissolver into 4-8.
2. Receive 9120 lb. (773 gal.) of metal solution from dissolver into 4-8.
3. Sparge with air and add 800 lb. 93% H_2SO_4 to 4-8.
4. Jet 4-8 to 4-7.
5. Jet to 8-1 as needed.

Solution Composition in 4-7

	Lbs.	%
HNO_3	134	1.1
H_2SO_4	744	5.9
UNH	4640	36.8
H_2O	7082	56.2
	12600 (1120 gal)	

[redacted]

DISSOLVER OPERATION

Date _____
Run No. _____

DECLASSIFIED

I.- CHARGING DISSOLVER

1. When approved by the dispatcher, jet the wash water from the preceding run from 4-5L (3-5R) to 4-8. (See page 2.1, step 2)

   Time start____Time end_____

2. Beckman reading on 4-5L (3-5R)

   Meter _____Factor_____

3. See that the vent exaust jet is operating at 100 to 105 lbs. per sq. in. and that there is a negative cell pressure before the cell cover blocks are removed.

   Steam Pressure _____

   Differential pressure_____

   Cell pressure _____

4. See that water is turned on the column condenser (50 GPM) and pot coil (50 GPM).

   Condenser water flow____GPM
   Cold water flow _____GPM

5. Notify the dispatcher that charging is ready to start.

   Time called _____

6. Charge the dissolver with the specified number of buckets of slugs. Check with the crane operator for bucket numbers and enter data below.

   Note: If charging operation is interrupted for more than 12 hours, add 1000 lbs. of 26% Sodium Nitrate solution (Line L) to the dissolver via 4-5LA (3-5RB).

   Lbs added_____Time added____

[redacted]

DECLASSIFIED

[redacted]

DISSOLVER OPERATION

Date_____
Run No._____

I - CHARGING THE DISSOLVER (Cont.)

9. When all the metal has been charged, read and record the dissolver differential pressure. See that this pressure is above the minimum for the conditions listed below before the cell cover blocks are replaced.

4-5L (3-5R) Differential Pressure _____
Steam pressure_____

	Minimum Differential Pressures
A. All metal charged, dissolver cover on	- 7 ins.
B. All metal charged, dissolver cover on, 1000 lbs. of Nitrate solution in pot	- 10 ins.
C. All metal charged, dissolver cover on, 3850 lbs. of Nitrate solution in pot	- 20 ins.

10. Beckman reading on 4-5L (3-5R). Meter_____ Factor_____

11. Add sufficient 26% Sodim Nitrate solution (Line L) to 4-5L (3-5R) via 4-5LA (3-5RB) to make 3850 lbs. of solution. The solution in the dissolver is to be maintained below 30°C until ready for the coating removal step.

Lbs. added_____ Time added _____

4-5L / 3-5R Wt. Ftr._____Lbs._____

4-5L / 3-5R Sp. Gr._____ Temp._____

[redacted]

[redacted]

DISSOLVER OPERATION

Date_____
Run No._____

DECLASSIFIED

II - COATING REMOVAL

1. Beckman reading on 4-5L (3-5R) — Meter_____ Factor_____

2. Turn on 4-5L (3-5R) air sparger and increase cooling water on condenser coil to 80-120 G.P.M. — Air sparger on_____ Condenser water flow _____ GPM

3. See that 291 Bldg. fans are operating. — Time_____

4. See that all drain valves on tank 4-5LE (3-5rd are closed. — Time_____

5. Bring 4-5L (3-5R) to boiling (100-105°C.) using steam on the coil and sparger. — Time start_____ Time boiling_____

6. Turn off steam sparger and continue to boil 4-5L (3-5R) with 2 - 10 lbs. of steam on the coil. — Steam sparger off_____

7. Add 1070 lbs. of 50% Sodium Hydroxide (Line Q) to 4-5L (3-5R) via 4-5LE (3-5RD) at the rate of 10 lbs. per min. Add in two portions of 535 lbs. each. Regulate the steam on the coil so that the column pressure drop does not exceed 10 inches of water. DO NOT EXCEED THE 10 LB. PER MINUTE ADDITION RATE — Time start_____ 1st Port._____ Lbs. Time end_____ Time start_____ 2nd Port_____ Lbs. Time end_____ Minimum Pot Vacuum _____ Maximum Column Pressure_____

   Note: If the pot vacuum falls below 15 inches of water during this step, stop Caustic addition, turn off steam and turn on coil cooling water until vacuum is regained.

8. Flush 4-5LE (3-5RD) to 4-5L (3-5R) with 150 lbs. of process water. — Lbs. added_____ Time added_____

9. Digest 4-5L (3-5R) at boiling (100-108°C) for 2 hours. — Time start_____ Time end_____

10. Turn on 4-5L (3-5R) air sparger and coil cooling water and cool to 35°C. — Air sparger on_____ Coil water on_____ Time 35°C_____

11. Call dispatcher and obtain permission to jet coating solution from dissolver to 15-7. — Time called_____ Time jetted_____

 [redacted]

[redacted]

DISSOLVER OPERATION

Date_____
Run No._____

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II - COATING REMOVAL (Cont.)

12. Add 835 lbs. of water to 4-5L (3-5R) via 4-5LA (3-5RB). — Lbs added____ Time added____

13. Air sparge 4-5L (3-5R) for 10 minutes. — Time start____ Time end____

14. Call dispatcher and obtain permission to jet wash solution from 4-5L (3-5R) to 15-7. Shut off air sparger when the Wt. Ftr. reading reaches 0.2. — Time called____ Time jetted____ Air sparger off____

15. Add 355 lbs. of 60% Nitric Acid (Line A) to scale tank 4-5LA (3-5RB) add 2345 lbs. of water to 4-5LA (3-5RB) on top of acid. — Lbs. added____ Time added____ Lbs. added____ Time added____

16. Air sparge 4-5LA (3-5RB) for 10 min. — Time start____ Time end____

17. Add entire batch from 4-5LA (3-5RB) to 4-5L (3-5R). Turn off air sparger. — Lbs. added____ Time added____ Air sparger off____

18. Flush 4-5LA (3-5RB) to 4-5L (3-5R) with 1600 lbs. of water. — Lbs. added____ Time added____

19. Air sparge 4-5L (3-5R) for one hour, keeping temperature below 35°C. Leave air sparger on. — Air sparger on____ Time end____

20. Call dispatcher and obtain permission to jet wash from 4-5L (3-5R) to 15-7. Shut off air sparger when the Wt. Ftr., reading reaches 0.2. — Time called____ Time start____ Time end____ Air Sparger off____

[redacted]

[redacted]

Date_____
Run No._____

DECLASSIFIED

III - METAL DISSOLVING

1. See that tank 4-5F (3-5C) contains 4000 lbs. of water. This water is to be used for killing the charge if all other means fail to stop the reaction during an emergency, and it is necessary to sacrifice a run to protect personnel and equipment.

   Lbs water in 4-5F (3-5C)_____

   IN THE EVENT OF A POWER FAILURE DURING THE DISSOLVING OPERATION WHICH FOLLOWS:

   A. Shut off steam to coil
   B. Turn cooling water on coil and jacket and bring charge to 30°C.
   C. Notify the dispatcher

2. See that the 291 Bldg. fans are operating.

   Time_____

3. Notify the dispatcher that metal dissolving is ready to start, and obtain permission to jet the wash water in 4-5L (3-5R) to 4-8. (For a charge immediately following coating removal, the wash is jetted to 15-7. See page 1.4 step 20)

   Time called_____

   Time start_____Time c[illegible]

4. Beckman reading on 4-5L

   Meter_____ Factor_____

5. Turn on the coil cooling water and see that the condenser cooling water flow to 80 - 120 GPM.

   Coil cooling water on____GPM
   Condenser water flow____GPM

6. Add 5200 - 5500 lbs. of 60% Nitric Acid (Line A) to 4-5L (3-5R) in two portions (3000 and 2000 lbs.) via 4-5LA (3-5RB).

   1st Port.____Lbs. Time Added___
   2nd Port.____Lbs. Time added___
   Total lbs. added_____

7. After all the nitric Acid is added, turn off coil water and start heating with 20 lb. steam on the dissolver coil. Start data record on page 1.7 when the steam is turned on. Keep the vent jet at 100-105 psi. Run reaction as fast as possible not permitting the pot vacuum to drop below 10 ins. At times cooling water will be required in the coil, at other times steam up to 20 psi, will be required as explained in the note which follows.

   Time steam on_____
   Time reaction starts_____

   Minimum pot vacuum____Time____

   Maximum column pressure__Time___

[redacted]

[redacted]

DISSOLVER OPERATION

Date _____
Run No. _____

DECLASSIFIED

III - METAL DISSOLVING (Cont.)

NOTE: The first run of a triple charge will usually start reacting at about 60-70°C. at which point the steam should be shut off. The second run will usually start a little higher while with the third run, it may be necessary to keep the staam on during the entire cycle. In any case, the reaction at the outset will be more vigorous than later. During the initial period the reaction will, or may, have to be cooled in order to maintain 10 in. vacuum during the latter period the reaction will become sufficiently slow that even with 20 lb. steam on the coil, the pot vacuum will increase.

8. Record the maximum vent gas temperature and the inlet and outlet condenser water temperature and flow at the time the temperature is read. Hold the off gas temperature below 50°C. by adjusting the steam or water flow to the coil.

Max. vent gas Temp. _____ Time ____
Condenser water temperature:
Inlet _____
Outlet _____
Flow _____ GPM

9. Digest at 100-115°C. until a Sp.Gr. of 1.81 (by manometer) is reached. The temperature during digestion must be maintained at boiling. With the first run of a triple charge this gravity is usually reached in about 5 hours, the second in about 6 hours and the third in about 8 hours.

Time start _____ Time end _____

10. Add 2500 lbs. of water to 4-5L (3-5R) via 4-5LA (3-5RB). Turn on the air sparger and cool to 40°C with water on the coil.

Lbs. added _____ Time added _____
Air Sparger on _____
Coil water on _____
Time start _____ Time 40°C _____

11. Shut off the air sparger and read the Wt. Ftr., Temp. and Sp. Gr. when the Sp. Gr. meter becomes constant (both manometer and Ring Balance) then turn on the air sparger again.

Air Sparger off _____
4-5L Wt. Ftr. _____ Lbs. _____
3-5L
4-5L Sp. Gr. _____ Temp. _____
3-5R
Air sparger on _____

12. Call dispatcher and obtain permission to jet 4-5L (3-5R) to 4-8

Time called _____
Time start _____ Time end ____

13. Beckman reading on 4-5L (3-5R)

Meter _____ Factor _____

[redacted]

[redacted]

DISSOLVER OPERATION

Date _____
Run No. _____

DECLASSIFIED

III - METAL DISSOLVING (Cont.)

Record data at 20 min. intervals for first 2 hours; then at hourly intervals.

TIME	COIL		POT VACUUM INS.	COLUMN ΔP IN	COND. WATER GPM	VENT GAS TEMP	EXIT WATER TEMP.	MANOMETER SP.GR.
	WATER GPM	STEAM PSI						

[redacted]

DISSOLVER OPERATION

Date_____
Run No._____

DECLASSIFIED

III - METAL DISSOLVING (Cont'd)

14. Add 2500 lbs. of water to 4-5L (3-5R) via 4-5LA (3-5RB)

    Lbs. added_____ Time added_____

15. Air sparger 4-5L (3-5R) for 15 minutes and shut off the air sparger and turn on the coil cooling water. Allow the wash water to remain in the dissolver until the next run is ready to start Maintain the temperature below 30C.

    Air sparger on_____

    Time start_____ Time end_____

    Air sparger off_____

    Coil water on____ Flow_____ GPM

16. Notify the dispatcher that the run is completed.

    Time called_____

[redacted]

[redacted]

Date _____
Run No. _____

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I - METAL SOLUTION STORAGE

1. Beckman reading on 4-8 — Meter_____Factor _____

2. Receive wash water from preceding dissolver run into 4-8 from 4-5L (3-5R) (TW 2580 lbs.) — 4-8 Wt. Ftr._____ Lbs. _____ Time start_____Time end _____ 4-8 Wt. Ftr._____ Lbs. _____ Wt. received in 4-8 _____

Note: After receiving the wash water from 4-5L (3-5R) so that a dissolving run may proceed several hours will elapse, depending on the number of the run, before receipt of the metal solution. DO NOT PROCEED UNTIL IT HAS BEEN RECEIVED.

3. When approved by the dispatcher, receive metal solution into 4-8 from 4-5L. (3-5R) (TW from dissolver 2580 / 9120 lbs.) — Time start_____ Time end _____ 4-8 Wt. Ftr._____Lbs. _____

4. Turn on 4-8 air sparger — 4-8 Air sparger on _____

5. Add 940 lbs. of 93% Sulfuric Acid (Line K) to 4-8 via 4-8B. — Lbs. added_____Time added _____

6. Beckman reading on 4-8. — Meter_____Factor _____

7. Shut off 4-8 air sparger and read Wt. Ftr., Temp. and Sp.Gr. when Sp.Gr. meter becomes constant then turn on 4-8 air sparger. — 4-8 Air sparger off _____ 4-8 Wt. Ftr._____Lbs. _____ 4-8 Sp. Gr._____ Temp. _____ 4-8 Air sparger on _____

8. When wash water, metal solution, and acid are in correct proportions, call dispatcher, give charge data and obtain permission to jet 4-8 to 4-7. Shut off 4-8 air sparger when the Wt. Ftr. reaches 0.2. — Time called _____ Time start_____Time end _____ 4-7 Wt.Ftr._____Lbs. _____ 4-7 Sp.Gr._____Temp. _____ 4-8 Air sparger off _____

9. Beckman reading on 4-8 — Meter_____ Factor_____

10. Air sparge 4-7 for 1 hour before jetting a run to 8-1. Leave air sparger on. — 4-7 Air sparger on _____ Time end _____

11. When notified by the dispatcher, jet 18,800 lbs. of metal solution from 4-7 to 8-1. (Approx. 19,600 lbs. when received in 8-1) "See page 3.1 step 4".

 [redacted]

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CHAPTER IV - PREPARATION OF METAL SOLUTION

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HANFORD ENGINEER WORKS TECHNICAL MANUAL

SECTION C - SEPARATIONS

CHAPTER V - EXTRACTION

The extraction of product from the metal solution is one of the basic steps of the separation process. The product is removed from the bulk of uranium solution by precipitation with a bismuth phosphate carrier. Many of the fission elements are removed with the product, these are separated out by decontamination cycles subsequent to extraction. The process and the equipment used in the extraction step are described in this chapter.

[redacted]

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CHAPTER V - EXTRACTION

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THE EXTRACTION PROCESS

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The product is extracted from 19,600-pound batches of UNH (35.5%) containing 1120 pounds of H_2SO_4 and representing 3300 pounds of active metal in solution.

The metal solution, containing sulfuric acid to complex the uranium and thus prevent its subsequent precipitation as uranyl phosphate (See Chapter I) is transferred from storage to a precipitator and diluted to 25% UNH. A pre-extraction treatment with sodium nitrite is made at 75 - 85°C to insure that the product is in a form which can be carried by the bismuth phosphate.

The extraction precipitate (direct, or phosphoric acid strike) is made by first adding bismuth solution and then phosphoric acid, uniformly over 3 hours, to the UNH solution maintained at 85°C with thorough agitation. The slurry is digested for 2 hours at 85°C with continued agitation.

After cooling, the slurry is centrifuged and the precipitator and centrifuge washed with water to remove UNH and soluble fission elements held up in the precipitator heel and centrifuged cake.

The combined effluent and washings are received in the catch tank and constitute the waste metal solution. This waste contains about 90% of the fission activity present in the original metal solution and not more than 1% of the product.

The nitric acid used to dissolve the washed product cake is added to the precipitator where it is circulated through the sprays to wash down the inner surfaces and dissolve any bismuth phosphate remaining. It is then jetted, in portions, to the centrifuge where it is used to slurry out the cake, after which it is jetted to the solution tank. Dissolving is completed by agitating for 1 hour in the solution tank. A yield of 98-100% is normal, associated with 10% or less of the original fission activity. The product solution is jetted to crude product storage.

Precipitation Variables and Their Effect on Product Carrying

Process variables influence the completeness of product carrying by bismuth phosphate in both a chemical and physical manner. Variables which have a definite effect upon the yield of product obtained in the extraction step are: oxidation state of the plutonium, UNH concentration, iron concentration, nitric acid concentration, bismuth concentration, phosphoric acid concentration, strike temperature, rate of striking, and time of digestion.

In order that plutonium may be carried quantitatively on bismuth phosphate, it must be in the IV oxidation state. Other known states of oxidation are III, V, and VI. The III and VI phosphate are not carried, due in part to their high solubility (3 to 5 grams/liter), and the V state has not been found in process solutions (See Chapter I). The function of the pre-extraction nitrite treatment is to insure that all of the product is in the IV state;

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nitrite will reduce VI to IV and will oxidize III to IV. The time required for this reaction is approximately 20 minutes; 1 hour provides a margin of safety. Other agents which have been used in this step are: formic acid, which is satisfactory chemically, but introduces hazards in chemical handling; uranium ion (IV) which reduces Pu VI to IV but does not oxidize Pu III to IV, and was found to be unsatisfactory in the pilot plant; and a combination of potassium dichromate and oxalic acid, which also was found to be not entirely successful in pilot plant operation.

The concentration of the UNH solution from which the extraction is made has an important effect upon the extraction waste loss. High UNH concentration results in increased solubility of both plutonium phosphate and bismuth phosphate with resultant increased loss. Whenever the UNH concentration is increased, other variables such as temperature, acidity phosphoric acid concentration and rate of striking should be altered in such a way as to decrease the solubility of bismuth and plutonium phosphate, (Table I). The process employed at the startup of the T and B Canyons was based on 20% UNH in the final slurry. This concentration has been systematically increased (Production Test SE-221-T-PA-4) to 24% UNH, at the start of the strike, which is now the standard concentration. The use of concentrations up to 30% UNH have been found feasible on laboratory and semi-works scales, by increasing the phosphate concentration and decreasing the total acidity by either neutralizing part of the sulfuric acid or by replacing the sulfuric acid by sodium sulfate Decontamination tends to be greater at higher UNH concentrations, but the difference is not great.

TABLE I

Interdependence of Process Variables

Run No. T-5-04-	UNH %(a)	H_3PO_4 M (b)	Strike Temp., C°	Strike Time, Hrs.	Digestion Time, Hrs.	Extraction Waste, %
B-1	20	0.6	75	2	1	0.80
B-2	20	0.6	75	2	1	0.35
B-3	22	0.6	75	2	1	1.5
B-4	22	0.6	75	2	1	2.1
B-5	22	0.6	85	2	2	0.77
B-6	22	0.6	85	2	2	0.81
B-7	24	0.7	85	2	2	0.98
B-8	24	0.7	85	2	2	1.5
D-11	24	0.7	85	3	2	0.76
D-12	24	0.7	85	3	2	0.70

a) at start of strike

b) in final slurry

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Iron is not present in the UNH solution, except as a corrosion product from dissolver operation. At this concentration (approx. 0.001M or 0.005%) it has no effect upon the completeness of product carrying or upon the decontamination factor obtained. However, if the iron concentration is increased ten-fold it results in increased waste loss and at 0.05M Fe the loss may exceed 30%.

Nitric acid enters the extraction solution by three routes: (1) unreacted acid from the dissolver, (2) released by the sulfuric acid used in complexing the UNH, and (3) with the bismuth solution. Of these, the latter two are relatively constant but the first varies with dissolver performance and may materially affect the extraction step yield. Both the waste loss and the decontamination factor increase with increasing nitric acid concentration and at concentrations above 4% (free acid in 40% UNH) the waste losses become excessive. At 10% HNO_3 (free acid in 40% UNH) the waste loss may approximate 50%. This may be explained on the basis of increased solubility of bismuth phosphate with increased acid concentration.

Considerable laboratory data have been accumulated which show that the optimum bismuth concentration in this step is approximately 2.5 grams/liter. Concentrations higher than this result in slightly lower waste loss and a greater margin of safe operation, in respect to other variables such as acidity, UNH concentration, temperature, etc., but also increase the process volume in the subsequent decontamination cycle. Reducing the concentration below 2.5 grams/liter results in increased waste loss, due to less complete precipitation of bismuth and plutonium. At 1.5 grams bismuth/liter, the waste loss may exceed 10%.

At low UNH concentration (20%) the concentration of phosphoric acid is not critical, within the range 0.4M to 0.8M. As the UNH concentration is increased, however, the phosphoric acid concentration must be increased in order to maintain the insolubility of the bismuth phosphate, e.g., when the process concentration was increased from 20-24% UNH, the phosphoric acid concentration was increased from 0.6M to 0.7M. Within the normal operating range, phosphoric acid concentration has no effect upon the decontamination factor obtained.

Since bismuth phosphate has a retrograde solubility, it follows that higher strike and digestion temperatures result in more complete precipitation with more quantitative carrying of plutonium. The effect of temperature on product carrying is more pronounced at high UNH concentrations (Table I), but even at 20% UNH a decrease in temperature from 85 to 55 °C may increase the waste loss ten-fold. The strike and digestion temperature was increased from 75 to 85 °C when the UNH concentration was increased from 20 to 24%.

Agitation is required to mix and suspend thoroughly the bismuth, plutonium and phosphoric acid. Whereas the extraction yield is low at low power input, in the range 1-10 H.P./thousand gallons (the process was designed for 3.0 H.P./thousand gallons) yield is improved only slightly with increased agitation. In a case where the agitation failed, during a strike, air sparging plus extra time during the strike and digestion permitted a yield of 93% to be obtained; however, restoration of the agitator to service during the centrifugation may have contributed to additional carrying.

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Properties of Bismuth Phosphate as Related to the Extraction Step

Since plutonium is removed from solution by being carried by bismuth phosphate, the manner in which it is precipitated can affect the completeness of carrying. The most efficient carriers are those which precipitate with crystals isomorphous with the compound to be carried (See Chapter I). Bismuth phosphate, however, is not isomorphous with plutonium phosphate, yet the carrying obeys the laws of isomorphous carrying. This is explained by postulating strong internal absorption due to two-dimensional lattice similarities.

Three allotropic forms of bismuth phosphate are reported: (1) the "alpha", or normal form, is of the metastable, hexagonal modification, (2) the "beta" or insoluble form is of the monoclinic modification, and (3) a form whose X-ray pattern differs from that of either the alpha or beta form and has been designated the "gamma" form.

Plutonium can be carried by bismuth phosphate which is precipitated either in the solution or externally and then added to the solution. Using externally prepared carrier, however, the rate of plutonium removal is slow, but increases with increasing amount of $BiPO_4$ surface and is independent of the age of the precipitate. That such a process is largely a surface phenomenon has been shown by fractionally dissolving, in successive portions of nitric acid, externally prepared bismuth phosphate which carried product and observing rapidly decreasing radial plutonium concentrations as the center of the crystal was approached. Contrasted with this are data obtained with bismuth phosphate precipitated "in situ" by slow phosphoric strike, which showed a constant radial distribution of plutonium.

All bismuth phosphate precipitates employed in the separations process are formed "in situ." In the extraction step the alpha modification is the only form observed, UNH inhibiting the formation of the other forms. For maximum plutonium carrying, the following conditions are necessary:

Minimum bismuth phosphate solubility (see preceding section)

Slow precipitation

The rate of striking (precipitating) is of greater importance at high plutonium concentration (250 grams/ton) than at low concentration (5 grams/ton). The postulated explanation for this is that at low product concentrations the "adsorptive capacity" of the bismuth phosphate is not exceeded by the plutonium to be carried.

Whereas the rate of striking has an important influence on the carrying properties of bismuth phosphate, the type of strike has an important effect upon the physical (and handling) properties of the compound. The types of strikes may be divided into three groups: (1) direct (bismuth first, or co-formed), (2) reverse (phosphoric acid first, or preformed), and (3) combinations or modifications of the above. The direct strike produces a precipitate composed of large (approximately 5x10 microns with a surface area of 0.1 square meter/gram) imperfectly formed hexagonal crystals which are non-caking, filterable, easily centrifuged (specific gravity of 2) and readily soluble in

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nitric acid (See Figures 1 and 2 on the rate of solution and equilibrium solubility). The reverse strike, however, produces a precipitate which is fine, very "gummy" and therefore tending to cake, is more difficult to filter but not significantly more difficult to centrifuge, and less readily soluble in nitric acid. This type of strike was employed at the start-up of the pilot plant but was abandoned in favor of the direct strike because of the difficulty encountered in removing the cake from the centrifuge. Other modifications and combinations of strikes (simultaneous, direct-reverse) have been investigated in the laboratory and found to possess no advantage over the slow direct strike.

Rework of the Metal Waste Solution

No metal waste solution from either the B or T Plants has been reworked to recover product, although one charge (B-5-04-B-1) was reprocessed to recover neptunium. In this instance a large quantity of metal compound precipitated, in addition to the bismuth phosphate, which was difficulty soluble in nitric acid. The cause of this precipitation was subsequently shown to be due to the presence of ammonium ion, introduced as ammonium silicofluoride, resulting in the precipitation of ammonium uranyl phosphate ($NH_4UO_2PO_4$) together with some uranyl phosphate.

In the normal rework of a metal waste for the recovery of product no ammonium ion would or should be introduced. The waste, returned to the precipitator, should be adjusted to 0.5M sulfate with either sodium sulfate (preferable) or sulfuric acid and sodium carbonate and a slow (2-hr.) reverse strike made at 75-85 °C. No additional phosphoric acid need be added unless dilution has reduced its concentration below 0.6M.

Since the bismuth phosphate precipitated by this reverse strike is more difficult to remove from the centrifuge, special care must be taken to insure its complete removal.

After the cake is dissolved the product solution is processed as a normal run.

EXTRACTION EQUIPMENT

The extraction step is performed in a "standard section", Figures 3 and 4, which is composed of two cells, one containing a precipitator and a solution tank and the other a centrifuge and a catch tank (See Chapter II). The extraction is normally performed in Section 8 (Figure 5), Section 7 (Figure 6) being a spare, with Section 6 (Figure 7) capable of being used for this step with a few piping changes. The equipment in Section 8 is described below.

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FIGURE 1

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RATE OF SOLUTION OF $BiPO_4$ (EXTRACTION) IN 50% HNO_3

PILOT PLANT PRECIPITATE – EXCESS SOLIDS IN SUSPENSION

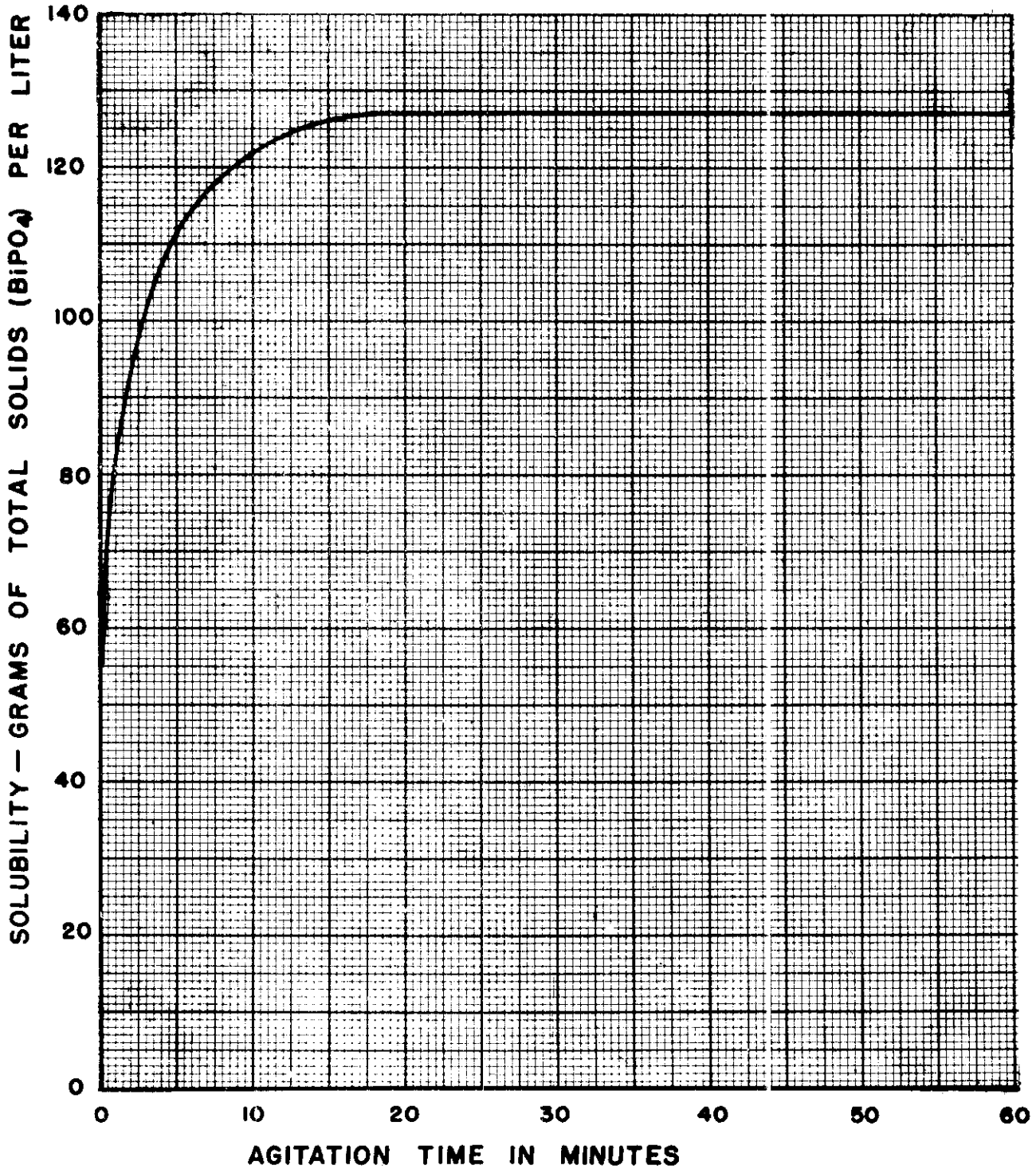

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FIGURE 2

SOLUBILITY OF $BiPO_4$ (EXTRACTION) IN HNO_3 AT 25° C

PILOT PLANT PRECIPITATE — EXCESS SOLIDS IN SUSPENSION

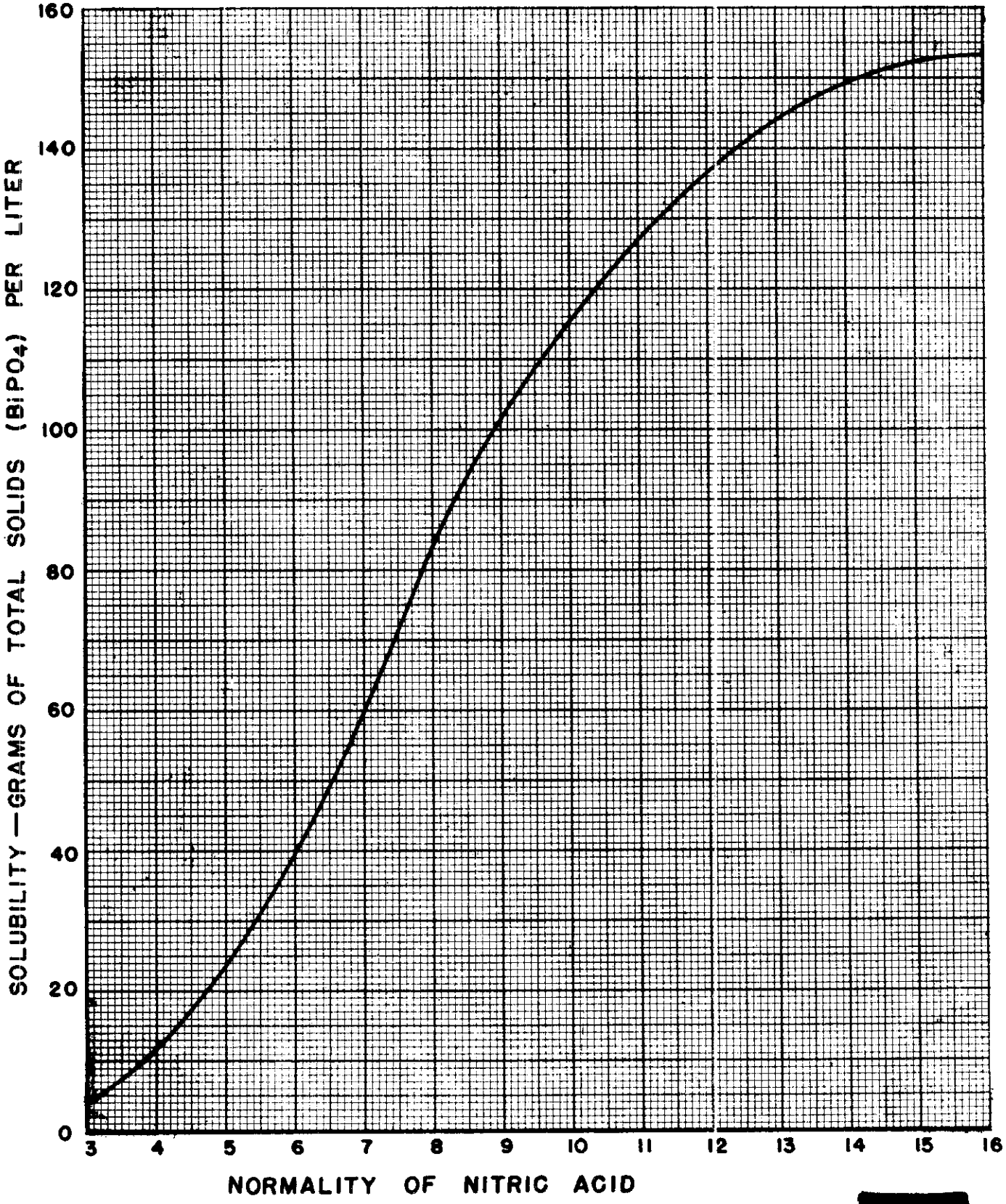

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FIGURE 3

CELL 13 - SECTION 7

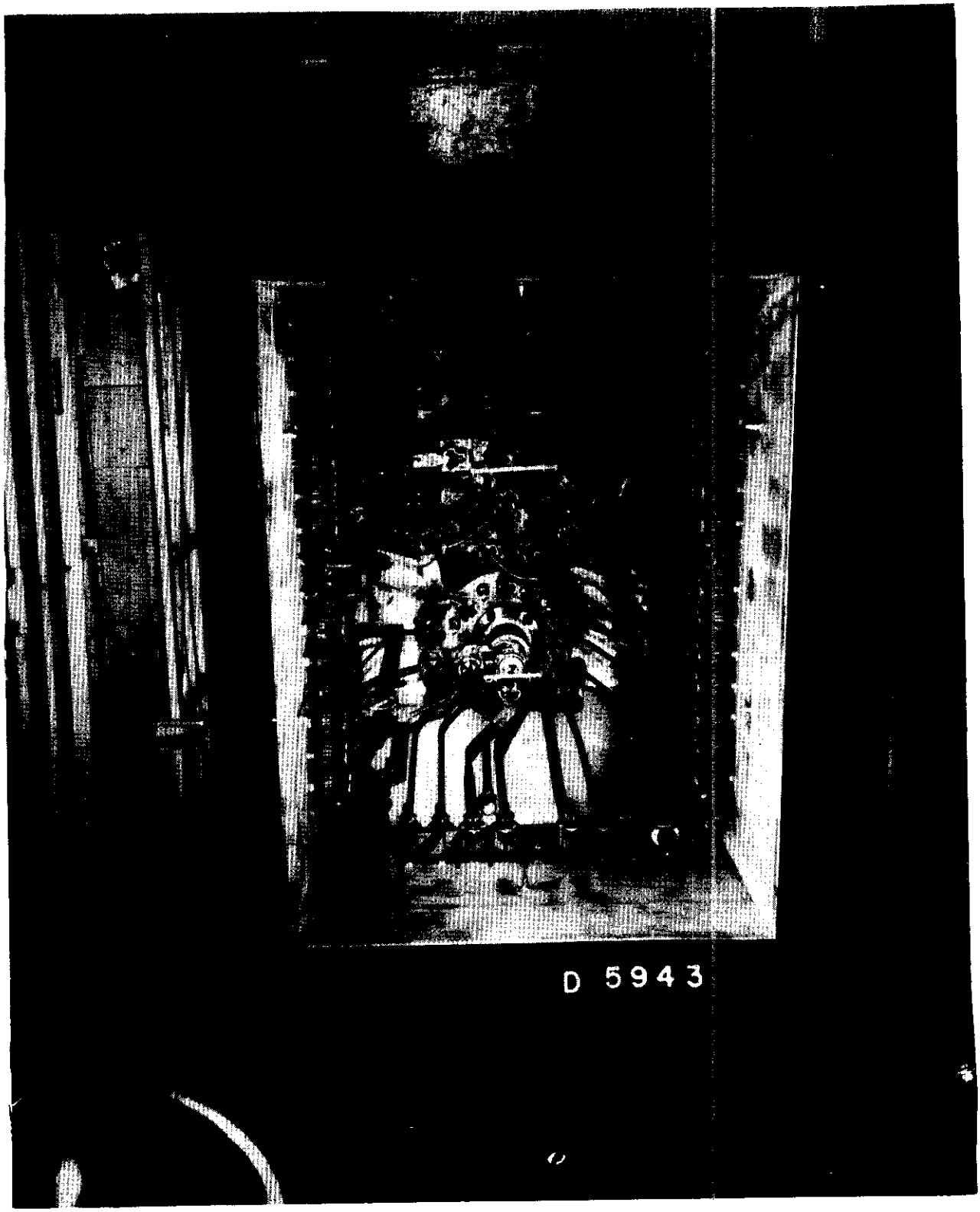

FIGURE 4

CELL 14 - SECTION 7

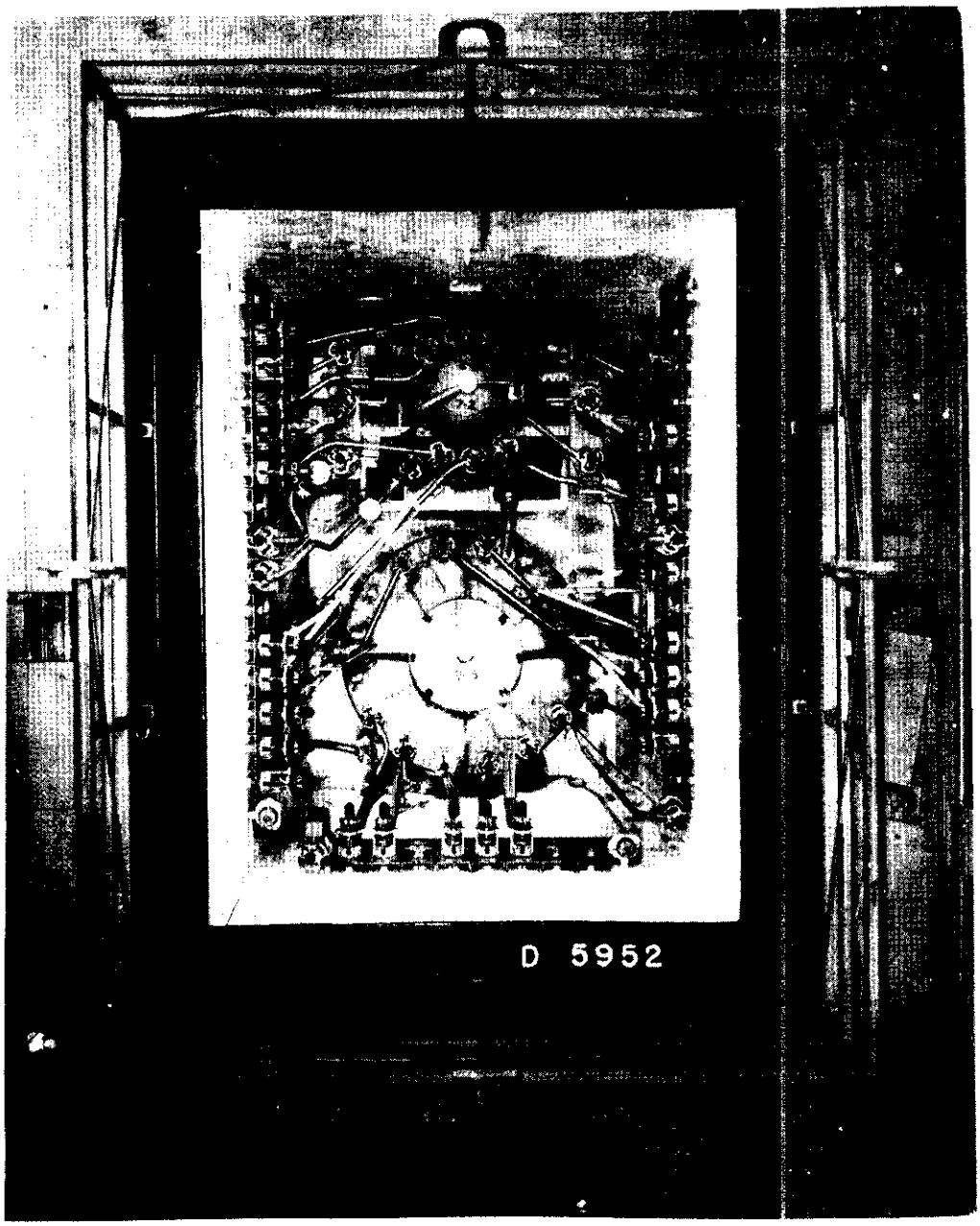

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FIGURE 5

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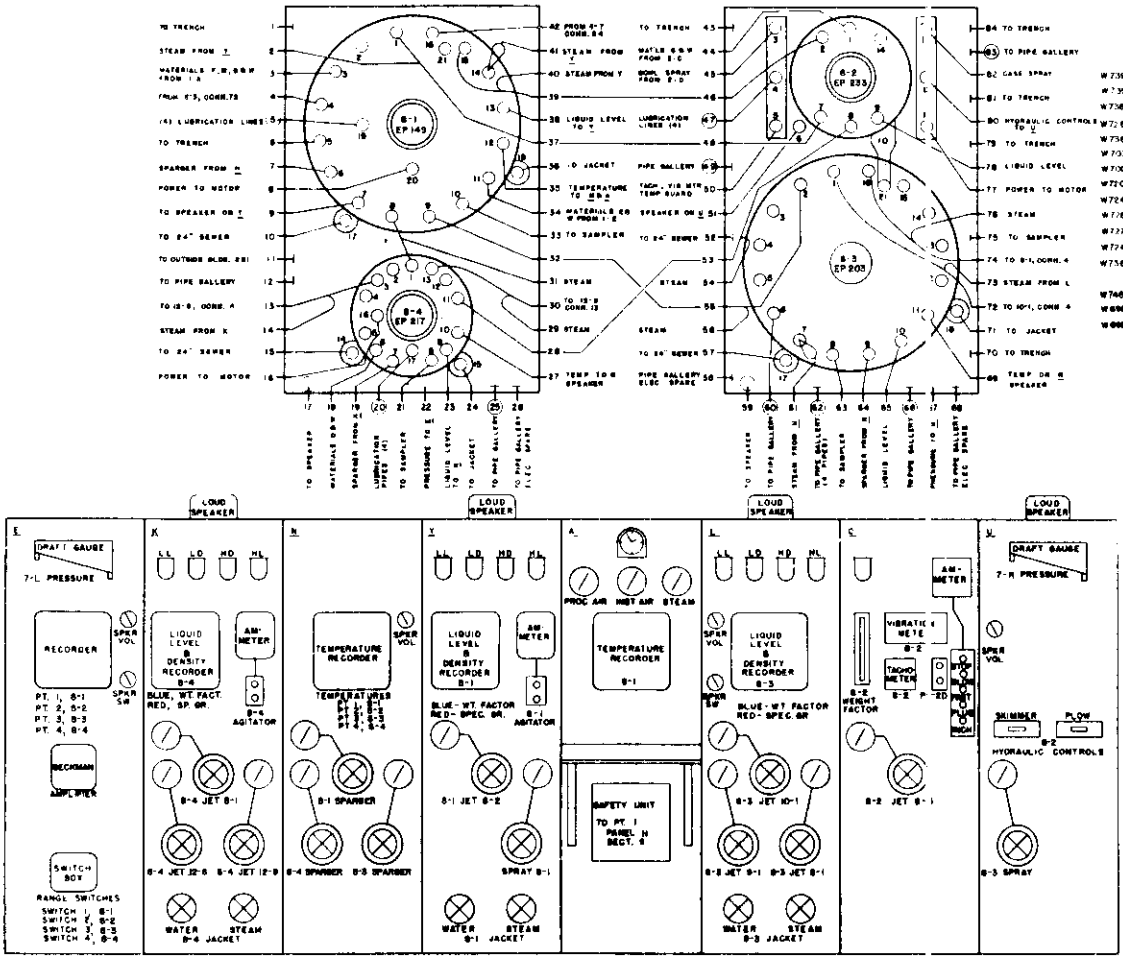
CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION B



LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WALL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM

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FIGURE 6

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 7

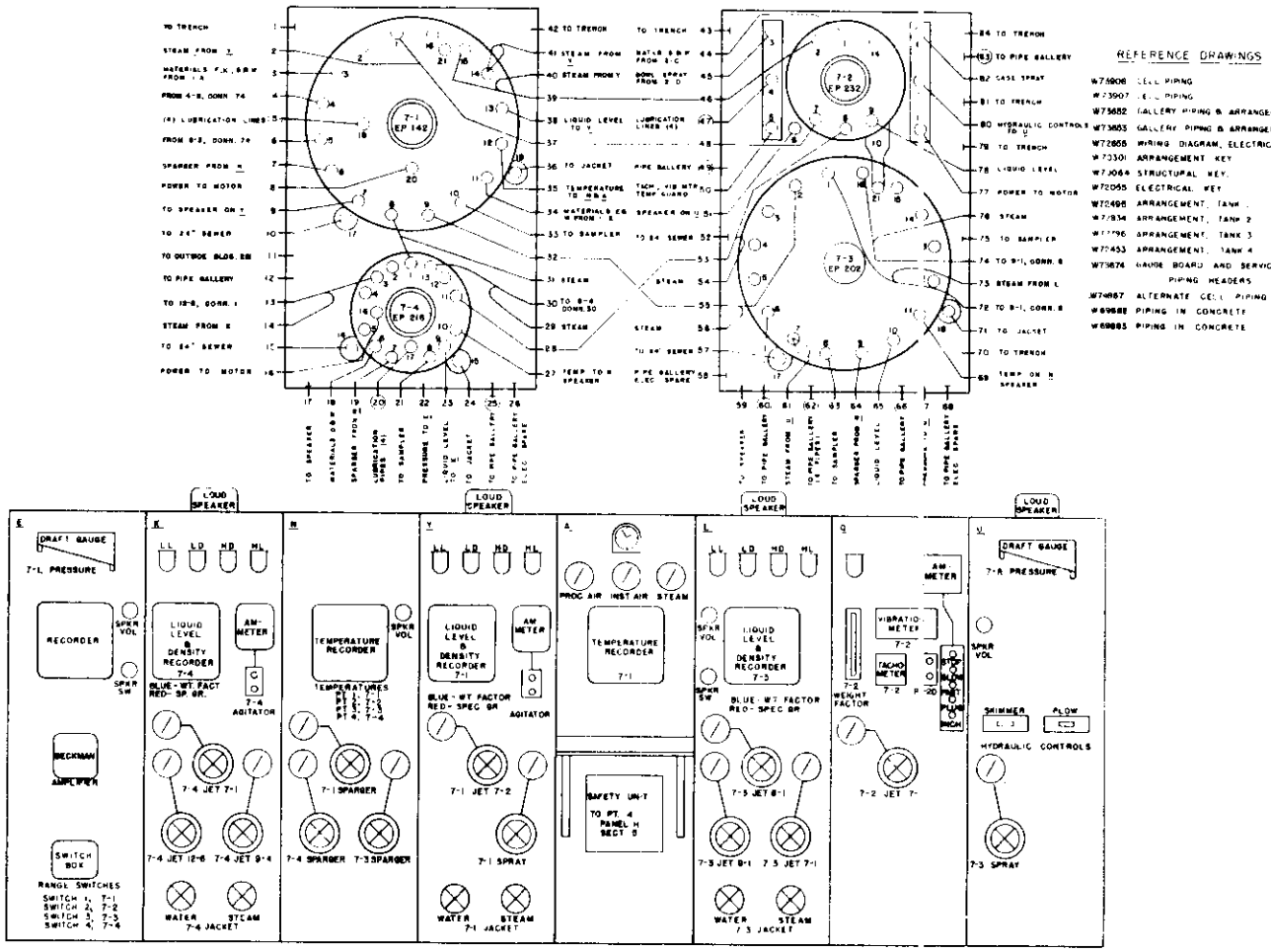

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FIGURE 7

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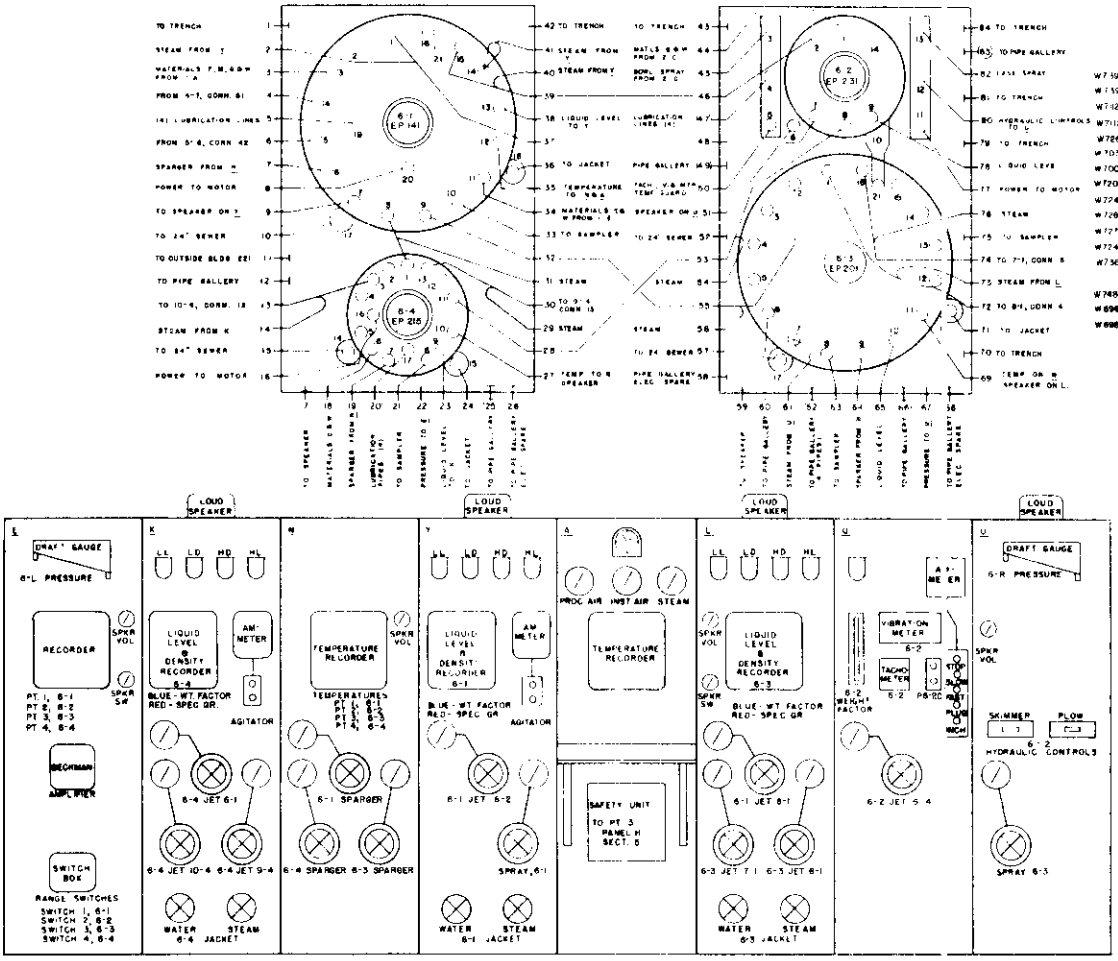
CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 6



LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WALL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM

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Precipitator

The precipitator, which is shown in Figure 8, is a 9 foot by 9 foot heat-treated 25-12 S-Cb tank. It has an overflow capacity of about 3900 gallons and about 205 gallons is required to reach the bottom of the agitator. (Drawing W-72493)

The tank is equipped with an agitator, a jacket, and an overflow line which drains to the cell floor. Suitable flanges are provided for connecting the motor, lubrication lines, and jacket inlet and outlet to the cell walls. Other connections are made through sixteen flanged openings around the top of the precipitator. These include a liquid level and density recorder, two thermometers, a sampler, a spray distributor, a sparger, two chemical feed lines, and process transfer lines. In addition there are some spares that are blanked off.

The spray distributor (Drawing W-73245) is used for washing down the vessel walls to prevent the build-up of bismuth phosphate cake. This is a jet operated device which pulls liquid up from the tank through a suction leg and forces it back through an outer concentric pipe terminating in a horizontal spray opening a few inches inside the vessel. This spraying causes a dilution of about 8 pounds of water/minute when the jet is operated at 100 lbs./sq.in. steam pressure. The nitric acid, used in dissolving the product cake from the centrifuge bowl, is usually added to the precipitator and circulated through the distributor to pick up any residual bismuth phosphate

The sparger (Drawing W-73235) is simply an inlet pipe with a slotted cap at the lower end.

The two thermometers are connected to different recorders; thus furnishing a check on each other. This standard arrangement is used on all precipitators because temperature control is more critical in the precipitation steps than in other operations. Also, since higher temperatures are used in the precipitators (and dissolvers) than in other process vessels, failure of the thermometers due to corrosion of the wells is more probable. While the two thermometers are usually brought in through the same connector, they are in separate wells so that a process step can be properly controlled and completed even if one thermometer fails.

Centrifuge

The centrifuge (Drawing BPF-71238), as shown in Figure 9, is a Bird 40-inch solid bowl machine operating at 870 or 1740 revolutions/minute (430 or 1730 G, i.e., to give an effective centrifugal force of 430 or 1730 times the force of gravity). The higher speed is usually not necessary for bismuth phosphate but may be used for other precipitates, such as lanthanum fluoride, which are difficult to separate cleanly.

The bowl hold-up is about 60 gallons. At the usual feeding rate of approximately 140 pounds/minute (or about 17 gallons/minute) the hold-up time is therefore about 3.5 minutes.

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FIGURE 8

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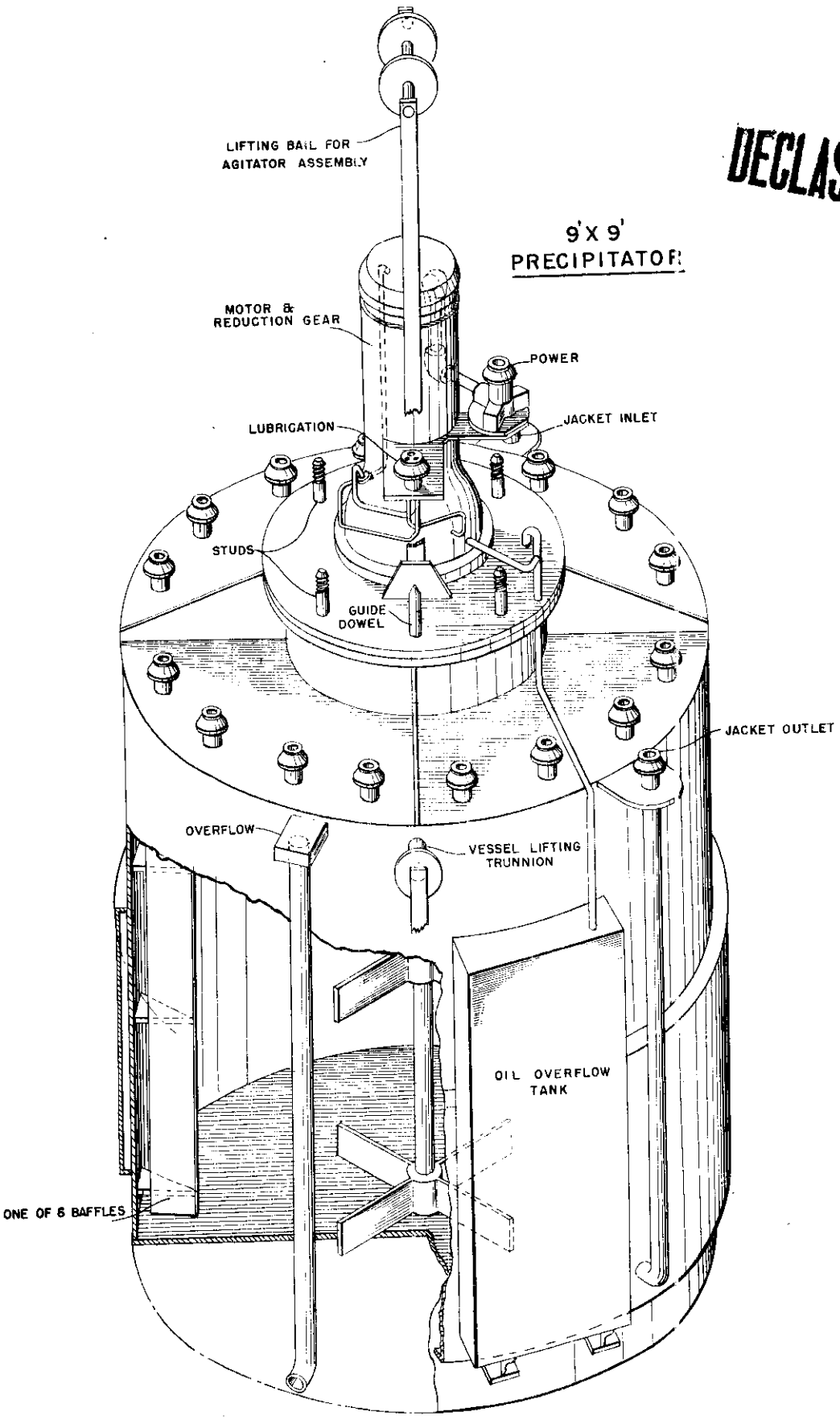

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FIGURE 9

CENTRIFUGE

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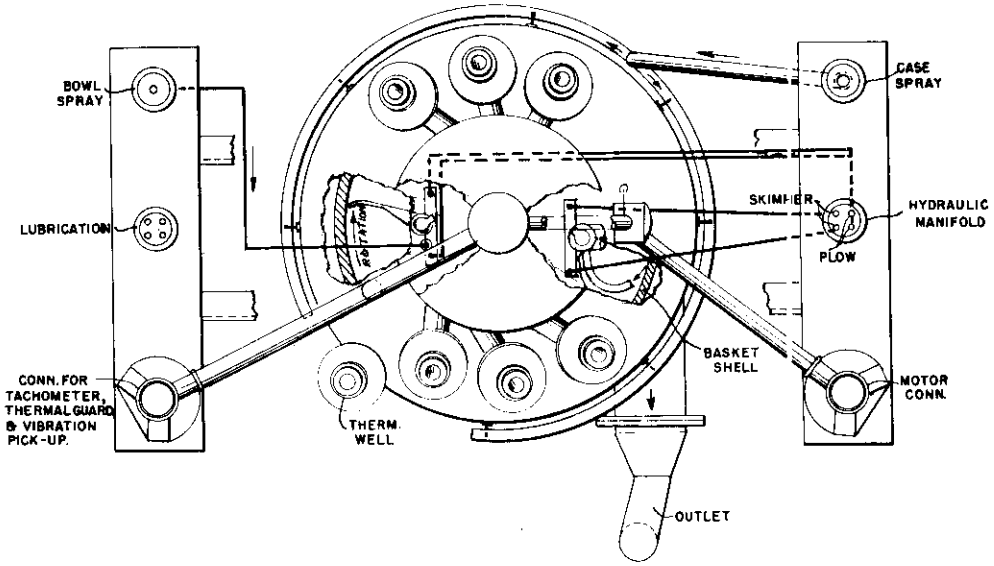

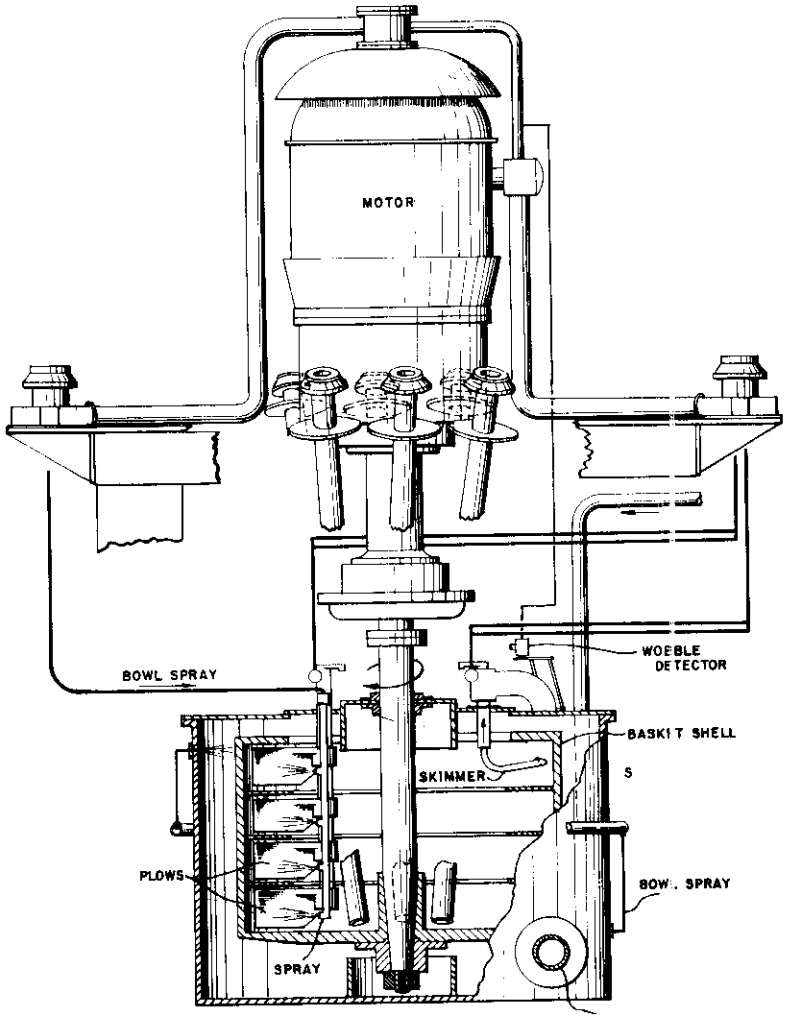

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The bowl effluent flows from an opening near the bottom of the casing of the centrifuge into the catch tank (Drawing W-72796). The centrifuges are not placed on the floor as are other process vessels, but on concrete blocks which give the elevation required for gravity flow to the catch tank. To obtain the accurate placement necessary for connections to the cell walls, the blocks are located with the usual type of equipment guides and contain studs to which the centrifuges are bolted with the impact wrench.

The hold-up can be reduced from 60 gallons to 10 gallons by use of the skimmer which is a curved pipe projecting down into the bowl. Through operation of hydraulic controls the pipe can be rotated to bring the lower end near the bowl's inner surface, facing counter to the direction of rotation. The liquid on the wall of the spinning bowl is then driven up the pipe and discharged into the casing.

To assist in cake removal, two sets of plows (scraper blades or unloaders), which could be moved into position close to the inner surface of the bowl, were designed into the centrifuge. These, like the skimmer, were hydraulically controlled, but are disconnected from the hydraulic system and blocked in the "out" position with wooden blocks inserted in the hydraulic cylinder.

For clean-up or cake washing, two sprays are available, one for the inside of the bowl, the other for the case. These are operated by a pump in the operating gallery.

The power, hydraulic and lubrication lines, and the leads to the vibration meter and tachometer are brought in through six connectors located on the "A" frame of the centrifuge.

Connections into the bowl are made through six sleeves from the cover. These sleeves are brought up at a slant in order to clear the motor assembly. Due to difficulties of manipulation presented by the slant, inlet pipes are not welded to the cell piping. Instead, they are inserted into the sleeves separately. The sleeves have the lower half of a connector flange at their upper end; the inserts have the upper half of the connector flange. When the inserts are dropped in place, the two halves function like an ordinary flange.

These connections are used for inlets from the precipitator, outlets for jetting cake solution and slurries to the solution tank, chemical feed lines, and manometer leads.

A simple manometer indicates the depth of liquid or "weight factor" in the centrifuge. No recording or density indicating devices are used.

A thermometer and microphone connection is made to the top of the case. The microphone is the contact type used for listening to operation of jets and motors. The thermometer well does not dip into any liquid, but is in contact with the outside of the case.

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Catch Tank

The catch tank is identical to the precipitator in design, but has no agitator. The agitator opening is blanked off with a manhole cover. A 4-inch hole is cut in the top of the vessel to accommodate the discharge line from the centrifuge case (Drawing W-72796). The similar hole in the precipitator is covered by a standard plug.

Through the openings around the top of the catch tank connections are made for liquid level and density recorder, thermometer, sampler, sparger, and chemical feed and transfer lines. The thermometer line also contains a contact microphone for listening to the outlet steam jets. This indicates when a jet is "gassing", i.e., has emptied the vessel.

Solution Tank

The solution tank is a small edition of the precipitator equipped with an agitator and accessory connections (Drawing W-72453). It has an overflow capacity of about 756 gallons and requiring about 36 gallons to reach the bottom of the agitator. The dimensions of the tank are 4 1/2 feet in diameter by 7 feet high.

Piping to the solution tank is similar to that of the precipitator: weight and density recorder, one thermometer and contact microphone, sampler, sparger, one chemical feed and process transfer line.

Process Piping

The process transfer connections in Sections 6, 7 and 8 are shown in Figures 41 and 51, Chapter II.

The piping between vessels in a section is arranged as in all standard sections. Two lines from the precipitator to the centrifuge are provided because plugging is more likely to occur in these jets which handle slurries. The catch tank is connected to the precipitator for recycling effluent from the centrifuge. Cake slurries and solution are jetted from the centrifuge bowl through a line to the solution tank. There are also lines from the catch tank to the solution tank, and from the solution tank to the precipitator, so that liquids can be jetted around through all vessels in the section in clean-up operations.

The trench piping establishes the operations for which a particular standard section can be used. Sections 7 and 8 are designed for extraction. Normally, only Section 8 is operated, Section 7 being held as a spare. It is considered possible that a pre-extraction decontamination step may be introduced into the process and either Section 6 or 7 is available for this purpose. All three sections can receive metal solution from storage in Section 4-R. For extraction, the catch tank is piped for disposal of the centrifuge effluent to Section 9 or 10 (waste metal) while the product goes from the solution tank to storage in Section 12-L. For a decontamination

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step preceding extraction, the catch tank is connected to an extraction precipitator and the highly active by-product cake solution is collected in the solution tank and disposed of along with the metal wastes.

Gallery Equipment

The arrangement and connections of Section 8, 7 and 6 are diagrammed in Figures 5, 6 and 7. The function of each cell connector is indicated by the number of the weigh tank or building section, or the letter of the panel board to which the line goes. Unlabeled connectors are spare lines to the pipe gallery.

Gallery Tanks

At the left end of the gauge board is a 400-gallon scale tank, 81-A, through which nitric and phosphoric acids can be added to the precipitator. (Drawing D-61773-A)

A 60-gallon scale tank, 82-C, is for adding nitric acid and wash acids to the centrifuge, and is located at the right of the gauge board. (Drawing D-61772-A)

Across from the gauge board near the back wall is 82-D (Drawing D-63505-A), a tank and pump which operate the centrifuge case or bowl sprays.

On the left of the pump is 81-E, for feeding sodium nitrite and bismuth nitrate to the precipitator. (Drawing D-61772-A)

Gauge Board

The functions of the several panels are apparent from the section diagram. In general, the process transfer gang valves, weight and density recorder, and motor switches for a particular vessel are located together on one panel. Two panels are required for the large number of centrifuge instruments and controls.

On panel "E" (Drawing W-71308), at the left, are the 8-L cell pressure draft gauge and the Beckman recorder for the section. By means of the four-point switch this can be made to function as a four-point recorder for the ionization chambers looking at all of the process vessels, or it can be kept continuously on any one chamber.

Two other panels serve the section generally. "N" (Drawing W-72477) has the four-point temperature recorder with leads to all four of the cell equipment pieces and valves for the three spargers (none on the centrifuge). "A" (Drawing W-71193) has a clock, telephone to the dispatcher's office, gauges for process air, instrument air and steam, and a safety meter to measure radiation levels in the gallery. The second precipitator thermohm is connected to a one-point temperature recorder on this panel.

The centrifuge boards, "Q" (Drawing W-72478) and "U" (Drawing W-72635),

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contain a tachometer, vibration meter (See Chapter X), gang valve for jetting to the solution tank, hydraulic controls for the skimmer and plow, and the centrifuge motor switch. Also located on these boards are the 8-R Draft Gauge, the catch tank distributor valve, and the switch for Pump 82-D which operates the centrifuge sprays.

Adjacent to all agitator and centrifuge switches is an ammeter to indicate the load on the motors. The agitator switches are simple two-button switches allowing only one speed. A five-button switch controls the centrifuge. The positions are stop, slow (870 rev./min.), fast (1740 rev./min.), plug, and inch. The centrifuge will coast for a long time after the current is turned off, but the plug bottom provides a breaking action by reversing the current through the motor. The inch button allows fairly constant low speeds, of the order of 100 rev./min., to be maintained during slurrying or washing operations. This supplies just enough current to overcome frictional losses. The centrifuge is brought up to the desired speed with the slow button and then switched over to inch.

The gang valves on all spargers and jets are so designed that, as the flow of steam is shut off, compressed air is automatically blown into the line. Further turning of the valve brings it to the completely off position. This prevents condensation of steam in the line which might draw active process solution up into the gallery pining. Beyond the off position is a setting which opens the cell line to a vent pipe at the bottom of the gauge board. This can be used to flush out the piping between process vessel and gallery. Pressure-vacuum gauges are provided to indicate any development of negative pressure in the gang valve lines, as well as to show the flow of steam to the jets.

It will be noted that the two jets transferring from the precipitator to the centrifuge are connected to the same gang valve. Near the pipe gallery wall, one of the lines is plugged with a blank and the other contains a filler, so that a quick change-over can be made if one jet fails.

All jacket lines are valved for air, steam and water for heating or cooling as required. The air is used to blow out the water before starting to heat with steam.

EXTRACTION OPERATIONS

Normal Operation

Product Precipitation

A 19,600-pound batch (1720 gallons) of 35% UNH solution is jetted into the Section 8 precipitator from storage in Section 4. The UNH Concentration is reduced to 27% by diluting with 6000 pounds (720 gallons) of water from the 81-A Scale Tank. Sodium nitrite solution (720 lbs. of 25% salt) is then added at a controlled rate from Scale Tank 8-1-E for the pre-extraction

treatment and the tank heated to 85 °C with steam on both the sparger and jacket. The slow addition rate is used to prevent too rapid NO_2 evolution and combined heating is used to save time. The dilution resulting from this sparging is approximately 1500 pounds (0.0055%/(1000 lbs.)(°C)). The solution is digested at 85 °C for 1 hour and then diluted to a total weight of 28,150 pounds (2840 gallons), at which point it is sampled for the "basis" figures. The dilution water plus the bismuth solution added later, bring the UNH concentration down to 24% at the start of the strike.

The precipitator agitator is operated throughout the extraction, until centrifuging is complete and the vessel is empty. Thorough agitation improves the yield and is necessary to prevent settling of the precipitate in the tank.

The precipitate is formed at 85 °C by adding 344 pounds of 24% $BiONO_3$/19% HNO_3 solution, following by 2625 pounds of 73.5% H_3PO_4/1.2% HNO_3 from Scale Tanks 8-1E and 8-1A respectively. The nitric acid in these reagents serves no purpose in the process itself, but is required in the bismuth subnitrate to keep it in solution and in the phosphoric acid to reduce storage tank corrosion. The corrosion products in unstabilized phosphoric acid can cause losses in by-product precipitates by reducing the product.

The bismuth is added as rapidly as possible. The phosphoric acid is added at a controlled rate over a period of 3 hours. It is important to the process that the phosphoric acid be added slowly, and after the bismuth (direct strike).

The solution is cooled to 50 °C before starting transfer to the centrifuge and cooling continued until the temperature is 25° or the tank empty. The total volume at this point is about 3100 gallons.

Centrifuging and Washing

The centrifuge is brought up to 870 rev./min. and the slurry is jetted into the bowl at a rate of about 140 pounds/minute using the "A" jet (See appendix). At this rate, the holdup time in the bowl is 3.5 minutes, which is adequate for clean separation of the bismuth phosphate. Completion of the transfer is indicated by levelling off of the catch tank weight factor trace. The centrifuge speed may increase slightly (20 rev./min.) and the centrifuge ammeter reading may drop slightly due to the decreased load on the machine. At this point three 300-pound portions of effluent are recycled from the catch tank through the precipitator and centrifuge to remove sediment from the precipitator. The 60-gallon bowl holdup is then reduced to about 10 gallons by gradually moving in the skimmer to a fixed stop.

The first water wash of 500 pounds (60 gallons) is added to the precipitator from Scale Tank 81-E. This wash is circulated through the distributor for 3 minutes to wash the walls, and jetted to the centrifuge through the "A" jet. When the transfer stops, the "A" jet is turned off and the "B" jet turned on in order to pick up any heel which may have been left by the "A" jet. The bowl is again skimmed to a 10-gallon heel.

The bowl is plugged down to about 10 rev./min. and a 250-pound wash (30 gallons) is added through the sprays from the tank and pump, 8-2 D.

This cuts most of the cake from the walls and helps to mix the wash and the hold-up liquid. The mixture is slurried three times by bringing the bowl up to 140 rev./min., holding 2 minutes, and plugging to a stop. A special automatic control is attached to the centrifuge tachometer to prevent running the centrifuge backward by plugging past the stopped position. (See Chapter X) This series of operations is intended to mix the cake and the wash thoroughly and avoid any tendency of the water to pass over the cake without flushing out soluble impurities. After stopping for the second time, the centrifuge is brought up to 870 rev./min., held 5 minutes, and skimmed to a 10-gallon heel.

The procedure is repeated with a second 30-gallon portion to conclude the washing operation.

The final 10 gallons of heel contains 88 pounds of bismuth phosphate and about 40 pounds of water.

The combined washings and effluent in the catch tank amount to about 32,000 pounds (3,200 gallons). The composition is 21.5% UNH, 3.6% H_2SO_4, 5.7% H_3PO_4 and 0.9% HNO_3, added with the reagents or carried over from metal dissolving. This metal waste is sent to Section 9 for neutralization and disposal.

Cake Solution

The cake is dissolved using 2710 pounds (240 gallons) of 60% nitric acid added to 8-1 from 8-1-A. In spite of the recycling and washing, some bismuth phosphate remains on the bottom and walls of the precipitator. The dissolving acid is therefore added to the precipitator and agitated to dissolve the solids lying on the bottom (5-10% of the charge) and circulated through the distributor for 20 minutes to dissolve solids adhering to the walls.

The acid is jetted from the precipitator to the centrifuge in portions of 330 pounds (30 gallons). Each portion is slurried three times by bringing the bowl up to 140 rev./min. for 2 minutes, then plugging to a stop. After thus mixing the cake and acid, the solution and slurry are jetted to the solution tank. After about half of the acid has been transferred in this manner, the remainder is continuously jetted from the precipitator and the centrifuge to the solution tank. The alternate jet is again used to make certain that all of the acid has been removed from the precipitator. The solution tank is agitated for 1 hour to complete the dissolving of the cake and then sampled.

The cake solution should contain 97-99% of the product and not more than 10% of the original fission activity. It is analyzed for completeness of solution and product content, then sent to storage in Section 12-L.

Alternate Operation

Section 7 is essentially an alternate for Section 8. The operation of Section 7 is the same as that of Section 8 except that the metal solution must come from Tank 4-8 instead of 4-7. Tank 8-4 has a transfer jet to 12-9 that is not found in Section 7, but both Tanks 7-4 and 8-4 have a transfer jet to Tank 12-6, the one normally used.

Section 6 is equipped for making a by-product precipitation step before the extraction, with transfer jets from 6-4 to the metal waste disposal cells and from 6-3 to further processing cells (Tanks 7-1 and 8-1). By slightly altering the trench piping on two jet transfer lines, the section may be altered to permit a product precipitation step. This may be done by changing the line going from Tank 6-4 to Tank 9-4, in the trench, so that it goes from Tank 6-4 to Tank 12-7, Connector 84, permitting the product solution to be sent to Section 12. In order to dispose of the metal waste the line from Tank 6-3 to Tank 8-1 can be changed to go from Tank 6-3 to Tank 9-3, Connector 72. An equivalent change could be made on the transfer line from Tank 6-3 to Tank 7-1 so that it would go from Tank 6-3 to Tank 9-3. Since Tank 9-3 has no agitator, the waste solution could then be jetted to Tank 9-1 for neutralization.

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REFERENCES

Report CN 2021	Recommendations for Process Conditions at HEW, and Review of Process Chemistry
Report CN 2050	Bismuth Phosphate Precipitation Studies
Report CN 1879	Progress Report on First Nine Months Operations of Separations Plant (Pilot Plant)
File No. 3-2609	SE-PC-[illegible] - Summary of Results of Investigations [illegible] Influence of Hydrazine in the Bismuth Phosphate Extraction Step
Report CN 2519	[illegible] Separation Process
File No. 3-2516	SE-PC-24 - Effect of Lead upon the $BiPO_4$ [illegible] Process
File No. 3-2535	SE-PC-32 - Solubilities of Plutonium Compounds in [illegible] Process Solutions
Report CN 2226	Investigation of the Use of Externally Prepared Bismuth Phosphate for the Extraction of Plutonium from Uranyl Nitrate Solutions
Report CN 2265	The Relation of Bismuth to Plutonium and Rare Earth Phosphates
Report CN 2224	The Coprecipitation of Plutonium (IV) with Bismuth Phosphate
File No. 3-2028	Production Test SE-221-T-PA-1 - Increasing the Extraction Charge Size by Increasing both the Extraction Volume and the Extraction Concentration
Report CN 2557	Solubility of $BiPO_4$ and Uranyl Phosphate in UNH Solutions
Report CN 2561	Physical Properties of the X-Plant Product, By-Product, and Scavenger Precipitates
Report CN 2223	The Aging of Bismuth Phosphate Precipitate
File No. 3-2537	SE-PC-36 - Cause and Prevention of Uranium Precipitates in the Extraction of Neptunium
File No. 3-2707	SE-PC-39 - Investigation of Partial Neutralization of the Uranyl Nitrate Solution Prior to Extraction

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APPENDIX

SAMPLING

Representative sampling of process solutions in the Canyon Building is of prime importance in the control of the process. In order to obtain a representative sample, the solution to be sampled must be agitated sufficiently to insure uniformity and the sampling device must not permit concentration, dilution, or segregation in any part of the system. The sampler used in the Canyon Buildings is an air lift operated in conjunction with an air jet and is of stainless steel construction (Drawings W-73746 and D-63381). Its purpose is to make accessible at the deck level a representative portion (about 4 ml.) of the active solution contained in a given tank in the heavily shielded cells.

The air lift principle is used in addition to simple jet circulation because solutions of high specific gravity must be raised 20-25 ft.; in the Concentration Building the low head makes the air lift feature of the sampler optional. The samplers in the Canyon Building are located in the cell walls in lead shielded pits approximately 2 feet below deck level (See Fig. 10.) A 1/2 inch stainless pipe with a special splash plate on the bottom and a lead shielded cap, runs from the sampler cup (See Fig. 11.) to the deck and provides a passage for withdrawing samples. The air control valve for the circulating jet is located under a 1/4 inch steel cover plate in the deck.

In taking the more active samples - during the extraction step and first decontamination cycle (8-1 MR, 8-4P, 8-3WS, 13-4BP) - the jet is operated 10 minutes and fresh representative solution is collected in the cup. During this time a Koroseal stopper mounted in the lead cap seals the end of the 1/2 inch riser pipe. The cap is then removed and the "Trombone" apparatus (Drawing D-63135, See Fig. 12) inserted in the riser pipe. With this a 0.5 - 1.0 ml. portion of solution is withdrawn from the cup and placed in a portable shield or "Door Stop." A 1 ml. sample of 8-1-MR Solution (20-25% UNH) held in the "Trombone" may give off radiations at the rate of about 2000 mr./hour 2 inches from the sample. When placed in the "Door Stop" the intensity of radiation is reduced to 20 - 60 mr/hour at the outside surface of the shield.

Sampling of the less active solutions (Section 14 and cooler cells) is similar to the above procedure, except that a "Bayonet" pipette (Drawing D-65232) with a rubber stopper seal built on it, is used to withdraw a 3.0 - 5.0 ml. sample (See Fig. 13). After the regular 10-minute circulation time with the lead shielded stopper in place, the cap is removed and the pipette inserted to extend to the liquid level in the cup (about 15 inches). The sample air jet is again started and the cup and pipette evacuated; when the air is turned off, the vacuum in the sampler cup is broken and the pipette fills. This procedure can be repeated to flush out the pipette; the pipette is then transferred to a portable shield and taken to the laboratory.

The special splash plate (mentioned above in connection with the 1/2 inch riser pipe - See Figures 10 and 11) baffles downward the fresh solution entering the cup and insures complete flushing of old samples from the cup. A small hole at the top of the baffle vents the suction line and prevents suckback of the sample when the jet is turned off. In order to obtain representative samples, a perfect seal of the 1/2 inch riser pipe during the circulation

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FIGURE 10

SAMPLER

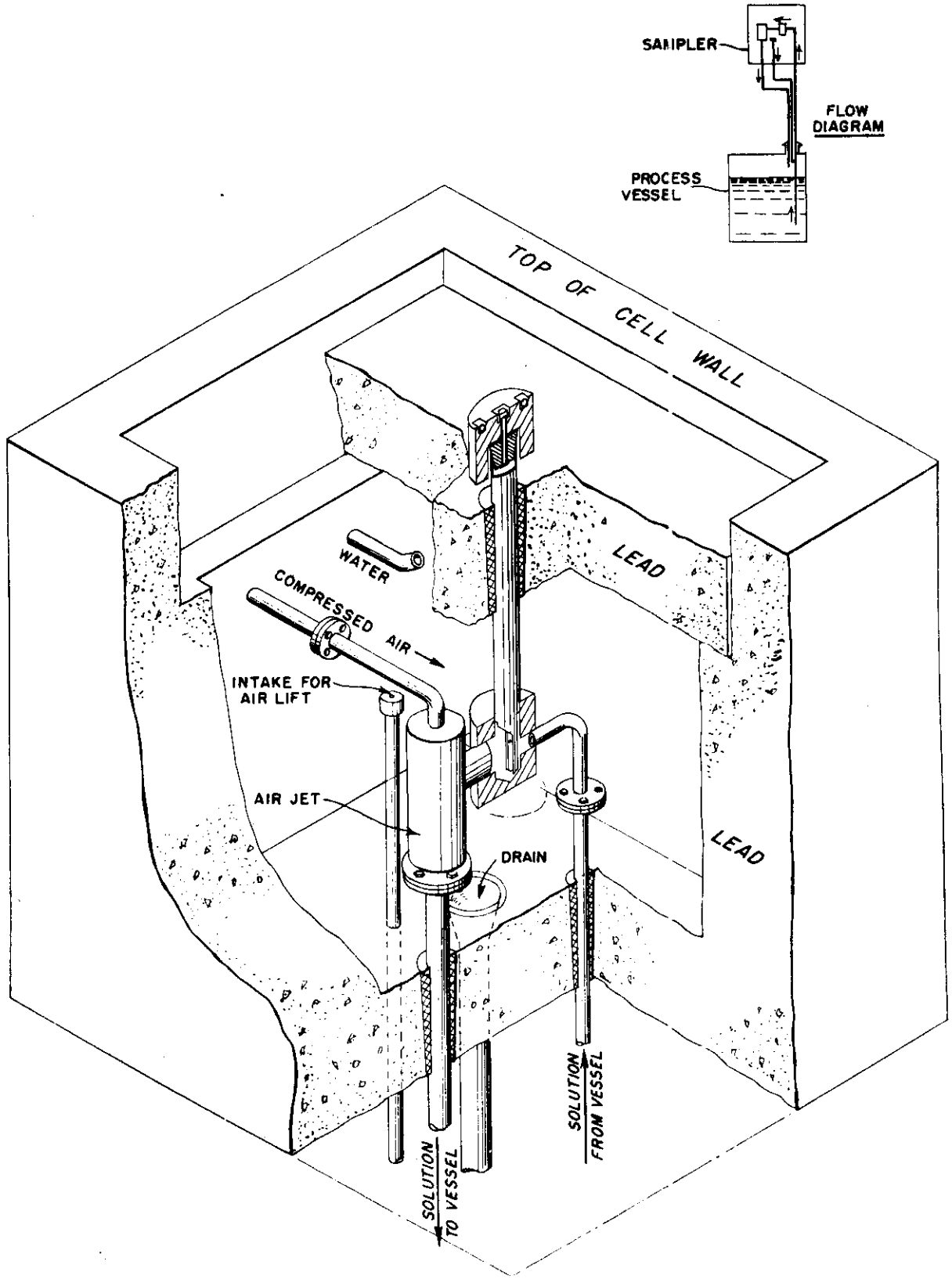

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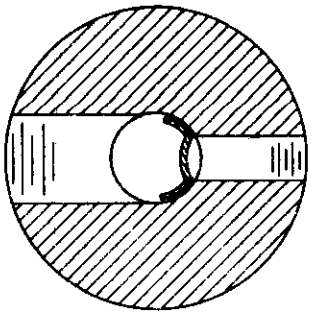
FIGURE 11



SECTION A-A

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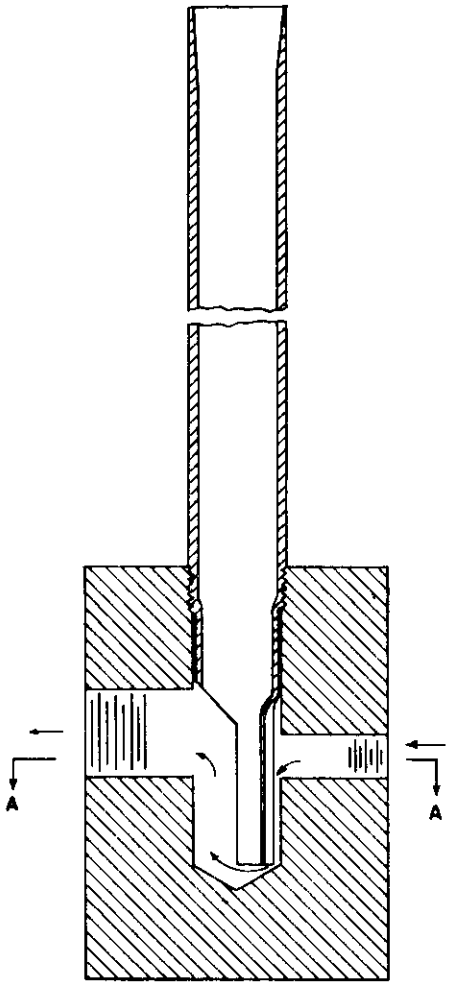

VERTICAL CROSS SECTION

DIAGRAMMATIC REPRESENTATION
OF BAFFLED SAMPLER CUP.

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FIGURE 12

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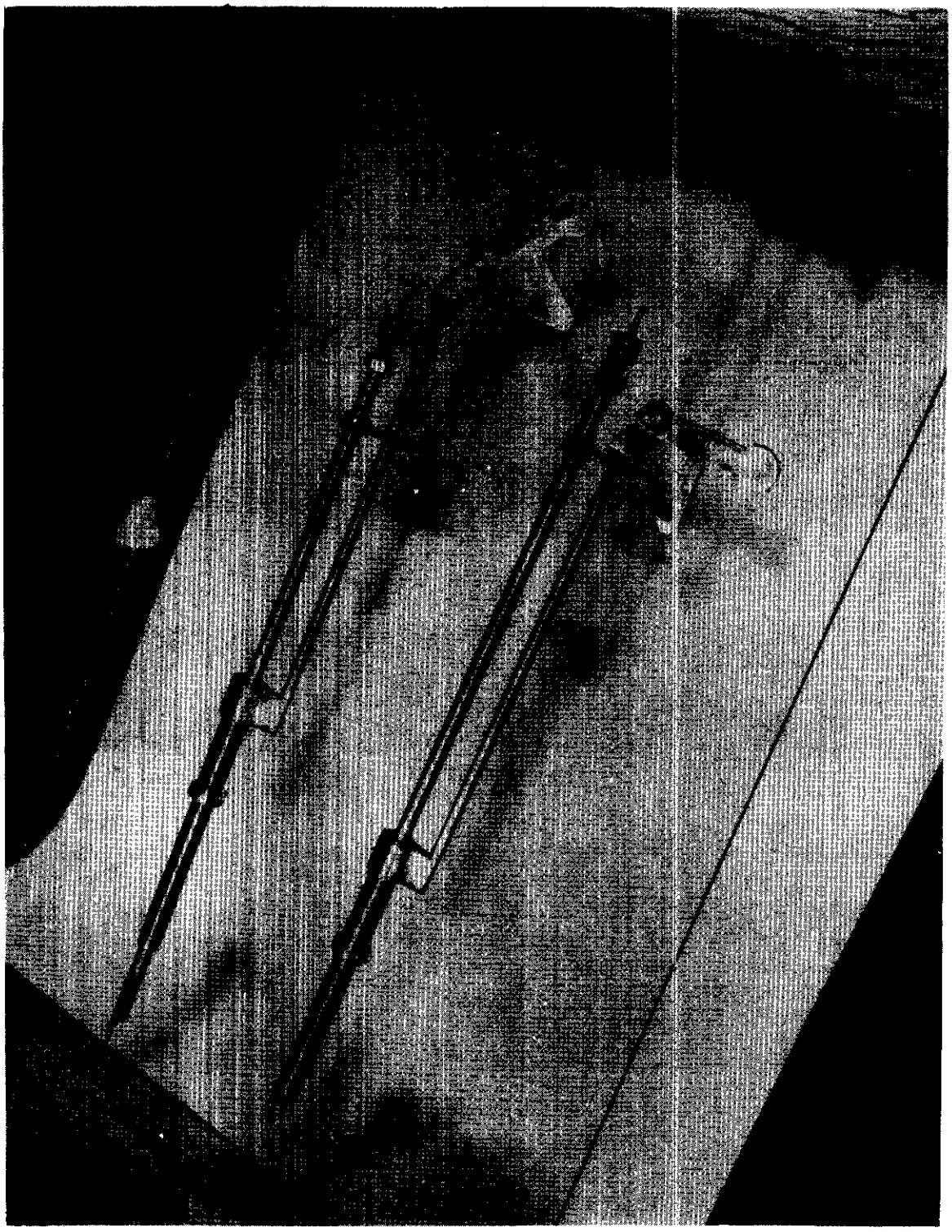
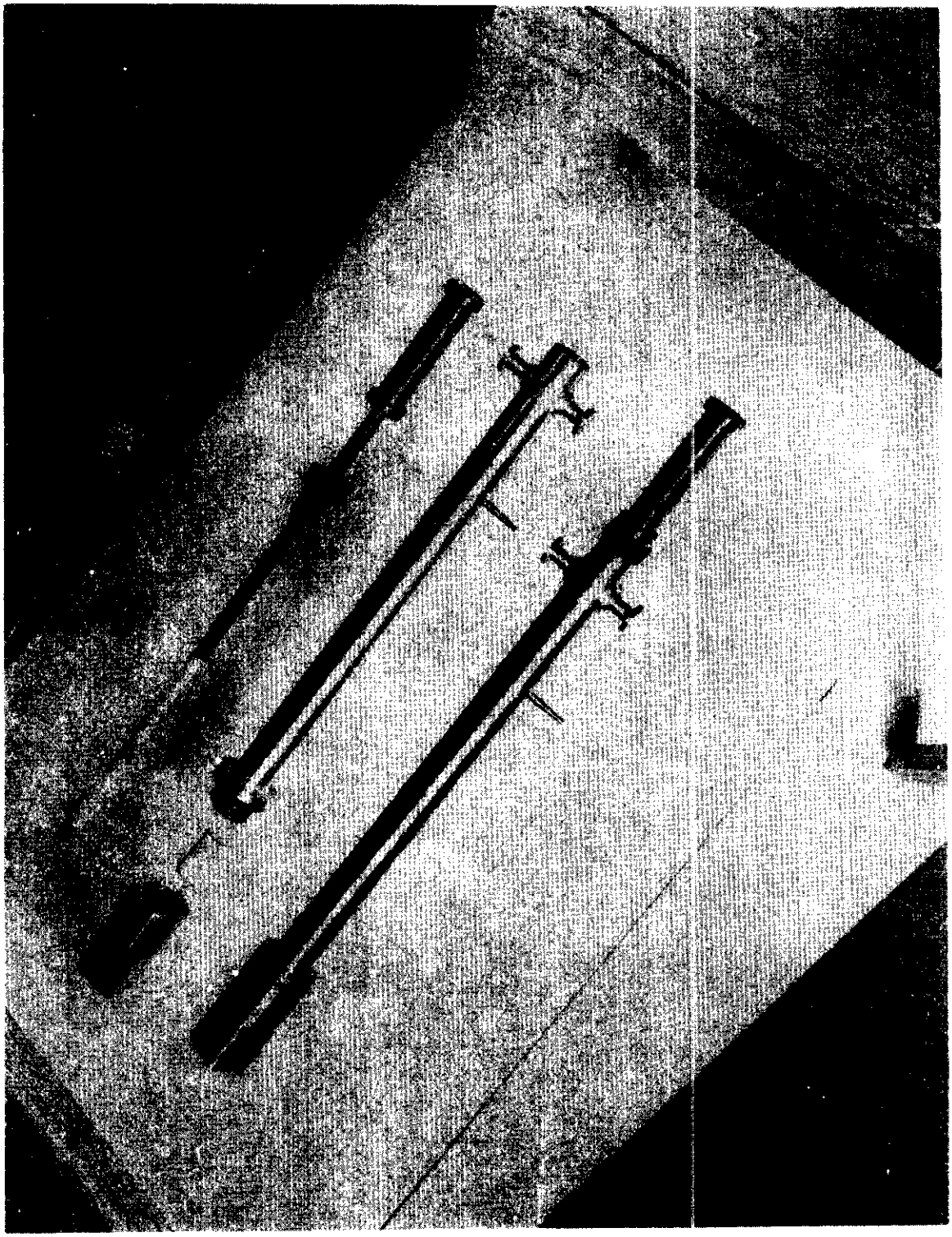


TROMBONE SAMPLER

FIGURE 13



BAYONET SAMPLER

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period is essential. The spray and mist from the circulating solution, especially the strongly acid product and by-product cake solutions, attacks the material used to seal the riser pipe. Rubber and Neoprene stoppers have not proved satisfactory in this service and have had to be replaced frequently; Koroseal stoppers have shown better acid resistance. No gasketing and a simple Koroseal gasket between the lead cap and the riser pipe were tried and found to give an unsatisfactory seal.

The optimum orfice size on the inlet line to the air lift is about 1/32 inch. With this orifice size, the vacuum in the cup created by the jet (operating at 100 lb./sq.in. air pressure) varies between 5 in. and 20 in. Hg, as slugs of air and liquid move along.

The safety precautions which must be exercised in the sampling of product solutions are very rigid. The details of these rules may be obtained by referring to the S Department Sampling Procedures.

FLOW SHEET

The flow sheet presented in Table II is for 1.5-ton charges at 24% UNH. The flow sheet may change from time to time due to limitations on the amount of permissible product per charge, but due to such limitations, the charge size may be less than 1.5 tons with resultant decrease in UNH concentrations and increase in operating latitudes.

STANDARD LOG FORMS

The operating log forms given at the end of this appendix constitute the detailed operating instructions by which the operators perform the extraction step.

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TABLE II

PRODUCT EXTRACTION FLOW SHEET

Section 8 (Section 7, spare)

A. Precipitation

1. Receive 19,600 lbs. (1720 gal.) of UNH solution from 4-7 into Precipitator 8-1.
2. Add 7,830 lbs. (940 gal.) of dilution water.
3. Add 720 lbs. (75 gal.) of 25% $NaNO_2$ solution.
4. Agitate for 1 hour at 85 °C.
5. Add 344 lbs. (27 gal.) of Bi solution A:

   24% $BiONO_3$

   19% HNO_3
6. Add 2625 lbs. (200 gal.) of H_3PO_4 solution:

   73.5% H_3PO_4

   1.2% HNO_3

   Rate of addition - 15 lbs./min.
7. Agitate for 2 hours at 85 °C.
8. Cool to 50 °C before jetting to Centrifuge 8-2.

	Slurry lbs.	Composition %	(In 8-1) M
UNH	6950	22.3	0.53
HNO_3	290	0.9	0.18
H_2SO_4	1150	3.7	0.45
H_3PO_4	1823	5.9	0.72
$BiPO_4$	88	0.3	----
$NaNO_2$	nil	---	----
$NaNO_3$	222	0.7	----
H_2O	20,696	66.2	----
Total	31,219 (3100 gal.)		

[redacted]

TABLE II
(Cont.)

DECLASSIFIED

B. Centrifugation and Washing

1. Centrifuge at 870 rev./min., 140 lbs/min.
2. Recycle 1200 lbs. of effluent through 8-1 and 8-2.
3. Skim to 10 gal. heel.
4. Add 500 lbs. (60 gal.) of water to 8-1.
5. Recirculate in 8-1 for 3 minutes using spray.
6. Jet wash water from 8-1 to 8-2.
7. Skim to 10 gal. heel.
8. Add 250 lbs. (30 gal.) of water to 8-2 through the sprays.
9. Wash cake and skim to 10-gal. heel.
10. Repeat (8) and (9).
11. Jet waste effluent and washings from Catch Tank 8-3 to Precipitator 9-1 (or 10-1).

Effluent Composition (in 8-3)

	lbs.	%	
UNH	6950	21.5	
HNO_3	290	0.9	
H_2SO_4	1150	3.6	
H_3PO_4	1823	5.7	
$NaNO_3$	222	0.7	
H_2O	21,965	67.6	
Total	32,400		(3200 gal.)

Washed Cake Composition (in 8-2)

	lbs.	%	
$BiPO_4$	88	69.3	
H_2O	39	30.7	
Total	127		(10 gal.)

[redacted]

[redacted]

TABLE II
(Cont.)

DECLASSIFIED

C. Product Cake Dissolving

1. Add 2710 lbs. (238 gal.) of 60% HNO_3 to 8-1.
2. Recirculate through the spray for 20 minutes with the agitator running. Keep acid below 50 °C.
3. Jet 330 lbs. of acid (30 gal.) from 8-1 to 8-2 and slurry cake.
4. Jet from 8-2 and Solution Tank 8-4 and start agitator.
5. Repeat (3) and (4) three more times.
6. Jet remaining acid from 8-1 to 8-2 to 8-4.
7. Agitate for at least 1 hour after all acid is in 8-4.
8. Jet solution from 8-4 to Storage Tank 12-6.

Solution Composition (in 8-4)

	lbs.	%	M
$BiPO_4$	88	2.6	0.11
HNO_3	1640	49.0	10.2
H_2O	1622	48.4	---
Total	3350 (305 gal.)		

[redacted]

[redacted]

PRODUCT EXTRACTION

Date _____
Run No. _____

DECLASSIFIED

I PRODUCT EXTRACTION

1. Call dispatcher and request transfer of metal solution from 4-7.
   Time called _____

2. Air sparge 4-7 for 30 minutes before jetting a charge to 8-1. Leave air sparger on.
   4-7 Wt. Ftr. _____ Lbs. _____
   4 7 Air sparger on _____
   Time start _____ Time end _____

3. Beckman reading on 8-1.
   Meter _____ Factor _____

4. When cleared by the dispatcher, receive 19,600 lbs. of metal solution from 4-7 into 8-1. Base charge on 8-1 Wt.Ftr. reading. (TW from 4 7 18,800 lbs. Care must be used during this transfer be cause of the high rate of the jet)
   Time cleared _____
   Time start _____ Time end _____
   8 1 Wt.Ftr. _____ Lbs. _____
   8 1 Sp.Gr. _____ Temp. _____

5. Shut off 4-7 air sparger and read Wt. Ftr., Temp. and Sp.Gr.
   4-7 Air sparger off _____
   4 7 Wt. Ftr. _____ Lbs. _____
   4 7 Sp.Gr. _____ Temp. _____

6. Start 8-1 agitator
   8 1 Agitator on _____

7. Add 6000 lbs. of water to 8 1 via 8 1A. Add water in two portions of 3000 lbs. each
   1st Port _____ Lbs. Time added _____
   2nd Port _____ Lbs. Time added _____
   Total lbs. added _____

8. Add 720 lbs. of 25% Sodium Nitrite (Line B) to 8 1 via 8 1E at a rate of 20 lbs. per minute.
   Time start _____ Time end _____
   Lbs. added _____

9. Flush 8-1E to 8 1 with 50 lbs. of process water.
   Lbs. added _____ Time added _____

10. Heat 8-1 to 85 C. with steam on the jacket and sparger. Maintain temperature at 85 C. with jacket until step 21 is completed.
    Time start _____ Time 85C _____

[redacted]

[redacted]

Date_____
Run No_____

DECLASSIFIED

I PRODUCT EXTRACTION (Con't)

11. Digest 8-1 at 85 C. for 1 hour	Time start____ Time end____
12. While digesting, shut off 8-1 agitator, read Wt.Ftr., Temp. and Sp. Gr. when Sp.Gr. meter becomes constant, then start 8-1 agitator.	8 1 Agitator off____ 8 1 Wt. Ftr.____Lbs. ____ Sp. Gr.____Temp. ____ 8 1 Agitator on____
13. Call dispatcher, give charge data and request that sample 8-1 MR be taken	Time called____ Time sampled____
14. When notified by the dispatcher that sampling is complete, add water from 8-1A to make total weight in 8 1 up to 28,150 lbs.	Lbs. added____ Time added____
15. Add 344 lbs. of 24% Bismuth solution (Line R) to 8-1 via 8 1F.	Lbs. added____ Time added____
16. Flush 8-1F to 8 1 with 50 lbs. of process water.	Lbs. added____ Time added____
17. Digest 8-1 for 15 minutes	Time start____ Time end____
18. Add 2625 lbs. of 75% Phosphoric Acid (Line F) to 8 1 via 8-1A at a rate of 15 lbs. per minute.	Time start____ Time end____ Lbs. added____
19. Flush 8 1A to 8-1 with 50 lbs. of process water.	Lbs. added____ Time added____
20. Beckman reading on 8-1.	Meter____Factor____
21. Digest for 2 hours at 85 C.	Time start____ Time end____
22. Turn on 8-1 cooling water	8-1 Jacket water on____
23. Stop 8-1 agitator and read Wt.Ftr., Temp. and Sp.Gr. when Sp.Gr. meter Becomes constant, then start 8-1 agitator again.	8-1 Agitator off____ 8-1 Wt. Ftr.____Lbs. ____ 8-1 Sp.Gr.____Temp. ____ 8-1 Agitator on____
24. Cool to 50.C. and leave cooling water on until 8-1 is empty or at 25°C	Time 50 C.____ Cooling water off____

[redacted]

[redacted]

PRODUCT EXTRACTION

Date _____
Run No. _____

DECLASSIFIED

II CENTRIFUGATION

1. Beckman reading on 8-2.

   Meter_____ Factor_____

2. Supervisor's approval to start centrifugation

   Beckman limit _____
   Array previous run (8-4) _____
   Approved by: _____ Time _____
   Supervisor

3. Set the centrifuge skimmer and bring the centrifuge to 870 RPM.

   Time 870 RPM _____

4. With 8-2 at 870 RPM and using the "A" jet, jet slurry from 8-1 to 8-2 at the rate of 140 lbs. per minute. Check the rate at intervals to insure proper rate. Shut off 8-1 agitator when the Wt.Ftr. reaches 0.2.

   Time start _____ Time end _____
   Transfer rate _____ Time _____
   _____ _____
   _____ _____
   Avg. _____
   8-1 Agitator off _____

5. Turn on 8-3 air sparger and jacket cooling water

   8-3 Air sparger on _____
   8-3 Jacket water on _____

6. When transfer from 8-1 to 8-2 stops, indicated by leveling off of the 8-3 Wt. Factor trace (do not shut off jet), jet from 8-3 to 8-1 for 30 seconds and empty 8-1 again. Repeat this operation 2 times to flush out 8-1. Shut off jet on completion of the recycling.

   Time start _____ Time end _____

7. After 5 minutes skim 8-2 to a 10 gallon heel.

   Time end _____

8. Add 500 lbs. of water to 8-1 via 8-1E.

   Lbs. added _____ Time added _____

9. With cooling water on the jacket, circulate through 8-1 spray for 3 minutes. Hold 8-1 temperature below 50C.

   Time start _____ Time end _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

II CENTRIFUGATION (Cont'd)

10. Jet 8-1 to 8 2, using the "A" jet When transfer stops, indicated by leveling off of the 8-3 Wt. Factor trace, shut off the "A" jet. Using the "B" jet, again jet 8-1 to 8-2. Time end _____

11. After 5 minutes skim 8-2 to a 10 gallon heel. Time end _____

12. Plug 8-2 to stop. Time stop _____

13. Add cake dissolving acid to 8-1 and start circulation (See page 3.6, steps 1-5 incl.) before starting cake washing. Time added _____

[redacted]

[redacted]

PRODUCT EXTRACTION

Date _____
Run No. _____

DECLASSIFIED

III- CAKE WASHING

1.	Bring 8-2 to 10 RPM, add 250 lbs. of process water through the 8-2D bowl sprays at full pump pressure.	Time 10 RPM _____ Lbs. added _____ Time added _____
2.	Bring 8-2 to 140 RPM, hold 2 minutes then plug to stop. Repeat twice.	Time end _____
3.	Read 8-2 Manometer.	Manometer _____ Lbs. _____
4.	Bring 8-2 to 870 RPM, hold 5 minutes and skim to 10 gallon heel.	Time end _____
5.	Plug 8-2 to stop.	Time stop _____
6.	Bring 8-2 to 10 RPM, add 250 lbs. of process water through the 8-2D bowl sprays at full pump pressure.	Lbs. added _____ Time added _____
7.	Bring 8-2 to 140 RPM, hold 2 minutes then plug to stop. Repeat twice.	Time end _____
8.	Bring 8-2 to 870 RPM, hold 5 minutes and skim to 10 gallon heel.	Time end _____
9.	Beckman reading on 8-2	Meter _____ Factor _____
10.	Plug 8-2 to stop.	Time stop _____

[redacted]

[redacted]

PRODUCT EXTRACTION

Date _____
Run No. _____

DECLASSIFIED

IV. PRODUCT CAKE SOLUTION

1.	Beckman reading on 8-4	Meter_____ Factor_____
2.	Add 2710 lbs. of 60% Nitric Acid (Line G) to 8-1 via 8-1A.	Lbs. added_____ Time added_____
3.	Flush 8-1A to 8-1 with 50 lbs. of process water.	Lbs. added_____ Time added_____ 8-1 Wt.Ftr._____ Lbs._____
4.	Start 8-1 agitator	8-1 agitator on_____
5.	With cooling water on the jacket, circulate through 8-1 spray for 20 minutes. HOLD 8-1 TEMPERATURE BELOW 50°C.	8-1 jacket water on_____ Time start_____ Time end_____ Maximum temperature_____ 8-1 jacket water off._____
6.	Jet slurry from 8-2 to 8-4.	Time jetted_____
7.	Using the "A" jet, jet 330 lbs. of acid from 8-1 to 8-2.	8-2 Wt.Ftr._____Lbs._____
8.	Bring 8-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time start_____Time end_____
9.	With 8-2 stopped, jet 8-2 to 8-4.	Time jetted._____
10.	Start 8-4 agitator and jacket cooling water.	8-4 agitator on_____ 8-4 jacket water on_____
11.	Using the "A" jet, jet 330 lbs. of acid from 8-1 to 8-2.	8-2 Wt. Ftr._____ Lbs._____
12.	Shut off 8-1 agitator.	8-1 agitator off_____
13.	Bring 8-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time start_____ Time end_____
14.	With 8-2 stopped, jet 8-2 to 8-4.	Time jetted_____
15.	Using the "A" jet, jet 330 lbs. of acid from 8-1 to 8-2.	8-2 Wt. Ftr._____ Lbs._____
16.	Bring 8-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time start_____Time end_____
17.	With 8-2 stopped, jet 8-2 to 8-4.	Time jetted_____
18.	Using the "A" jet, jet 330 lbs. of acid from 8-1 to 8-2.	8-2 Wt.Ftr._____ Lbs._____

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[redacted]

Date _____
Run No. _____

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IV. PRODUCT CAKE SOLUTION (Cont'd)

19. Bring 8-2 to 140 RPM, hold 2 min. then plug to stop. — Time end _____

20. With 8-2 stopped, jet 8-2 to 8-4. — Time jetted _____

21. Beckman reading on 8-2 — Meter _____ Factor _____

Note: If Beckman reading is greater than on the 10 scale, notify the supervisor and with his approval add the remaining acid in 330 lb. shots until the Beckman reaches this level or the acid is used up.

22. Bring 8-2 to 10 RPM and using the "A" jet remainder of acid from 8-1 to 8-2 to 8-4. Keep the bowl as empty as possible during this operation. When the 8-1 to 8-2 transfer stops, shut off the "A" jet and plug 8-2 to stop. — Time start _____ Time end _____ Time stop _____

23. Jet 8-2 to 8-4. — Time jetted _____

24. Using the "B" jet, again jet 8-1 to 8-2. When the transfer stops, shut off jet and read 8-2 manometer. — Time start _____ Time end _____ 8-2 Manometer _____ Lbs. _____

25. Jet 8-2 to 8-4. — Time jetted _____

26. Beckman reading on 8-2. — Meter _____ Factor _____

27. Shut off 8-3 air sparger and read Wt.Ftr., Temp, and Sp.Gr. when Sp.Gr. meter becomes constant, then start 8-3 air sparger again. (TW approx. 32,500 lbs.) — 8-3 Air sparger off _____ 8-3 Wt. Ftr. _____ Lbs. _____ 8-3 Sp. Gr. _____ Temp. _____ 8-3 Air sparger on _____

28. Call dispatcher, give charge data and request that sample 8-3 WS be taken. — Time called _____ Time sampled _____

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PRODUCT EXTRACTION

Date _____
Run No. _____

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IV PRODUCT CAKE SOLUTION (Cont'd)

29.	Agitate 8-4 for 1 hour below 40 C. after the final addition of dissolving acid (Step 25 above.)	Time end _____
30.	Stop 8-4 agitator and read Wt. Ftr., Temp. and Sp.Gr. when Sp.Gr. meter becomes constant, then start 8-4 agitator again. (TW approx. 3500 lbs.)	8-4 Agitator off _____ 8-4 Wt. Ftr. _____ Lbs. _____ 8-4 Sp.Gr. _____ Temp. _____ 8-4 Agitator on _____
31.	Beckman reading on 8-4	Meter _____ Factor _____
32.	Call dispatcher, give charge data and request that sample 8-4-P be taken.	Time called _____ Time sampled _____
33.	When the dispatcher approves, jet 8-4 to 12-6. When the transfer stops, put jet on air for 10 minutes and again jet 8-4 and 12-6.	Time approved _____ Time start _____ Time end _____
34.	Shut off 8-4 agitator and jacket cooling water.	8-4 Agitator off _____ 8-4 Jacket water off _____

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[redacted]

PRODUCT EXTRACTION

Date _____
Run No. _____

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V METAL WASTE SOLUTION

1. Beckman reading on 8-3 — Meter ____ Factor _____

2. When the dispatcher approves, shut of the air sparger and jet the first half of metal waste solution in 8-3 to 9-1.
   - Time approved _____
   - 8-3 air sparger off _____
   - Time start _____
   - Time end _____
   - 8-3 Wt.Ftr. ____ Lbs. _____

3. Turn on 8-3 air sparger.
   - 8-3 air sparger on _____

4. When the dispatcher approves, jet the last half of metal waste solution in 8-3 to 9-1.
   - Time approved _____
   - Time start _____ Time end _____
   - 8-3 Wt. Ftr. ____ Lbs. _____

5. Shut off 8-3 air sparger and jacket
   - 8-3 air sparger off _____
   - 8-3 Jacket water off _____

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CHAPTER V - EXTRACTION

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HANFORD ENGINEER WORKS TECHNICAL MANUAL

SECTION C - SEPARATIONS

CHAPTER VI - DECONTAMINATION

During the decontamination cycles, the fission elements are largely eliminated from the product. It is necessary to reduce the activity associated with the product by a factor of 10^5 in the Canyon Building before transfer to the more lightly shielded Concentration Building can be made safely. Some concentration of plutonium takes place during decontamination: in the extraction step the product is associated with 88 pounds of $BiPO_4$, as it enters the Concentration Building it is associated with 43 pounds of $BiPO_4$.

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CHAPTER VI - DECONTAMINATION

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PROCESS

General

During the decontamination cycles, the fission elements are largely removed from the product in a series of alternate precipitations of by-products and product. Advantage is taken of the difference in solubility of plutonium phosphate in its oxidized and reduced state to effect the separations. The product in the extraction cake solution is oxidized with sodium bismuthate to the VI valence state, in which form it is not carried by bismuth phosphate. The bismuth phosphate in solution is then reprecipitated, along with added ceric and zirconium phosphates which aid in removing phosphate insoluble fission elements, leaving the product in solution. After separation of this precipitate, the product is reduced to the IV state with ferrous ion and more bismuth phosphate is precipitated in the presence of ammonium silicofluoride. This bismuth phosphate carries the product, leaving in solution the major portion of any phosphate insolubles "leakage through" the by-product step, by solubilization with the silicofluoride, and a large portion of those phosphate soluble fission elements which have carried through to this point. This series of operations constitutes the first decontamination cycle. The second decontamination cycle is the same as the first, except no cerium and zirconium "scavengers" are added.

Oxidation

The oxidation states of plutonium have been discussed previously (See Chapter I), as have also the various oxidants capable of oxidizing plutonium to the VI state (PuO_2 - II). Sodium bismuthate is the only chemical which has been used for the primary oxidation at Hanford and the pilot plant. Alternate agents, $Na_2Cr_2O_7$, Ce^{+4}, $KMnO_4$, Pb_3O_4, have shown satisfactory performance in the laboratory, less promising results in semi-works, and have not been evaluated in plant operation. Since sodium bismuthate is decomposed in strong acids, sodium dichromate is also added as a "holding oxidant" to stabilize the oxidized plutonium.

Before any oxidation can be successful, the product to be oxidized must be in solution. Oxidation with sodium bismuthate is affected by such process variables as acid strength, temperature, presence of other salts, and bismuthate concentration. At $NaBiO_3$ concentrations below 0.005 M, the oxidation of plutonium is incomplete, decreasing rapidly with decreasing bismuthate concentration; at 0.005 M, the oxidation is critical with respect to variations in temperature and acidity; at concentrations above 0.01M, oxidation is complete and less affected by other process variations. At any given temperature, the rate of bismuthate decomposition increases with increasing acidity, and at any given acidity, the rate of decomposition increases with increasing temperature. The presence of dissolved bismuth phosphate decreases the rate at which sodium bismuthate is decomposed; the presence of sodium dichromate slightly increases the decomposition rate. Using 0.01M $NaBiO_3$, the range of acidity in which oxidation is complete within an hour is 1 to 8.5 N. In 1N acid, the temperature must be increased to 75° to obtain appreciable bismuthate decomposition;

in 8.5N acid the temperature must be reduced to 40-45 °C to prevent too rapid bismuthate decomposition. For practical purposes the acidity for oxidation must be between 3.5N and 8.5N; the lower limit determined by the solubility characteristics of bismuth phosphate, the upper limit determined by the completeness of product oxidation. In 10 N acid, plutonium is not completely oxidized under the process conditions.

Product oxidation by sodium bismuthate, or other oxidants, is greatly inhibited by the presence of fluoride or silicofluoride ions. Using higher concentrations and more drastic conditions, plutonium has been oxidized successfully in the presence of silicofluoride in the laboratory.

By-Product Precipitation

The purpose of making the by-product precipitation is to remove the dissolved bismuth phosphate from the oxidized solution, together with as much of the phosphate insoluble fission activity as possible, with as little product as possible.

Whether the by-product precipitation is made in the presence or absence of scavengers, process variables such as the method of striking, temperature of striking, concentration of nitric acid, concentration of phosphoric acid and length of digestion have very little effect upon the product lost in the precipitate. Nitric acidity up to 2 N has been shown to have no effect upon the product loss; the completeness of the preceding cake solution and oxidation steps being the single factor of greatest importance affecting product loss in the by-product step.

The by-product bismuth phosphate precipitation is made by a "dilution strike". Additional phosphoric acid is added to reduce the bismuth phosphate solubility to a minimum. The order of the dilution, i.e., the oxidized solution into water or water into the oxidized solution, has been shown to have no effect upon product loss, decontamination or precipitate characteristics. The precipitate consists of long rods, approximately 2x10 microns, with a surface area of approximately 0.2 square meters/gram. When cerium and zirconium are also precipitated, the surface area is increased approximately 100-fold to 20-25 square meters/gram.

The solubility of the by-product cake is approximately that of the product cakes (See Figure 1) although the possibility of forming the less soluble beta form is greater, due to the very low iron concentration in the by-product step (iron has been shown to prevent the formation of the beta form). The solubility of the beta form is shown in Figure 2. The rates of solution of the various precipitates is given in Figure 3.

When ceric and zirconium phosphates are also precipitated as scavengers in the by-product step, the product loss is increased several-fold, due largely to the increased cake size and the inability to wash it effectively.

Zirconium phosphate is a gelatinous, bulky, difficultly centrifugable precipitate which, when coprecipitated with ceric phosphate, becomes

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FIGURE 1

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SOLUBILITY OF PLANT PRECIPITATE CAKES

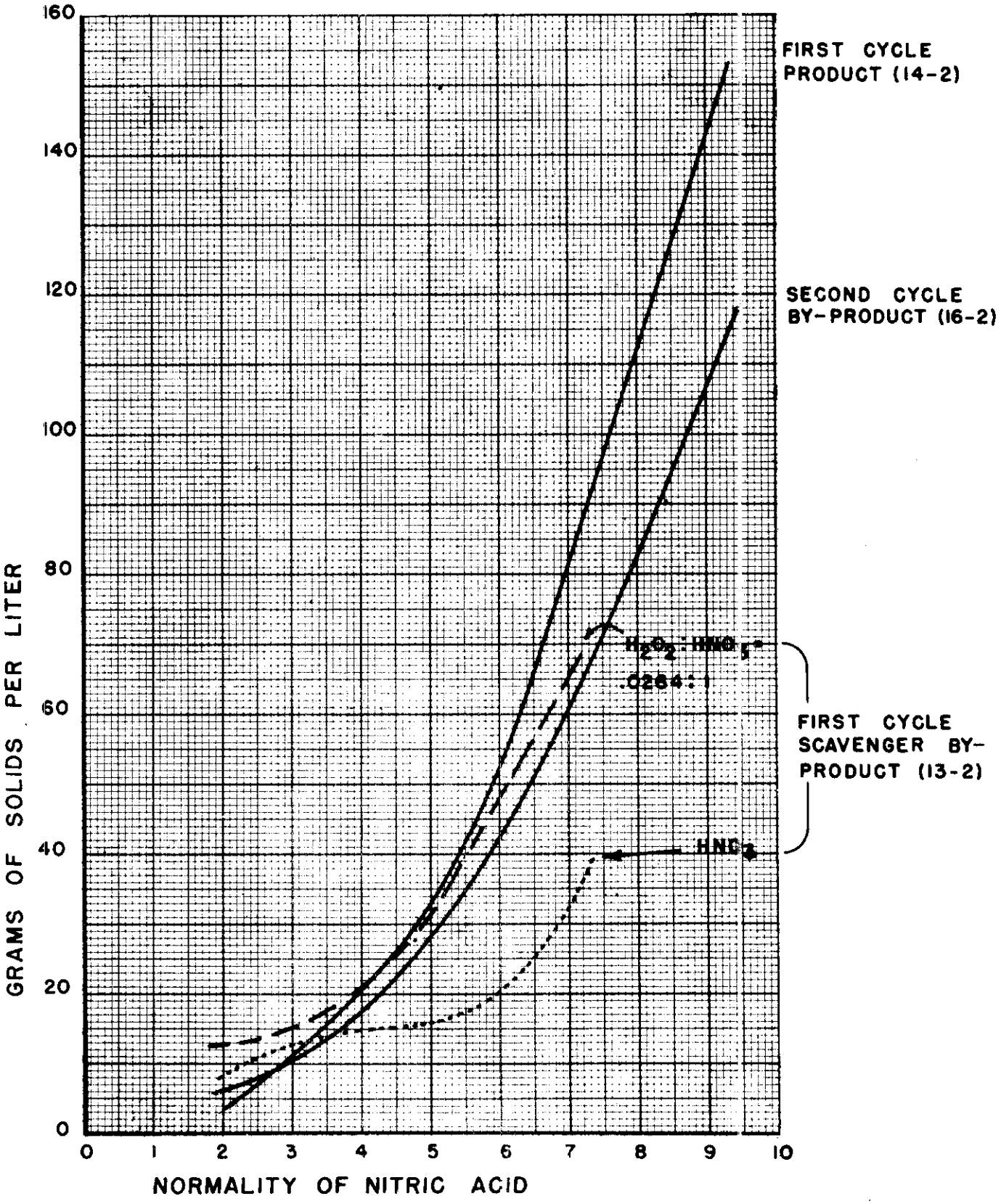

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FIGURE 2

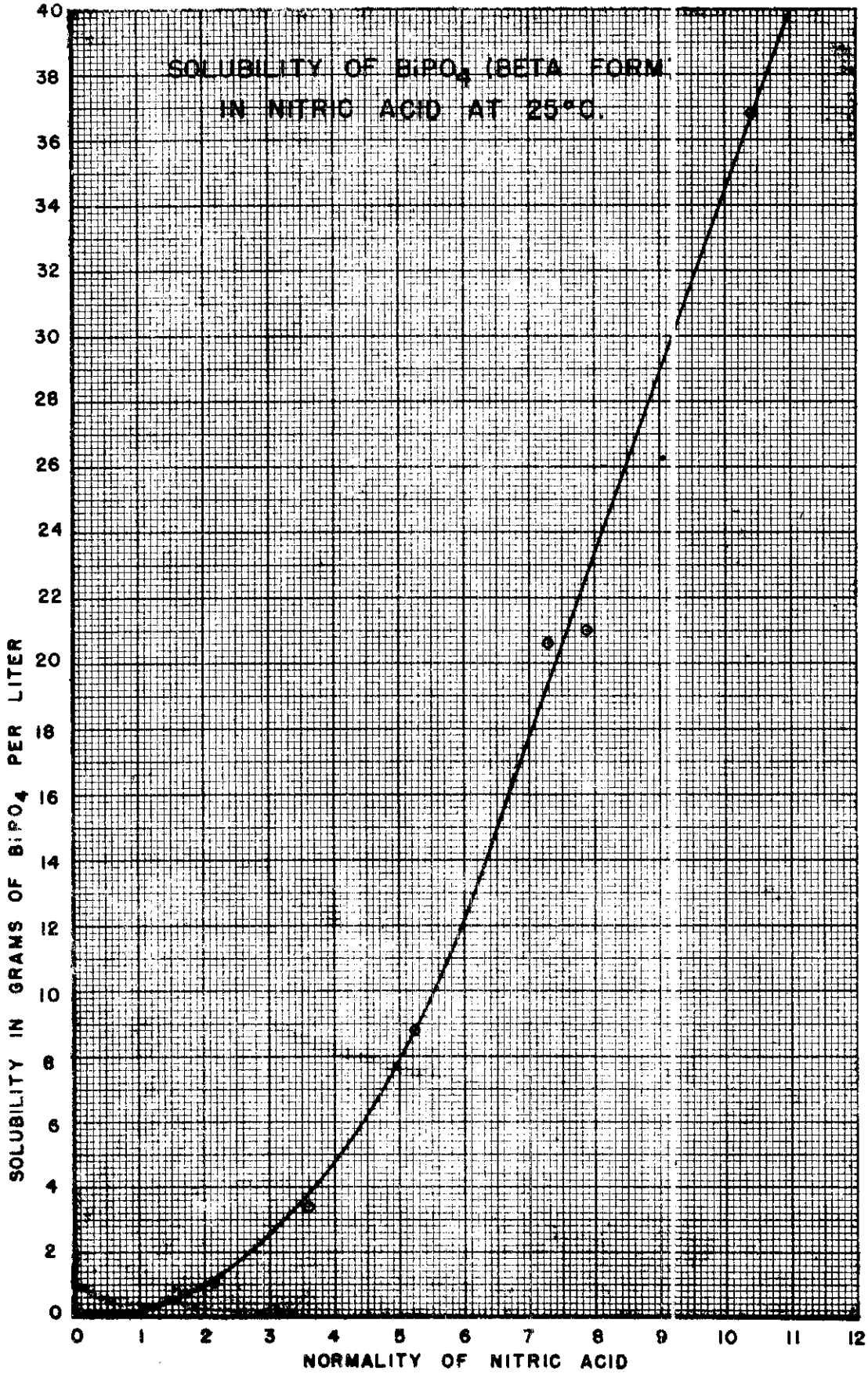

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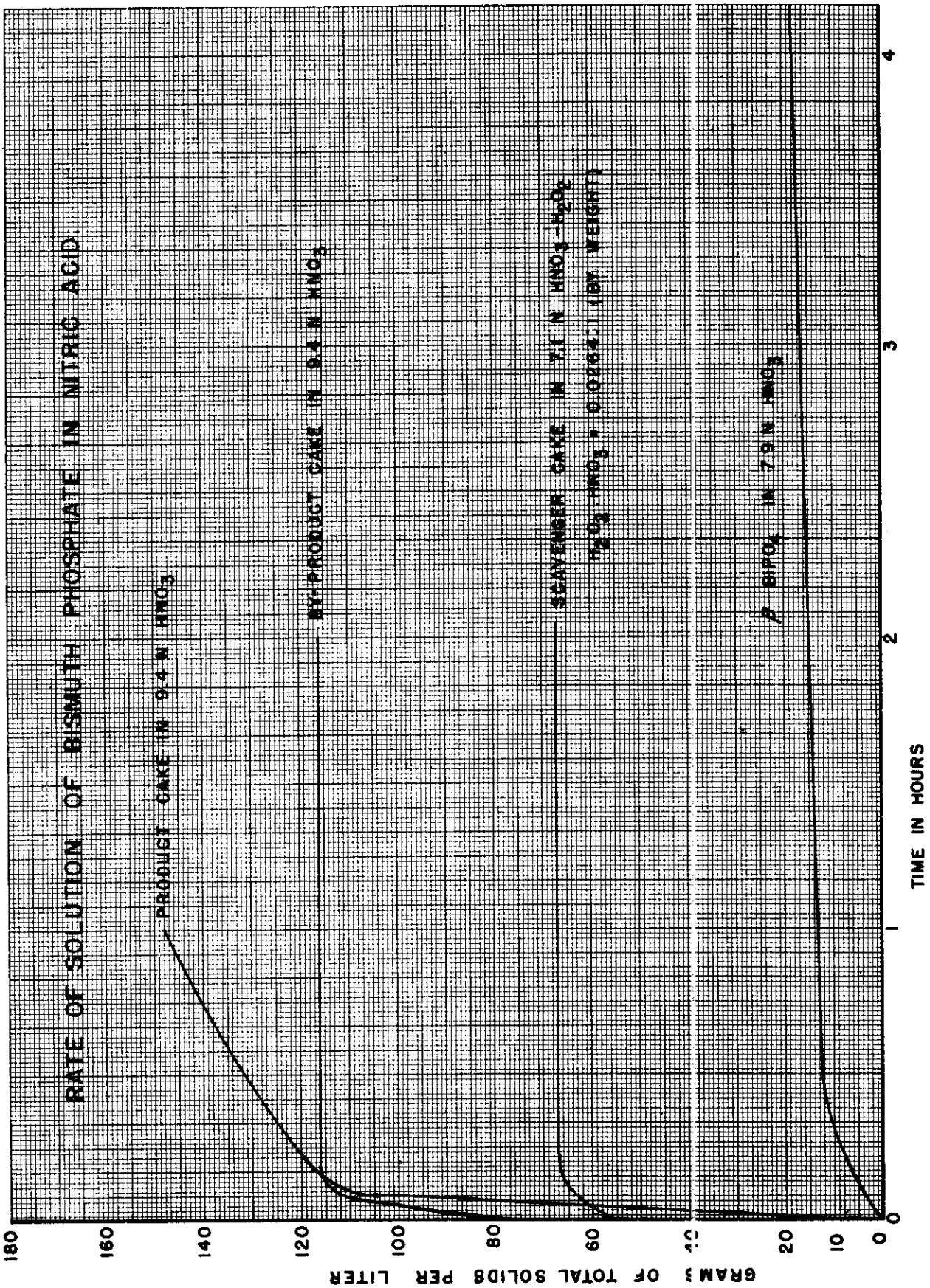
FIGURE 3



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sufficiently "cemented" to be easily centrifugable. The ceric phosphate also aids in removing the cake from the centrifuge bowl. Zirconium phosphate is essentially insoluble in nitric acid and ceric phosphate is only slightly soluble. However, if hydrogen peroxide is added to the dissolving acid, it reduces the ceric phosphate to the soluble cerous phosphate with liberation of oxygen. This gas liberation plus the dissolving of the bismuth and cerium aids in slurrying the insoluble zirconium out of the bowl. A mixture of nitric acid and peroxide is not stable in stainless steel, so the peroxide must be added separately to the centrifuge bowl.

Reduction

After the phosphate insoluble fission elements have been largely removed with the by-product precipitate, the plutonium is reduced to the IV state in order that it may be removed from solution and separated from the phosphate soluble fission elements.

The majority of the reducing agents which have been shown to reduce product - ferrous ion, hydrogen peroxide, hydrazine, hydroxylamine, sodium nitrite, uranous ion, sulfur dioxide, oxalic acid and hypophosphorous acid reduce it to the poorly carried (III) state or mixtures of (III), (IV) and (V) states. Uranous ion reduces plutonium directly to the (IV) state and has been demonstrated on both the laboratory and semi-works scale. However, in the absence of silicofluoride, the decontamination obtained in the product precipitation step is lowered several fold over that obtained with ferrous ion (data are unavailable on the effect of silicofluoride).

Ferrous ion rapidly reduces plutonium to the (III) state but the excess Fe (II) and Pu (III) are rapidly oxidized by heating in 1N HNO_3 solutions to Fe (III) and Pu (IV) respectively. In addition, decontamination is improved by its use which makes it the preferred reducing agent of all those investigated. Ferrous ion has been used continuously in both the pilot plant and Hanford for reducing product in the decontamination cycles.

Process variables affecting reduction with ferrous ion are: ferrous concentration, temperature, order of addition, and nitric acid concentration. Iron concentrations in excess of the holding oxidant present, within the range 0.002M to 0.05M, have been found satisfactory; at the lower concentration, the iron may be oxidized by air or nitric acid before reducing all of the plutonium, at higher concentrations (0.1M) interference with product carrying on bismuth phosphate results. The reverse procedure, transferring the oxidized process solution into a tank containing the iron solution, may result in oxidation of all the iron before all of the product has been transferred, with a resultant loss in the subsequent product precipitation step.

Temperature and nitric acid concentration influence somewhat the oxidation of Pu (III) to Pu (IV), but their effect on the Fe (II) is of greater importance. At higher temperatures and acidities the iron may be completely oxidized before it becomes uniformly mixed throughout the process solution, resulting in incomplete reduction of product.

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Product Precipitation

In order to obtain some volume reduction during the decontamination cycles for the purpose of improving the process performance in the Concentration Building, the product is carried on successively smaller quantities of bismuth phosphate in the two cycles. This is accomplished by reducing the concentration of bismuth in the product solution.

Due to the increase in the process volumes of the first decontamination cycle over that of the extraction step, the bismuth concentration required to produce the same weight of bismuth phosphate is decreased to 1.8 grams/liter. This concentration has been reduced successfully to 1.4 grams/liter, and laboratory data have shown that reducing it to 1.0 grams/liter results in only slight increase in waste losses. At lower bismuth concentrations, however, the process becomes more critical to variation in other variables as shown in the following:

Interdependence of Process Variables during Product Precipitation

Number of Precipitations	Bi Conc., g/l	Strike Time*, Minutes	Digestion Time, Hrs.	Digestion Temp., °C	Avg. Waste Loss, %
12	1.8	45	2	75	0.27
13	1.4	45	2	75	0.65
12	1.4	120	2	75	0.33

*Time required to add both the bismuth solution and the phosphoric acid.

The influence of phosphoric acid concentration upon product losses in this step has not been determined at Hanford, but laboratory results have shown that the effect is very minor within the concentration range of 0.2M to 0.6 M.

At nitric acid concentrations up to 1.5 N, this variable has very slight effect upon product carrying. As the acidity is increased, however, increased waste losses occur due to the increased solubility of bismuth and plutonium phosphates. At high acidities (1.75 N or higher), measures designed to decrease solubility of bismuth and plutonium phosphate - increased bismuth concentration, increased phosphoric acid concentration, increased temperature, and slower rate of precipitation-result in greater carrying and lowered losses.

The effect of temperature on product precipitations was discussed in Chapter V. The general effects are the same as in the extraction step, except to a lesser degree due to the absence of uranium. Temperatures in the range 85 to 65 °C influence product loss only slightly; below 65 °C the product losses increase with decreasing temperature.

Digestion time has been shown to have less effect upon the product carrying than the strike time. The bismuth phosphate formed during the product strike is less coarse than the extraction precipitate, but much more granular

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than the by-product precipitates. It has a surface area approximately 1 square meter/gram, which is not altered by the 2-hour digestion. The solubility characteristics of the product precipitates are presented in Figures 1 and 3.

Bismuth Phosphate Metathesis

In order to insure complete solution of the product-containing bismuth phosphate precipitates, it has been found inadvisable to reduce the HNO_3/$BiPO_4$ weight ratio below 22. In certain instances, however, it may become desirable or necessary to dissolve a cake in less than this amount of acid. For these instances, plant tests have demonstrated that the bismuth phosphate can be readily converted to the more acid soluble bismuth hydroxide by treating with 35% KOH. The potassium phosphate is removed by water washing before attempting to dissolve the bismuth hydroxide in the reduced quantity of acid. A loss of approximately 0.4% product results from such metathesis procedure.

Factors Influencing Decontamination

In order to obtain maximum decontamination by making precipitations alternately from oxidized and reduced solutions, it is necessary to incorporate as much fission activity as possible with the precipitates made in the oxidized solutions and as little as possible with the precipitates made in the reduced solutions.

Laboratory studies made on the incorporation of fission elements into bismuth phosphate precipitates have revealed that dynamic recrystallization plays a very important role in the up-take or self-purification of these precipitates. In solutions where the bismuth phosphate solubility is very low, it was found that the pickup of activity could be described quantitatively on the basis of diffusion through the solid. In general such aging processes as crystal self-perfection, agglomeration, irreversible cementing of particles, and polymorphic transitions were found to be unimportant or to become unimportant within a few minutes after precipitation.

The factors having the greatest effect upon the decontamination obtained are: concentration of fission elements, completeness of solids separation, and presence of added ions. At low concentrations (5 grams Pu/ton) the quantity of fission elements present in the extraction cake is small enough to permit the by-product bismuth phosphate precipitate to carry most of the phosphate insoluble elements, reducing their concentration by a factor of approximately 50-100. However, at higher concentrations (250 grams Pu/ton) the quantity present exceeds the solubility product of columbium and zirconium and the bismuth phosphate fails to remove them effectively from solution, decreasing their concentration only by a factor of 3-5. For this reason, cerium and zirconium "scavengers" are added to remove the phosphate insolubles more effectively, resulting in a decrease of fission activity of approximately 25-50 fold. The cerium and zirconium are added to a phosphate-free slurry to permit more complete exchange between the normal and radioactive ions before making the precipitation. In addition to this improved carrying, the

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flocculent ceric and zirconium phosphate help to sweep insoluble "crud" and mud from the solution. Precipitating and removing another cerium or cerium-zirconium phosphate precipitate from the same solution further reduces the fission activity by a factor of 2, but at a considerable cost of time.

After the first decontamination cycle, the phosphate insoluble fission elements are such a small fraction of those remaining with the product that another scavenger by-product precipitation reduces the total activity only slightly more than does a regular by-product precipitation. The bismuth phosphate by-product precipitation made in the second decontamination cycle reduces the total activity present by a factor of approximately 2-4.

In the presence of added ions such as dichromate or ferric, even at low concentrations (0.001 M), there is a marked up-take of these ions with a resultant reduction in the rate of fission element up-take.

During the product precipitation steps, the presence of added silicofluoride ion (0.05M) materially aids in decreasing the pick-up of residual phosphate insoluble activity. This results from solubilizing, by complexing, any remaining columbium or zirconium activities. Other metal fluo-complexes have this same property and have shown decontamination factors in the laboratory greater than or equal to those obtained with silicofluoride: fluomolybdate, fluoborate, fluovanadate, and fluouranate. The presence of silicofluoride, however, has a compensating, injurious effect upon decontamination; it increases the amount of phosphate soluble rare earth elements carried by the bismuth phosphate product cake. The net effect, however, is an increase in decontamination resulting from its use; a factor of 5-10 without silicofluoride and a factor of 10-20 with silicofluoride. The increased quantities of rare earth fission elements carried with the product present no special problem, since a special step designed for their removal is included in the concentration process (Chapter VII).

The effect of iron in the process solutions, noted previously, of excluding fission elements from the bismuth phosphate precipitate, may slightly decrease the removal of fission elements with the by-product precipitates, but increases several fold the self-purification of the product precipitates. Laboratory comparisons of ferrous, uranous, and oxalate ions for reducing plutonium showed relative decontamination factors for the product step of 20, 9 and 4 respectively.

Before the product can be processed with safety in the Concentration Building, the gross gamma fission activity must be reduced by a factor of 10^5. This is normally accomplished by the two decontamination cycles. However, a one-cycle process has been demonstrated in the laboratory to produce 10^5 decontamination, but at a cost of 5-10% yield.

Recovery of Product from Wastes

The wastes from the decontamination cycles consist of the scavenger by-product, the regular by-product, and the two product effluents. Losses in the product effluent great enough to justify reworking would result from

either incomplete reduction, incomplete carrying, or incomplete solids separation. In any of these cases the waste would be returned to the precipitator and either recentrifuged or processed as a regular charge, care being taken not to accumulate volume great enough to overflow the tanks.

In case the regular bismuth phosphate by-product precipitate had to be reworked for the recovery of product, it would be treated as a product cake (oxidized in the precipitate dissolver and returned to the precipitator where it would be processed as a regular by-product precipitation).

Reworking the scavenger by-product cake for the recovery of product may be accomplished by several methods. One method, developed in the laboratory but not tested in the plant, consists of solubilizing the zirconium phosphate by making the solution 0.3 M in ammonium silicofluoride, destroying residual peroxide with ferric ion, oxidizing the product with sodium bismuthate (0.1M) diluting and making another by-product precipitation without added scavengers, then proceeding with a regular product precipitation step. Another method, tested in the plant, consists of solubilizing the zirconium phosphate with ammonium silicofluoride followed by a product precipitation in the presence of 0.03 M ferrous ion and 0.3 M silicofluoride ion. This permits separation of the bismuth and plutonium from cerium, zirconium and silicofluoride. The product cake is then dissolved, oxidized, and processed as a regular by-product step, without scavengers. Since the presence of 50% extra bismuth makes the solution and subsequent oxidation of the product more difficult, a metathesis of the cake to bismuth hydroxide before solution in nitric acid would be beneficial.

EQUIPMENT

Following the extraction step in Section 8, the product solution is transferred to Section 12 where it is oxidized before proceeding to Section 13 wherein the first by-product precipitation is made. Sections 13,14,16, 17,18, and 19 are also "standard sections", which have been described earlier. Figures 4 and 5 show views of Cells 27 and 28, Section 14, in which the product precipitation of the first decontamination cycle is made.

Section 12 - Product Storage and Oxidation

Section 12 L contains two tanks (Drawing W-72798) for storage of crude product solution from the extraction step. These are 6 1/2 foot diameter by 14 foot high jacketed unagitated tanks. The over-flow capacity of each is 3230 gallons.

The connections to each tank include thermometer, liquid level, sampler, sparger, transfer to the other storage tank, and transfer to Section 12 R.

In 12 R there are two solution tanks identical in design and instrumentation with those in the standard sections. They are operated from "K" panels

FIGURE 4

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CELL 27 - SECTION 14

FIGURE 5

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CELL 28 - SECTION 14

as shown in Figure 6. These tanks are used for oxidation of the crude product solution which is then jetted to either Section 13 or 16 for the first decontamination cycle.

Section 13, 16 and 18 - By-Product Precipitation

Sections 13, 16 and 18 are standard sections. The cell equipment and instrumentation in them are identical to that described in Chapter V, and are shown in Figures 7, 9 and 11. The gauge boards differ from the standard section board in that the catch tank panel "F" is replaced by an "L" panel which has one less gang valve. On the right of Section 18 board is added an "H" panel which carries a safety meter recorder attached to the meters in Sections 16, 17 and 18.

The gallery tank arrangement is also similar to that in a standard section. The "A" tank supplies 60% nitric acid to the precipitator for cake dissolving. The "C" tank can be used to feed nitric acid directly to the centrifuge bowl. The phosphoric acid added in a by-product precipitation is approximately 300 pounds and is added through the 75 gallon "E" tank rather than through the 400-gallon "A" tank.

The standard arrangement is used for transfer lines within the section. The outlets to the trench consist of two lines for sending the by-product cake solution from the solution tank to waste disposal in Section 15, and a connection from the catch tank to the precipitator in the next section. The precipitators in Sections 13 and 16 can receive product solution direct from storage. In Sections 16 and 18 the precipitators can also receive product solution from a preceding decontamination cycle: Section 16 is connected to the solution tank in Section 14, Section 18 to the solution tanks in both sections 17 and 14. This arrangement makes it possible to run two successive cycles in any two pairs of the three double Sections 13-14, 16-17 and 18-19.

A special line in Section 19 jets from the solution tank to the Section 18 precipitator. This permits a troublesome batch, which decontaminates poorly, to be segregated in these sections and put through successive decontamination cycles until the activity is sufficiently reduced.

Sections 14, 17 and 19 - Reduction and Product Precipitation

These are also standard sections, similar to Section 3. The gauge boards have the solution tank panel "W" replaced by "K" which has one less gang valve. In Section 14, an "H" panel with safety meter recorder for Sections 13-16 inclusive, is added.

The gallery scale tank arrangement differs from Section 8 in that an extra tank is placed in position "F", for adding oxidizing agents to the solution tank.

Process piping between vessels within a section is connected in the standard manner. Other lines are shown in Figures 8, 10 and 12. Product solution

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FIGURE 6

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 12.

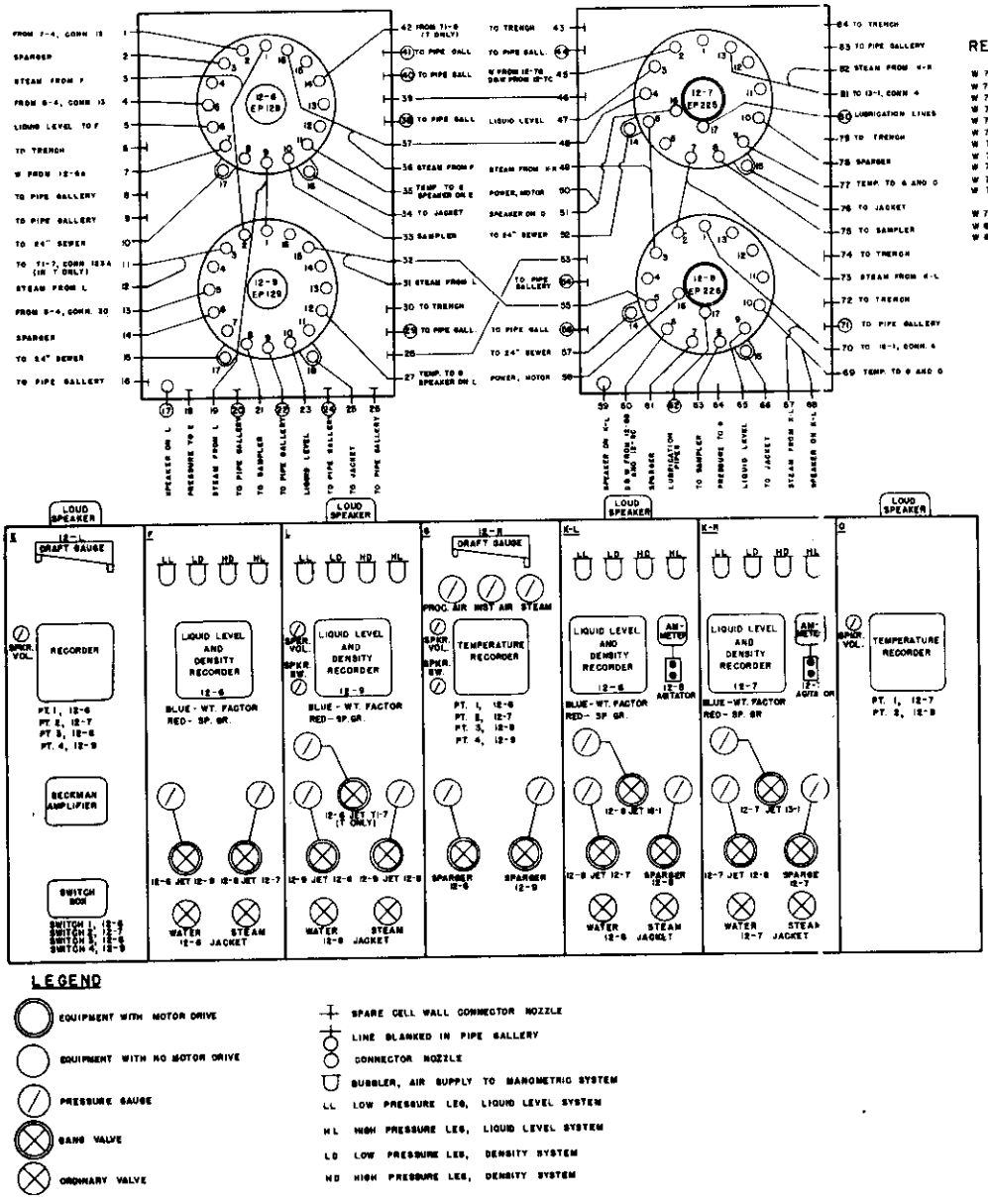

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FIGURE 7

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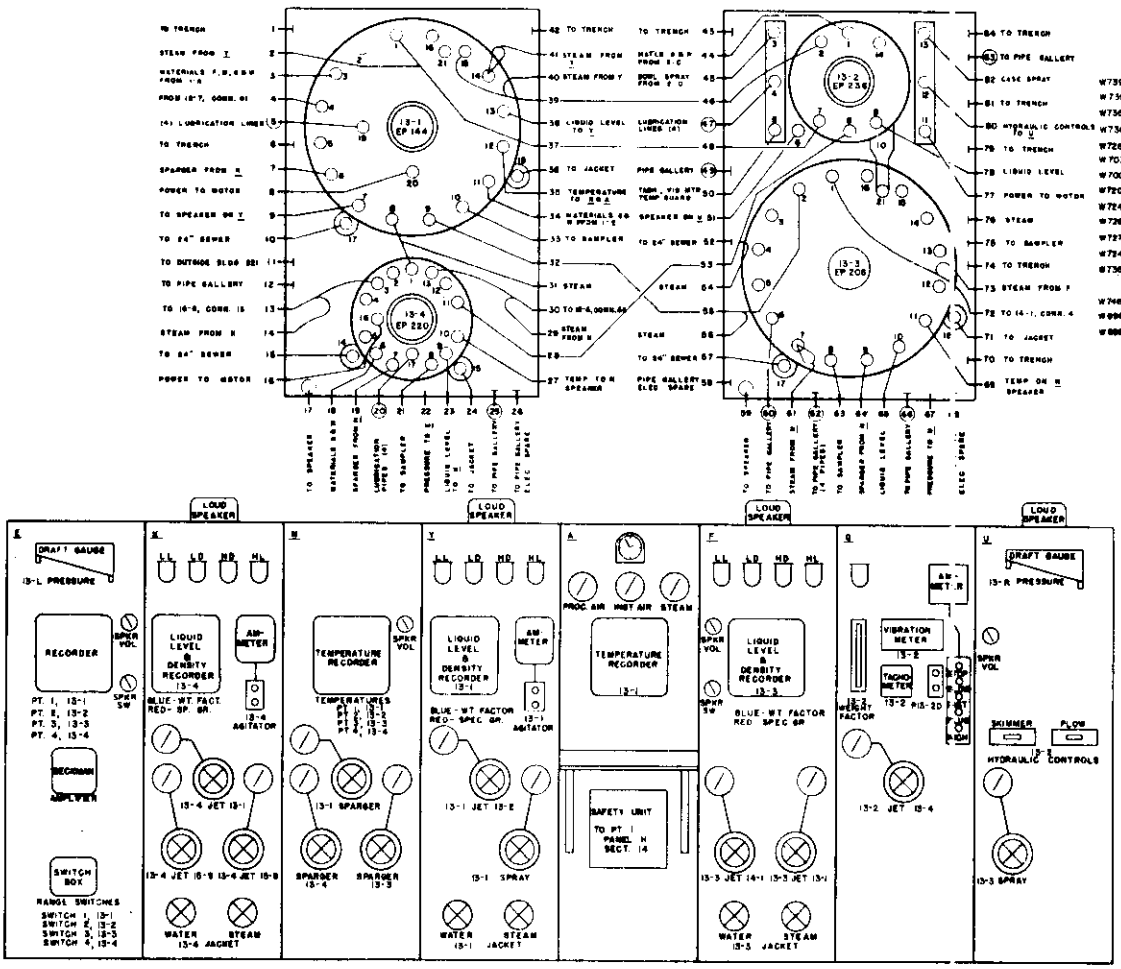
CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 13



LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WELL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM

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FIGURE 8

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 14

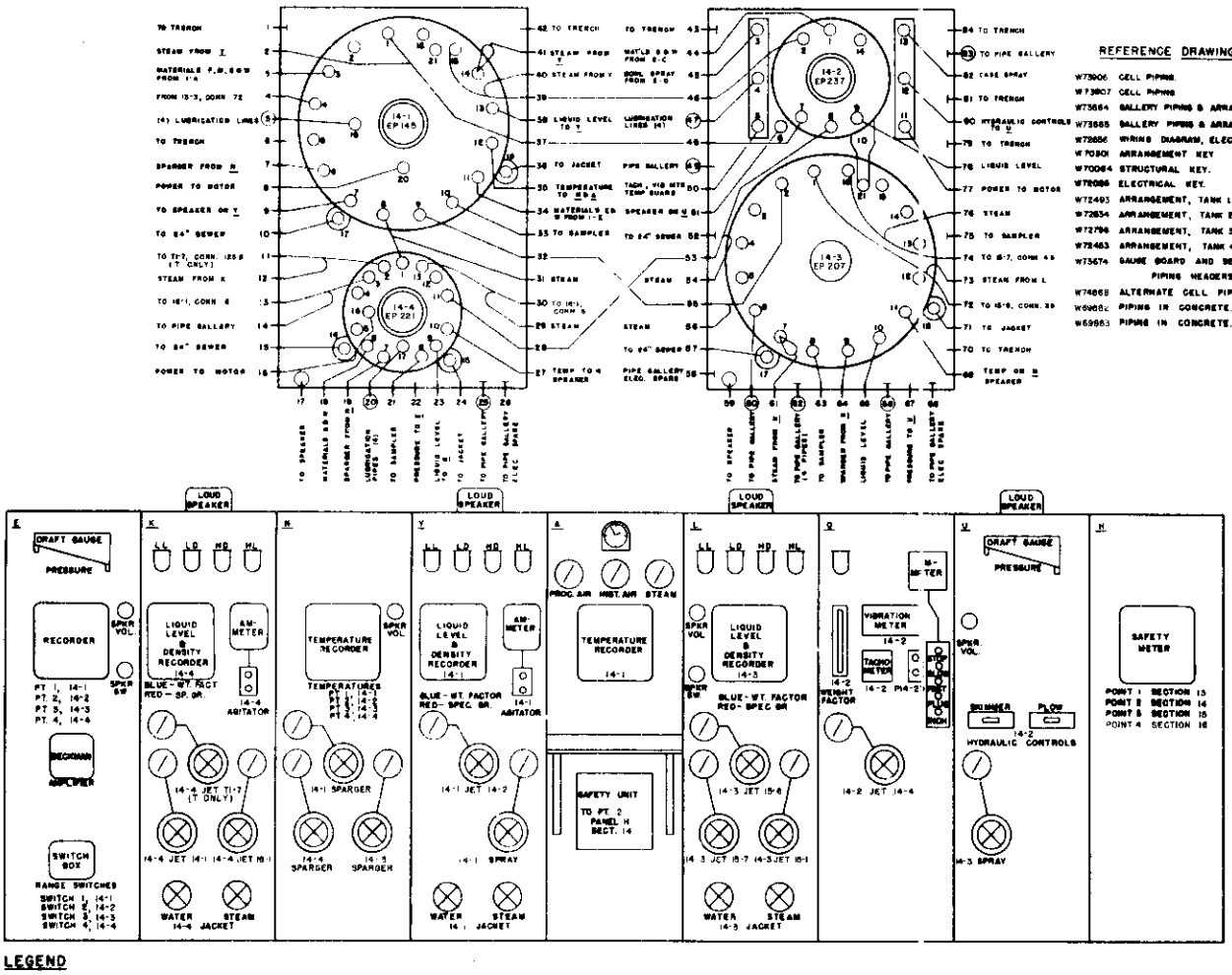

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FIGURE 9

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 16

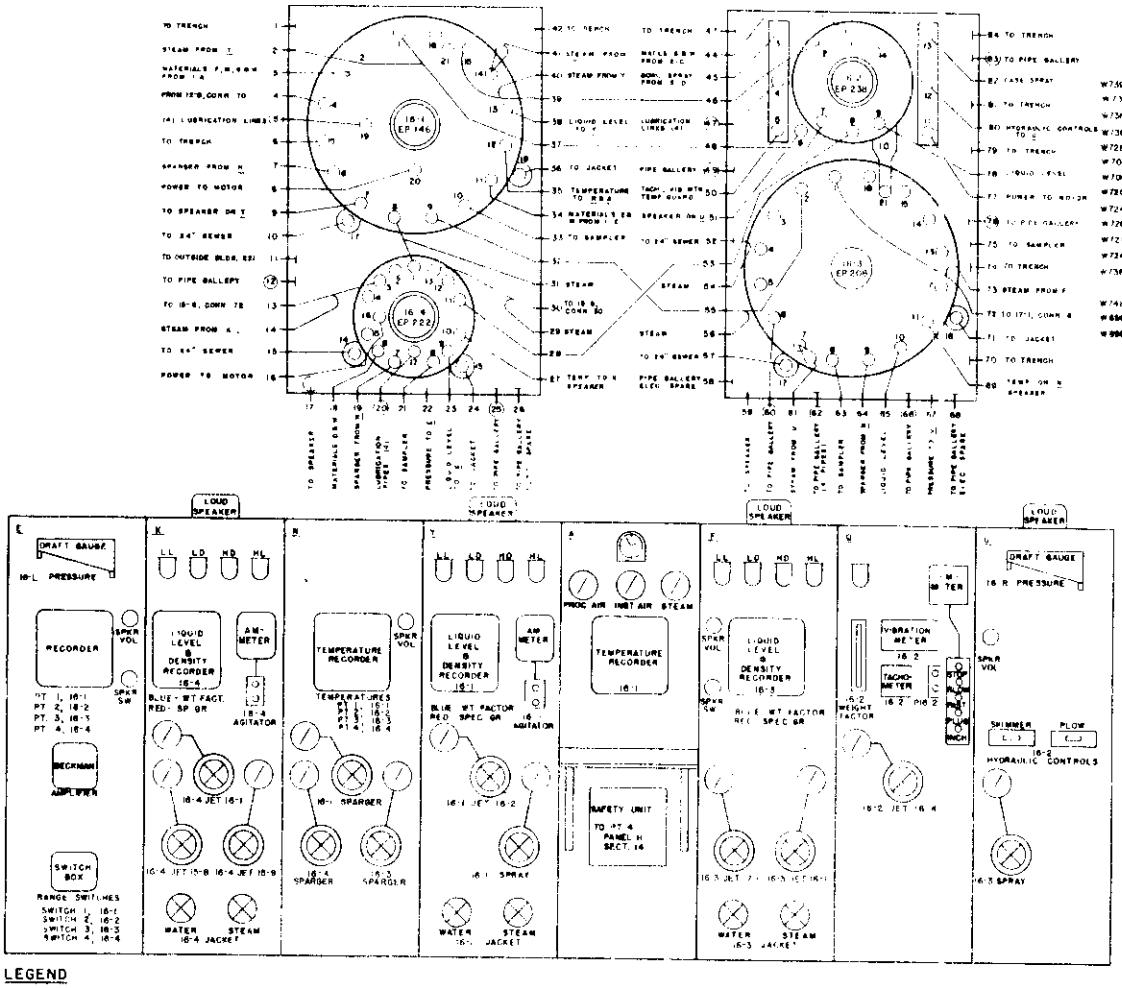

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FIGURE 10

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 17

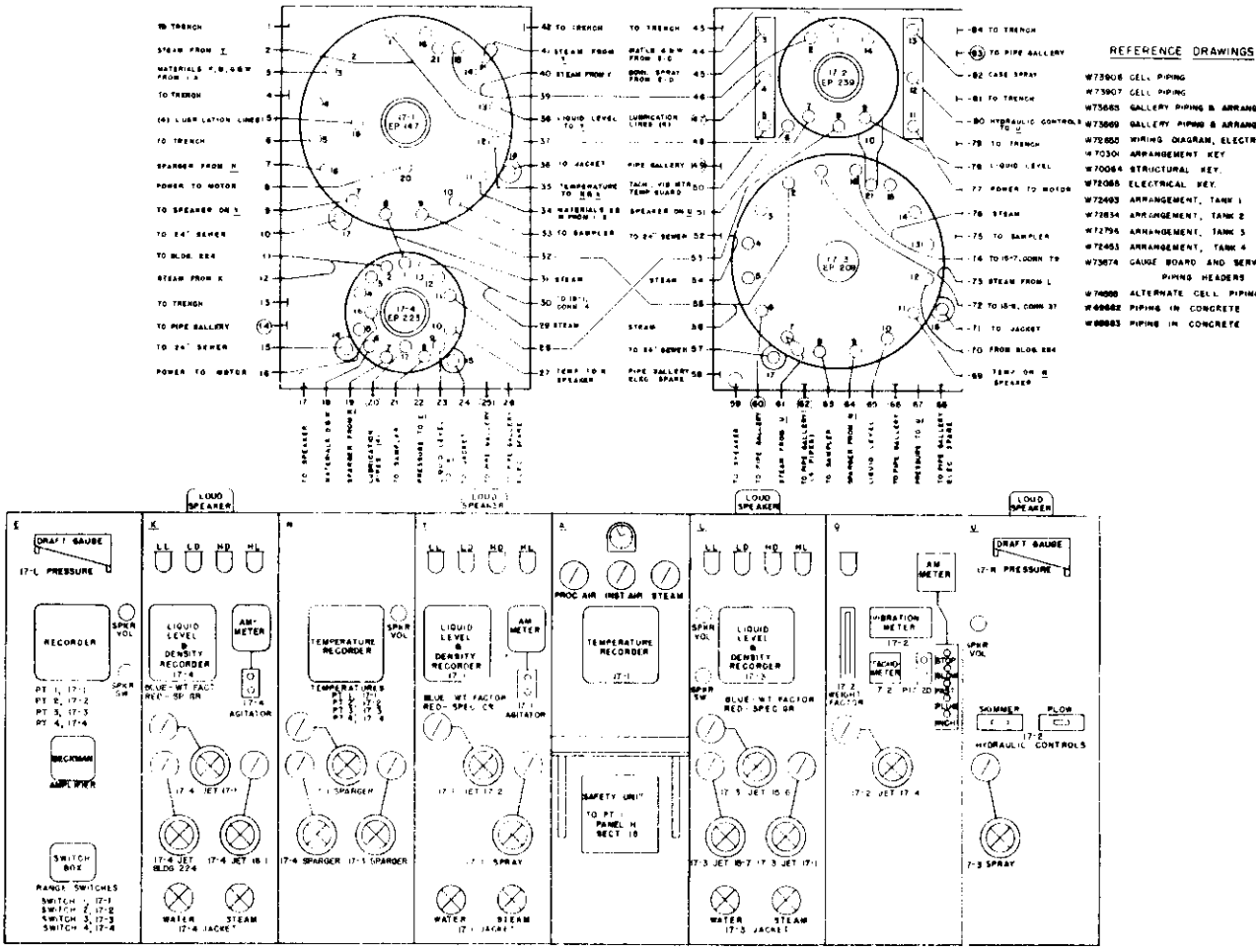

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FIGURE 11

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 18

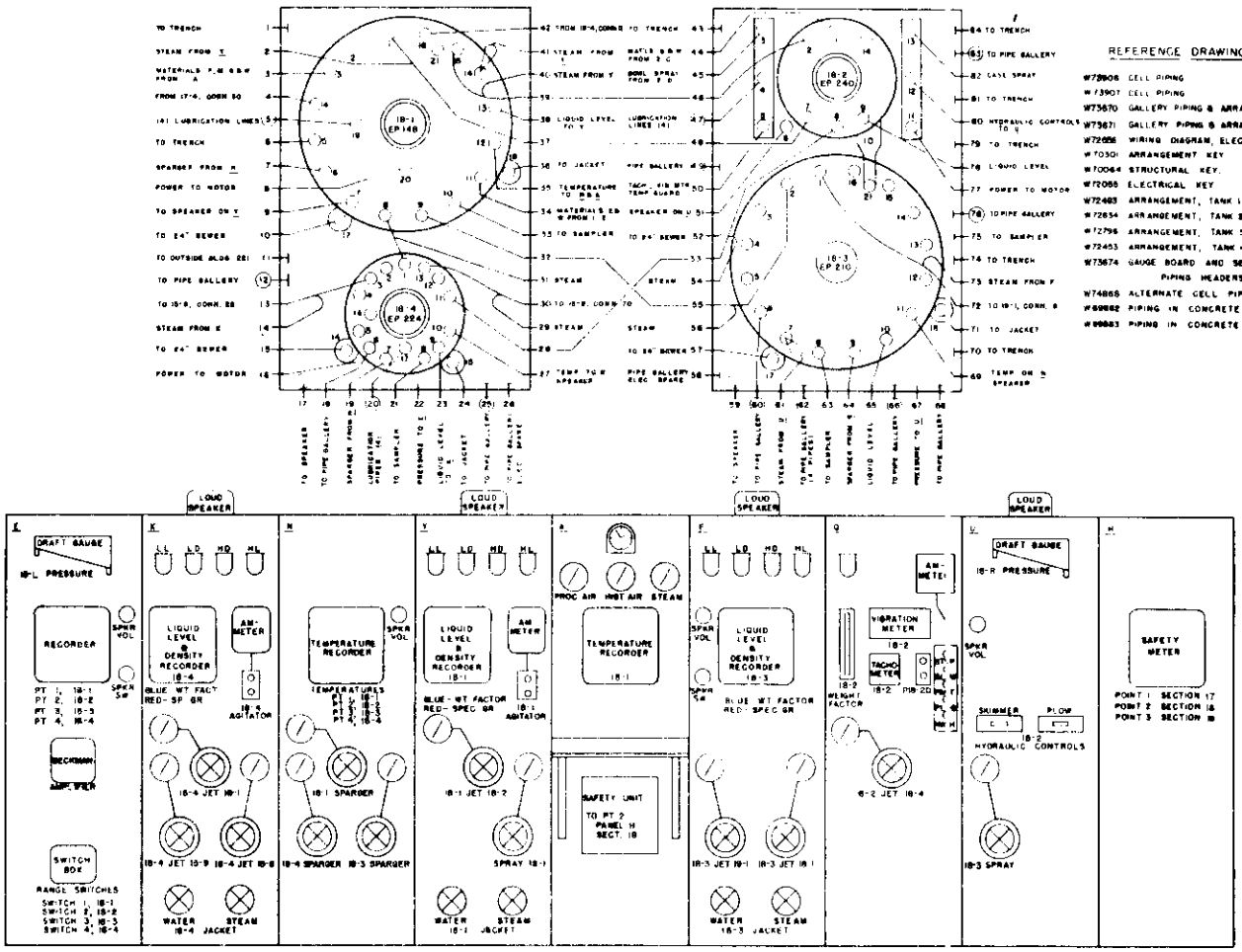

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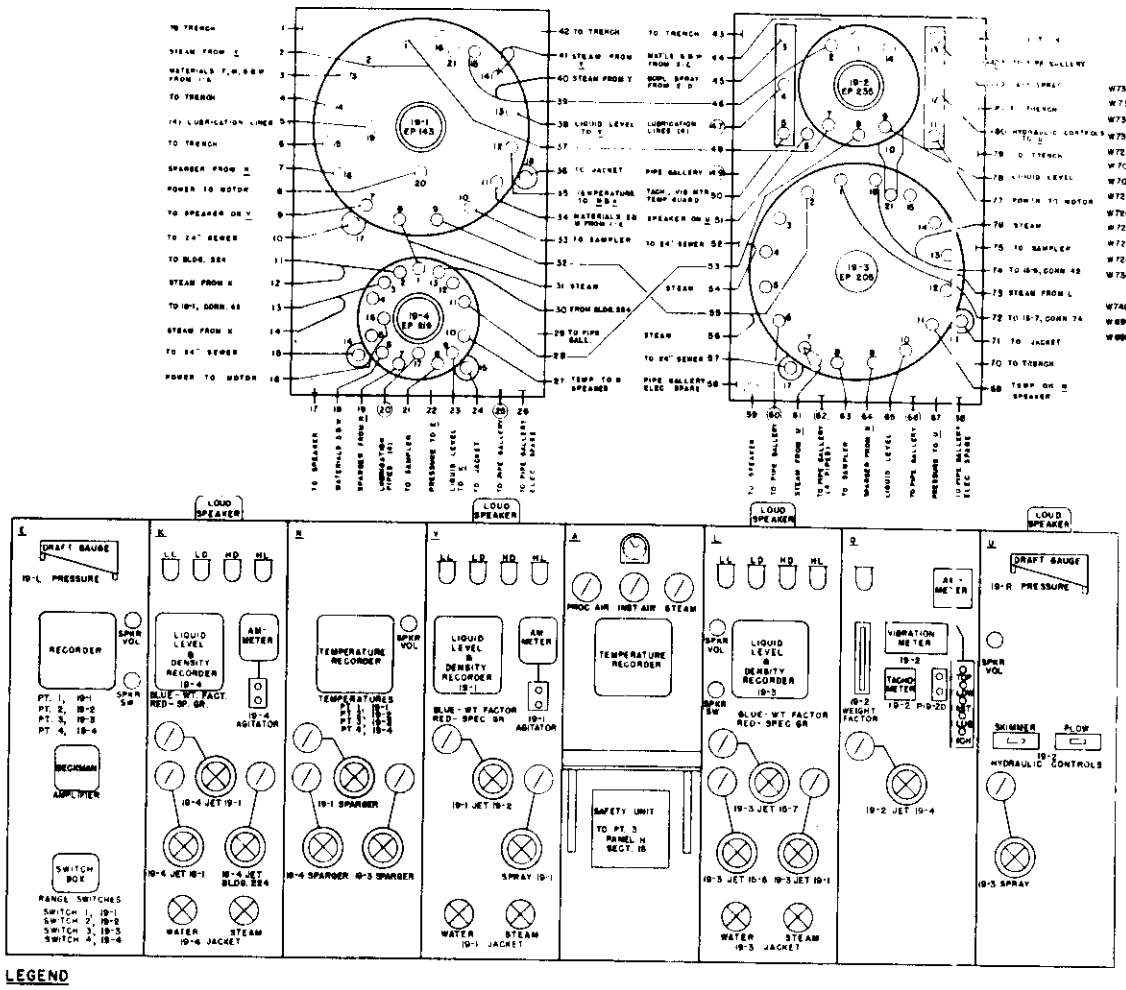
FIGURE 12

DECLASSIFIED

CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 19



REFERENCE DRAWINGS

W73906	CELL PIPING
W73907	CELL PIPING
W73672	GALLERY PIPING & ARRANGEMENT
W73673	GALLERY PIPING & ARRANGEMENT
W72656	WIRING DIAGRAM, ELECTRICAL
W70501	ARRANGEMENT KEY
W70084	STRUCTURAL KEY
W72065	ELECTRICAL KEY
W72493	ARRANGEMENT, TANK 1
W72854	ARRANGEMENT, TANK 2
W72796	ARRANGEMENT, TANK 3
W72453	ARRANGEMENT, TANK 4
W73674	GAUGE BOARD AND SERVICE PIPING HEADERS
W74868	ALTERNATE CELL PIPING
W69882	PIPING IN CONCRETE
W69883	PIPING IN CONCRETE

LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WALL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM

[redacted]

is received in the precipitators from the catch tank of the preceding section (by-product precipitation). After precipitation and separation of the product, the catch tank effluent is sent through one of two lines to waste disposal in Section 15.

All connections to the Concentration Building are in Sections 17 and 19. The regularly used line is from Section 17 solution tank. A spare from Section 19 is used if a second or third decontamination cycle is run in this section. If a batch delivered to the Concentration Building is found to be insufficiently decontaminated, it is sent back in a return line to Section 19 solution tank. Here it is oxidized and jetted to Section 18 precipitator for an additional decontamination cycle. A second line from the Concentration Building enters the catch tank in Section 17. This is for the return of active wastes which are jetted to Section 15 for disposal.

OPERATIONS

First Decontamination Cycle

Oxidation

To start the first decontamination cycle, the 3,350 pounds (305 gallons) of product solution jetted from Section 8 to crude product storage is transferred to one of the solution tanks in Section 12 R by way of the 12-6 Crude Product Storage Tank in Section 12 L. Although this transfer is through the 12-6 Crude Product Storage Tank, more than one batch of crude product solution is not normally held in storage and the identity of a single extraction charge is not lost. The concentration of nitric acid is brought down to approximately 34% (6.5 N) by diluting with 900 pounds (108 gallons) of water. For successful oxidation, the acid concentration should be close to the specified figure.

A 10% sodium bismuthate slurry (16 pounds of $NaBiO_3$ in 114 pounds of water) is made up in a 20-gallon stainless steel dolly tank in the 271 Building chemical preparation room and delivered to Section 12 when needed. The sodium bismuthate slurry is added to the acid cake solution and followed by 55 pounds of 10% sodium dichromate solution which is added from the "D" solution header by way of Weigh Tank 12-7C. The solution is heated to 45 °C and agitated for 1 hour at this temperature to complete the oxidation. The sodium dichromate serves as a holding oxidant and stabilizes the oxidized product. Without the dichromate, the product may be partially reduced if it is necessary to let the solution stand for a few hours in stainless steel equipment. The reduced product in that case is lost in the by-product precipitate.

On completion of the oxidation the solution is cooled to 35 °C and transferred to Section 13.

By-Product Precipitation

The 13,500 pounds (1620 gallons) of dilution water for reducing the nitric

acid concentration of the oxidized cake solution to 8% (1.4 N) is added to the precipitator in Section 13 and heated to 40 °C during the oxidation period. When oxidation is complete, the oxidized solution (4800 pounds or 470 gallons) is transferred from 12-7 to 13-1 after which 140 pounds of 24% bismuth subnitrate solution are added to the precipitator from Chemical Header R by way of Weigh Tank 13-1E and the solution heated to 75 °C. The bismuth phosphate precipitate is partially formed during dilution of the oxidized solution. This precipitate partially carries the phosphate insoluble fission elements, but leaves the oxidized product in solution. After digesting for 1 hour at 75 °C, the solution is cooled to 35 °C and precipitation is completed by the coprecipitation of the cerium and zirconium scavengers at 35 °C. These remove more completely the phosphate insoluble fission elements which otherwise remain in solution and contaminate the subsequent product precipitate to an important extent. Zirconium and columbium are the elements of this type for which the scavengers are specific.

Just prior to the scavenger precipitation, an additional 25 lbs. of 10% sodium dichromate is added to insure maintaining the product in the oxidized state. Cerium-zirconium scavenger solution, prepared in the 271 Building chemical preparation room by adding 4.5 pounds of ceric salt ($(NH_4)_2Ce(NO_3)_6$) to 70 pounds of 10% zirconium carbonate gel solution (1.5 ± 0.05% Zr^{+4}) in a 20-gallon stainless steel dolly, is added to the precipitator and agitated for 10 minutes at 35 °C. The precipitation is then completed by the rapid addition of 267 pounds (20 gallons) of 73.5% phosphoric acid, the solution agitated for 1 hour at 35 °C, cooled to 30 °C and centrifuged.

The by-product precipitation itself is simple and is not critical. Thorough agitation during all stages is important, but the times, quantities, and temperatures may be varied to a considerable extent.

Centrifuging of By-Product Precipitate

The centrifuge skimmer control is set, the centrifuge is brought up to 870 rev./min. and the slurry is jetted to the bowl using the "A" jet at the rate of 110 pounds/minute. This slower rate is used to give a larger (5-min.) retention time in the centrifuge for the more difficultly centrifugable precipitate. When the transfer stops, indicated by the leveling off of the catch tank weight factor trace, and with the 1 to 2 Tank jet running, approximately 350 pounds of effluent are jetted back to the precipitator from the catch tank to clean out settled precipitate. This recirculation is repeated twice.

The first water wash of 500 pounds (60 gallons) is added to the precipitator and circulated through the distributor for a few minutes to pick up as much as possible of the residual precipitate. It is then jetted to the bowl using the "A" jet. When the transfer stops, the precipitator is again jetted to the centrifuge bowl using the "B" jet. The use of the alternate jet arrangement (See Chapter V) is designed to reduce to a minimum the chance of leaving large heels in the precipitator due to premature cutout of the jets. After the wash has been centrifuged for 10 minutes, the bowl is skimmed to a 30-gallon heel. Special care must be exercised in skimming due to the possibility of skimming zirconium phosphate "fuzz", which is difficult to centrifuge, into the effluent. For the second wash, the bowl is brought to 10

rev./min. and the cake is cut off the walls by the addition of 85 pounds of water through the bowl sprays. The cake and wash are slurried three times by bringing the bowl to 140 rev./min. for 2 minutes and plugging to stop. After slurrying the cake and wash water are again separated by centrifuging at 870 rev./min. for 10 minutes and then skimmed to a 30-gallon heel.

The oxidized product solution and washes in the catch tank amount to approximately 20,400 pounds (2350 gallons). This is jetted to the precipitator in the next section for product separation. It should contain about 97-98% of the product and less than 1% of the fission activity present in the original metal solution.

Solution of the By-Product Cake

In spite of the recycling and water wash, some by-product precipitate adheres to the walls of the precipitator. If not removed, it continues to build up and form a deposit which may result in loss of both product and decontamination. To prevent this, the 2520 pounds of 60% nitric acid used for dissolving the by-product cake is added to the precipitator and agitated and circulated for 20 minutes.

The presence of the cerium and zirconium phosphates, resulting from the use of scavengers, makes necessary a special dissolution technique. In order to break up the cake so that it may be removed from the centrifuge bowl by jetting, the bismuth phosphate is dissolved in nitric acid and the cerium phosphate is solubilized with hydrogen peroxide solution. This leaves the insoluble zirconium phosphate in the form of a thin slurry. The acid is jetted to the centrifuge in 315-pound portions. The centrifuge is brought to 10 rev./min. and 24 pounds of 27% hydrogen peroxide is added directly to the centrifuge by way of the 13-2C Weigh Tank. As each portion of acid and hydrogen peroxide is received in the centrifuge it is slurried with the cake and transferred to the solution tank. In slurrying, the bowl is brought up to 140 rev./min., held for 2 minutes, and plugged to stop; this is repeated 5 times. Five separate shots of dissolving acid and peroxide are slurried through the bowl, and the remainder of the nitric acid in the precipitator is flushed through the bowl to the solution tank using the alternate jet procedure to guard against leaving a heel in the precipitator. On completion of the addition of the acid shots, the Beckman meter level is checked as a measure of the completeness of cake removal, and, if the Beckman reading has not returned to the initial background level, the remainder of the acid is slurried through the bowl in 315-pound portions.

The cake and acid are agitated in the solution tank for 1 hour below 40 °C to complete the dissolving and jetted to waste disposal in Section 15.

Reduction and Product Precipitation

The 20,400 pounds (2350 gallons) of oxidized product solution received in the Section 14 precipitator is reduced with ferrous ammonium sulfate, in the presence of ammonium silicofluoride. A ferrous ion concentration of 0.04M is obtained by the addition of 390 pounds of $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ in 20% solution. Besides reducing the product, the iron has a beneficial effect in

decreasing the amount of by-product carried, so that better decontamination is obtained. Excess iron, however, interferes with the carrying of product, resulting in a loss of yield. The reduction is completed by heating the solution to 75 °C.

A phosphoric strike is preferred for product precipitations because of better decontamination and yield, but is not entirely possible because some phosphoric acid is already present from the by-product precipitation. However, bismuth is added first, followed by a further amount of phosphoric acid.

The 216 pounds of 24% bismuth subnitrate in 20% nitric acid is added slowly over 1 hour and digested for an additional 30 minutes. The strike is completed by the addition of 1945 pounds of 73.5% phosphoric acid which is also added over a 1-hour period.

The complete strike is carried out at 75 °C with thorough agitation and after digestion for 2 hours, the slurry is cooled to 50 °C and centrifuged.

Centrifuging of Product Precipitate

The centrifuge skimmer controls are set, the centrifuge brought up to 870 rev./min. and the slurry jetted into the bowl at a rate of 140 pounds/minute using the "A" jet. On completion of the transfer three 360-pound portions of effluent are recycled to pick up the heel of precipitate which settles out when the liquid level drops below the bottom of the agitator blade.

The first water wash of 500 pounds (60 gallons) is added to the precipitator and circulated through the distributor to wash down the walls. The alternate jet procedure is used as described previously. The partially washed slurry is centrifuged and the centrifuge skimmed to a 10-gallon heel.

The centrifuge is brought to 10 rev./min. and 250 pounds of water is added through the bowl sprays. This cuts most of the cake from the wall and helps to mix the wash and holdup liquid. The mixture is slurried three times by bringing the bowl up to 140 rev./min., holding for 2 minutes, and plugging to stop. This series of operations mixes the cake and wash and avoids any tendency of the water to pass over the cake without flushing out impurities. Washing removes soluble fission elements and improves decontamination. After slurrying, the centrifuge is brought to 870 rev./min., held for 5 minutes, and skimmed. The procedure is repeated with a second 250-pound wash to conclude the washing operation.

The combined washings and effluent in the catch tank amount to 29,630 pounds (3290 gallons). Less than 0.5% of the product should be lost in this effluent. The solution is sent to Section 15 for waste disposal.

Product Cake Solution

The cake is dissolved in 2020 pounds (177 gallons) of 60% nitric acid. In spite of the recycling and washing, some bismuth phosphate remains on the precipitator walls. This precipitate holdup can cause 5-10% losses in product

due to the fact that the cake continues to build up more or less indefinitely unless steps are taken to remove it. The dissolving acid is therefore added to the precipitator, and circulated through the distributor for 20 minutes.

The acid is jetted to the centrifuge in portions of 330 pounds (30 gallons) using the "A" jet. Each portion is slurried three times by bringing the bowl up to 140 rev./min., holding 2 minutes, and plugging to stop. The slurry is then jetted to the solution tank. Four shots of dissolving acid are adequate to remove the cake; the remainder of the acid is flushed through the centrifuge. After all of the acid is transferred to the solution tank, the tank is agitated for 1 hour to complete solution of the cake.

The cake solution should contain 97-98% of the product and less than 1% of the fission activity present at the start of the cycle. Overall yields and decontamination factor to this point are about 95-98% and 3000, respectively.

Product Cake Oxidation

The product cake is oxidized in Section 14 solution tank before being transferred to Section 16 for the second decontamination cycle. The procedure is identical with the oxidation of crude product solution described earlier in this chapter, except for the lesser quantities involved.

Second Decontamination Cycle

The second cycle is run in Sections 16 and 17. The process differs from the first cycle operation in that the scavengers are not used in the by-product precipitation, the volumes are lower and the final product cake oxidation is carried out in the Concentration Building. The product solution, with a yield of around 96% and decontaminated by a factor of 10^5, is jetted to the 224 Building for concentration.

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Report CN 1879	Progress Report on First Nine Months Operation of Separation Plant - Pilot Plant.
File No. 3-2825	(SE-PC-57) Decomposition of $NaBiO_3$ and Oxidation of Product by $NaBiO_3$ and by Pb_3O_4.
File No. 7009	Index of Research Memoranda and Reports Issued by the Members of Site X Chemistry Section C-III during the Site W Process Decontamination Study Program 6/1/44 to 1/45.
File No. 3-2209	(SE-PC-18) Identification of the Radioactive Fission Elements Associated with Product at Various Stages Throughout the Separation Process.
File No. 3-1493	Decontamination of Fission Elements in the Separation Process.
File No. 3 - 2767	(SE-PC-63) Decontamination Obtained by the HEW Process if Scavengers are omitted.
Report CN 2027	Nomographs for the Solubility of $BiPO_4$ in HNO_3, HNO_3-H_3PO_4 and HNO_3-Bi Solutions.
Report CN 1863	The Solubility of Bismuth Phosphate in HNO_3
File No. 3 - 2531	(SE-PC-27) Rate of Solution and Solubility of Plant Bismuth Phosphate Cakes in Nitric Acid.
Report CN 2621	Rate of Solution of Alpha and Beta $BiPO_4$ in HNO_3 Solutions.
Report CN 2561	Physical Properties of the X-Plant Product By-Product and Scavenger Precipitates.
Report CN 2574	Studies on the Preparation of Beta $BiPO_4$ and its Carrying and Decontamination Properties.
Report CN 2050	Bismuth Phosphate Precipitation Studies.
File No. 3 - 2542	(SE-PC-39) Literature Survey of Reducing Agents for Plutonium for Possible Plant Use.

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File No. 3 - 2511	(SE-PC-39) Decrease in Reduction Time Prior to Bismuth Phosphate Product Precipitations.
Report CN 2688	Chemical Research - Separation Process for Plutonium, Jan. 1 to Feb. 15, 1945.
Report CC 1394	Chemical Research - Radiochemistry, Feb. 1944.
File No. 3 - 2371	(SE-PC-28) The Use of Ammonium Fluosilicate Analogs for Improving Decontamination in the Product Precipitation Steps.
File No. 3 - 2873	(SE-PC-55) The Effect of Ammonium Fluosilicate on Decontamination in the Second Cycle Product Precipitation Step.
File No. 3 - 2529	(SE-PC-25) Bowl Metathesis of Bismuth Phosphate.
File No. 3 - 2708	(SE-PC-53) Method for Obtaining a Decontamination Factor of 10^5 at the End of One Decontamination Cycle.
File No. 3 - 1951	(SE-PC-12) Recovery of Product from 1st Cycle Ce-Zr Scavenger By-Product Precipitates.
File No. 3 - 2824	(SE-PC-61) Recovery of Product from $BiPO_4$ By-Product Precipitate.

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APPENDIX

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FLOW SHEET

The process flow sheet, based on carrying an extraction charge from 1.5 tons of metal through the two decontamination cycles, is presented on the following pages.

OPERATING LOG FORMS

The operating log forms presented at the end of this appendix constitute the detailed operating instructions by which the operators perform the various steps in the decontamination cycles.

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APPENDIX

DECONTAMINATION

PREPARATION FOR FIRST BY-PRODUCT PRECIPITATION

Section 12

Oxidation

1) Transfer 3350 lbs. (305 gal.) of extraction cake solution from 8-4 to 12-6 to 12-7.

2) Dilute to 34% HNO_3 by adding 900 lbs. (108 gal.) of water.*

3) Add 130 lbs. (14 gal.) of 10% $NaBiO_3$ slurry.

4) Add 55 lbs. (6 gal.) of 10% $Na_2Cr_2O_7$ solution.

5) Agitate for 1 hour at 45 °C.

6) Cool to 35 °C.

7) Jet to precipitator 13-1.

Solution Composition (in 12-7)

	lbs.	%	M
HNO_3	1640	34.2	6.6
$BiPO_4$	88	1.8	--
$NaBiO_3$	13	0.3	0.012
$Na_2Cr_2O_7$	5.5	0.1	0.003
H_2O	2053.5	42.6	--
Total	4800	(470 gal.)	

* In addition to dilution obtained by jetting.

A. Precipitation

1) Add 13,700 lbs. (1650 gal.) of water to 13-1, start agitator and heat to 40 °C.

2) Transfer 4800 lbs. (470 gal.) of oxidized solution from 12-7 to 13-1.

3) Add 140 lbs. (11 gal.) of 24% bismuth solution
24% $BiONO_3$
19% HNO_3

4) Heat to 75 °C and digest for 1 hour.

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5) Cool to 35 °C.

6) Add 25 lbs. (2.8 gal.) of 10% sodium dichromate.

7) Add 74 lbs. (8 gal.) of Ce-Zr solution
3.6% $ZrO(NO_3)_2$
5.6% $(NH_4)_2Ce(NO_3)_6$
6.0% HNO_3

8) Agitate 10 minutes at 35 °C.

9) Add rapidly 267 lbs. of H_3PO_4 solution
73.5% H_3PO_4
1.2% HNO_3

10) Digest at 35 °C for 1 hour; cool to 30 °C.

11) Jet to centrifuge 13-2.

Slurry Composition (in 13-1)

	lbs.	%	M
HNO_3	1680	8.45	1.42
$BiPO_4$	138	0.69	--
$NaBiO_3$*	nil	--	--
H_3PO_4	183	0.92	0.10
$Na_2Cr_2O_7$	8	0.04	0.002
$Ce_3(PO_4)_4$	1.9	0.01	--
$Zr_3(PO_4)_4$	2.3	0.01	--
NH_4NO_3	1.3	0.01	--
$NaNO_3$	4.1	0.02	--
H_2O	17862.0	89.8	--
Total	19880.6	(2250 gals.)	

* Decomposed and bismuth precipitated as $BiPO_4$

B. Centrifuging

1) Centrifuge at 870 RPM, 110 lbs./min.

2) Recycle 1000 lbs. of effluent from catch tank 13-3 to 13-1 to 13-2.

3) Add 500 lbs. (60 gal.) of water to 13-1.

4) Recirculate through spray for 3 minutes.

5) Jet wash water from 13-1 to 13-2

6) After 10 minutes, skim 13-2 to 30 gallon heel.

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C. Cake Wash

1) Add 85 lbs. of water from 13-1 to 13-2 as a displacement wash.

2) Skim to 30 gallon heel and plug 13-2 to stop.

3) Jet effluent from 13-3 to 14-1.

Solution Composition (in 13-3)

	lbs.	%	M
HNO_3	1680	8.24	1.36
H_3PO_4	183	0.90	0.095
$Na_2Cr_2O_7$	8	0.04	0.002
NH_4NO_3	1	0.01	--
$NaNO_3$	4	0.02	--
H_2O	18524	90.8	--
Total	20400	(2350 gal.)	

Cake Composition (in 13-2)

	lbs.	%
$BiPO_4$	138	40.8
$Ce_3(PO_4)_4$	1.94	0.6
$Zr_3(PO_4)_4$	2.33	0.7
H_2O	196	57.9
Total	338	(30 gal.)

D. Cake Removal

1) Jet slurry from 13-2 to solution tank 13-4.

2) Add 2520 lbs. (220 gal.) of 60% HNO_3 solution to 13-1.

3) Recirculate through spray for 20 minutes with agitator running.

4) Jet 315 lbs. (28 gal.) from 13-1 to 13-2.

5) Add 24 lbs. (2 gal.) of 27.5% H_2O_2 to 13-2 and slurry cake.

6) Jet this portion from 13-2 to 13-4.

7) Start agitator in 13-4.

8) Repeat (4), (5), and (6) five more times, then jet remaining acid from 13-1 to 13-2 to 13-4.

9) Jet from 13-4 to waste neutralizer 15-8.

Slurry Composition (in 13-4)

	lbs.	%
$BiPO_4$	138	4.0
$CePO_4$	2	0.06
$Zr_3(PO_4)_4$	2.3	0.06
HNO_3	1510	43.7
H_2O_2*	nil	--
H_3PO_4	1	0.03
H_2O	1797	52.1
Total	3450 (310 gal.)	

* Decomposed to water and O_2.

FIRST PRODUCT PRECIPITATION

Section 14

A. Reduction and Precipitation

1) Transfer 20,400 lbs. (2350 gal.) of oxidized effluent from 13-3 into 14-1.

2) Add 1575 lbs. (175 gal.) of 14% ammonium silicofluoride solution.

3) Add 1950 lbs. (200 gal.) of 20% $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$ solution.

4) Heat to 75 °C.

5) Add 216 lbs. (17 gal.) of Bi solution

24% $BiONO_3$
19% HNO_3
Rate of addition: 3.5 lbs./min.

6) Agitate for 30 minutes at 75 °C.

7) Add 1945 lbs. (147 gal.) of H_3PO_4 solution:

73.5% H_3PO_4
1.2% HNO_3
Rate of addition: 35 lbs./min.

8) Agitate for 2 hours at 75 °C.

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9) Cool to 50 °C before jetting to centrifuge 14-2.

Slurry Composition (in 14-1)

	lbs.	%	M
HNO_3	1681	6.15	1.03
$FeSO_4$	nil	--	--
$Fe_2(SO_4)_3$	199	0.73	0.019
H_3PO_4	1595	5.87	0.63
$BiPO_4$	55	0.20	--
$Cr(NO_3)_3$	15	0.06	--
$(NH_4)_2SO_4$	65	0.24	--
$(NH_4)_2SiF_6$	220	0.81	0.048
$NaNO_3$	5	0.02	--
NH_4NO_3	80	0.29	--
H_2O	23,285	85.6	
Total	27,200	(3080 gal.)	

B. Centrifuging

1) Centrifuge at 870 RPM, 140 lbs./min.

2) Recycle 1000 lbs. of effluent from catch tank 14-3 to 14-1 to 14-2.

3) Add 500 lbs. (60 gal.) of water to 14-1.

4) Recirculate through the spray for 3 minutes.

5) Jet wash water from 14-1 to 14-2.

6) After 5 minutes, skim 14-2 to 10 gallon heel.

C. Cake Washing

1) Add 250 lbs. (30 gal.) of water to 14-2 through the sprays.

2) Wash cake and skim to 10 gal. heel.

3) Repeat (1) and (2).

4) Jet waste effluent and washings from catch tank 14-3 to waste receiver 15-7.

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Waste Effluent Composition (in 14-3)

	lbs.	%
HNO_3	1681	5.70
H_3PO_4	1595	5.38
$Fe_2(SO_4)_3$	199	0.67
$Cr(NO_3)_3$	15	0.05
$(NH_4)_2SO_4$	65	0.22
$(NH_4)_2SiF_6$	220	0.75
$NaNO_3$	5	0.02
NH_4NO_3	80	0.27
H_2O	25,770	
Total	29,630	(3290 gal.)

Washed Cake Composition (in 14-2)

	lbs.	%
$BiPO_4$	55	50.0
H_2O	55	50.0
Total	110	(10 gal.)

D. Product Cake Dissolving and Oxidation

1) Add 2020 lbs. (177 gal.) of 60% HNO_3 solution to 14-1.

2) Recirculate through the spray for 20 minutes.

3) Jet 330 lbs. (30 gal.) from 14-1 to 14-2 and slurry cake.

4) Jet from 14-2 to solution tank 14-4.

5) Start agitator in 14-4.

6) Repeat (3) and (4) three more times.

7) Jet remaining acid from 14-1 to 14-2 to 14-4.

8) Agitate for at least 1 hour after all HNO_3 is in 14-4.

9) Add 1000 lbs. (120 gal.) of water to 14-4.

10) Add 130 lbs. of 10% $NaBiO_3$ slurry.

11) Add 55 lbs. of 10% $Na_2Cr_2O_7$ solution.

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12) Agitate for 1 hour at 45 °C.

13) Cool to 35 °C.

14) Jet to precipitator 16-1.

Solution Composition (in 14-4)

	lbs.	%	M
$BiPO_4$	55	1.45	--
HNO_3	1212	32.18	5.25
$NaBiO_3$	13	.33	.015
$Na_2Cr_2O_7$	5.5	.14	.005
H_2O	2499.5	65.90	--
Total	3785	(370 gal.)	

DECONTAMINATION

SECOND BY-PRODUCT PRECIPITATION

Section 16

A. Precipitation

1) Add 12,060 lbs. (1448 gal.) of water to 16-1, start agitator and heat to 40 °C.

2) Transfer 3785 lbs. (370 gal.) of oxidized solution from 14-4 into 16-1.

3) Add rapidly 208 lbs. of 75% phosphoric acid.
73.5% H_3PO_4
1.2% HNO_3

4) Heat 16-1 to 75 °C and digest for 1 hour.

5) Cool to 50 °C and jet to centrifuge 16-2.

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Slurry Composition (in 16-1)

	lbs.	%
HNO_3	1212	7.37
$BiPO_4$	69	0.42
H_3PO_4	201	1.23
$NaBiO_3$*	nil	--
$Na_2Cr_2O_7$	5.5	0.03
$NaNO_3$	4	0.02
H_2O	14,968.5	90.93
Total	16,460	(1950 gal.)

*Decomposed and bismuth precipitated as $BiPO_4$

B. Centrifugation

1) Centrifuge at 870 RPM, 140 lbs./min.

2) Recycle 1000 lbs. of effluent from 16-3 catch tank to 16-1 to 16-2.

3) Add 500 lbs. (60 gal.) of water to 16-1.

4) Recirculate through spray for 3 minutes.

5) Jet wash water from 16-1 to 16-2.

6) After 5 minutes, skim 16-2 to 10 gallon heel.

C. Cake Washing

1) Add 250 lbs. (30 gal.) of water to 16-2 through the sprays.

2) Wash cake and skim to 10 gallon heel.

3) Repeat (1) and (2).

4) Jet effluent from 16-3 to 17-1.

Solution Composition (in 16-3)

	lbs.	%	M
HNO_3	1212	6.82	1.10
H_3PO_4	201	1.13	0.12
$Na_2Cr_2O_7$	5.5	.03	0.002
$NaNO_3$	4.0	.02	--
H_2O	16,377.5	92.0	--
Total	17,800	(2100 gal.)	

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Cake Composition (in 16-2)

	lbs.	%
$BiPO_4$	69	59.0
H_2O	48	41.0
Total	117	(10 gal.)

D. Cake Removal

1) Jet slurry from 16-2 to solution tank 16-4.

2) Add 1100 lbs. (96.4 gal.) of 60% HNO_3 solution to 16-1.

3) Recirculate through spray for 20 min.

4) Jet 330 lbs. of acid from 16-1 to 16-2 and slurry cake.

5) Jet acid from 16-2 to 16-4.

6) Start 16-4 agitator.

7) Repeat (4) and (5) three more times, then jet any remaining acid from 16-1 to 16-2 to 16-4.

8) Jet from 16-4 to waste neutralizer 15-9.

Cake Solution Composition (in 16-4)

	lbs.	%
$BiPO_4$	69	3.9
HNO_3	660	36.9
H_2O	1061	59.2
Total	1790	(170 gal.)

SECOND PRODUCT PRECIPITATION

Section 17

A. Reduction and Precipitation

1) Transfer 17,800 lbs. (2100 gal.) of oxidized effluent from 16-3 into 17-1.

2) Add 1160 lbs. (127.5 gal.) of 14% ammonium silico fluoride solution.

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3) Add 1575 lbs. (168 gal.) of 20% $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ solution.

4) Heat to 75 °C.

5) Add 168 lbs. (13 gal.) of bismuth solution.
24% $BiONO_3$
19% HNO_3
Rate of addition: 3 lbs./min.

6) Agitate for 30 minutes at 75 °C.

7) Add 1510 lbs. (115 gal.) of H_3PO_4 solution (F):
73.5% H_3PO_4
1.2% HNO_3
Rate of addition: 25 lbs./min.

8) Agitate for 2 hours at 75 °C.

9) Cool to 50 °C before jetting to centrifuge 16-2.

Slurry Composition (in 17-1)

	lbs.	%	M
HNO_3	1262	5.46	0.92
$FeSO_4$	nil	--	--
$Fe_2(SO_4)_3$	161	0.70	0.018
$Cr(NO_3)_3$	15	0.06	
H_3PO_4	1311	5.67	0.61
$BiPO_4$	43	0.19	--
NH_4NO_3	64	0.28	--
$(NH_4)_2SiF_6$	163	0.71	0.042
$NaNO_3$	5	0.02	--
$(NH_4)_2SO_4$	53	0.23	--
H_2O	20,003	86.67	--
Total	23,080	(2620 gal.)	

B. Centrifuging

1) Centrifuge at 870 RPM, 140 lbs./min.

2) Recycle 1000 lbs. of effluent from catch tank 17-3 to 17-1 to 17-2.

3) Add 500 lbs. (60 gal.) of water to 17-1.

4) Recirculate through the spray for 3 minutes.

5) Jet wash water from 17-1 to 17-2.

6) After 5 minutes, skim 17-2 to 10 gallon heel.

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DECLASSIFIED

C. Cake Washing

1) Add 250 lbs. (30 gal.) of water to 17-2 through the sprays.

2) Wash cake and skim to 10 gal. heel.

3) Repeat (1) and (2).

4) Jet waste effluent and washings from catch tank 17-3 to waste receiver 15-6.

Waste Effluent Composition (in 17-3)

	lbs.	%
HNO_3	1262	5.12
H_3PO_4	1311	5.33
$Fe_2(SO_4)_3$	161	0.65
$Cr(NO_3)_3$	15	0.06
$(NH_4)_2SO_4$	53	0.22
$(NH_4)_2SiF_6$	163	0.66
$NaNO_3$	5	0.02
NH_4NO_3	64	0.26
H_2O	21,616	87.68
Total	24,650	(2800 gal.)

Washed Cake Composition (in 17-2)

	lbs.	%
$BiPO_4$	43	41.0
H_2O	62	59.0
Total	105	(10 gal.)

D. Product Cake Dissolving

1) Add 1570 lbs. (138 gal.) of 60% HNO_3 solution to 17-1.

2) Recirculate through the spray for 20 minutes.

3) Jet 330 lbs. (30 gal.) from 17-1 to 17-2 and slurry cake.

4) Jet from 17-2 to solution tank 17-4.

5) Start agitator in 17-4.

6) Repeat (3), (4), and (5) three more times.

7) Jet remaining acid from 17-1 to 17-2 to 17-4.

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8) Agitate for at least 1 hour after all HNO_3 is in 17-4.

9) Jet solution from 17-4 to receiving tank C-4 in Building 224.

Solution Composition (in 17-4)

	lbs.	%
$BiPO_4$	43	2.0
HNO_3	940	44.7
H_2O	1117	55.3
Total	2100 (195 gal.)	

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DECONTAMINATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

I - CRUDE PRODUCT STORAGE

1. Beckman reading on 12 6. Meter _____ Factor _____

2. Turn on 12-6 cooling water and air sparger. Leave cooling water on until 12 6 is empty or at 25°C. 12 6 cooling water on _____
12 6 air sparger on _____

3. Receive crude product cake solution in 12 6 from 8 4. Time start _____ Time end _____

4. Beckman reading on 12 6 Meter _____ Factor _____

5. Approximately 3 hours before oxidized solution is desired in 13-1, notify dispatcher of impending transfer. Time called _____

6. When the dispatcher approves, proceed with oxidation, (see page 5.1, step 1). Time approved _____

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DECONTAMINATION OPERATION

Date _____
Run No. _____

DECLASSIFIED

I FIRST DECONTAMINATION (PRODUCT) OXIDATION

1. Jet 12-6 to 12-7. When the transfer stops, put jet on air for 10 min. and again jet 12-6 to 12-7.
   Time start _____ Time end _____
   12-6 Wt.Ftr.Start _____ Lbs.
   12-7 Wt.Ftr.End _____ Lbs.
   Lbs. jetted from 12-6 _____
   Lbs. received in 12-7 _____

2. Turn off 12-6 air sparger.
   12-6 air sparger off _____

3. Beckman reading on 12-6.
   Meter _____ Factor _____

   NOTE: If the reading is greater than _____ on the 10 _____ scale, notify supervisor and again jet 12-6 to 12-7.

4. Start 12-7 agitator.
   12-7 Agitator on _____

5. Add 900 lbs. of water to 12-7 via 12-7B. (TW in 12-7 approx. 4400 lbs.)
   Lbs. added _____ Time added _____

6. Adjust 12-7 temperature to 45°C. and maintain at this temperature.
   Time 45°C _____

7. Add 130 lbs. of 10% Sodium Bismuth Slurry (Dolly) to 12-7 via funnel F-12-7C.
   Lbs. added _____ Time added _____

8. Flush dolly and funnel with 50 lbs. of process water.
   Lbs. added _____ Time added _____

9. Add 55 lbs. of 10% Sodium Di chromate solution (Line D) to 12-7 via 12-7C.
   Lbs. added _____ Time added _____

10. Flush 12-7C to 12-7 with 50 lbs. of process water.
    Lbs. added _____ Time added _____

11. Digest 12-7 at 45°C for one hour DO NOT EXCEED 45°C. During this operation proceed to page 5.2, steps 1-4 inc.
    Time start _____ Time end _____

12. Cool 12-7 to 35°C.
    Time start _____ Time end _____

13. Call dispatcher and obtain permission to jet 12-7 to 13-1. When the transfer stops, put the jet on air for 10 min. and again jet 12-7 to 13-1. (TW approx. 4700 lbs.)
    Time called _____
    Time start _____ Time end _____
    12-7 Wt.Ftr. _____
    Lbs. jetted to 13-1 _____

14. Shut off 12-7 agitator
    12-7 Agitator off _____

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DECONTAMINATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

II. FIRST DECONTAMINATION (BY PRODUCT) FIRST PRECIPITATION

1.	Beckman reading on 13-1	Meter _____ Factor _____
2.	Add 11,500 lbs. of water to 13 1 via 13-1A in three portions of 3,000 lbs. and one of 2,500 lbs.	1st Port. _____ Lbs. Time added ____ 2nd Port. _____ Lbs. Time added ____ 3rd Port. _____ Lbs. Time added ____ 4th Port _____ Lbs. Time added ____ 13-1 Wt. Ftr. _____ Lbs. _____
3.	Start 13-1 agitator	13-1 Agitator on _____
4.	Heat water in 13-1 to 75°C with steam on the sparger and jacket. Continue heating during the transfer and Bismuth addition which follow, and maintain at 75°C until step 8 is completed.	Time start _____ Time 75°C _____
5.	Receive oxidized solution from 12-7 (TW in 13-1 approx. 15,600 lbs.)	Time start _____ Time end _____
6.	Add 140 lbs. of 24% Bismuth solution (Line R) to 13-1 via 13-1E.	Lbs. added _____ Time added _____
7.	Flush 13-1E to 13-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____
8.	Digest 13-1 at 75°C for 1 hour	Time start _____ Time end _____
9.	Beckman reading on 13-1 during digestion.	Meter _____ Factor _____
10.	Cool to 50°C and proceed with step 11. Leave cooling water on. 13-1 temperature should reach 30°C by completion of step 19.	Time start _____ Time 50°C _____
11.	Call 271 Building and request them to prepare 74 lbs. of Co-Zr solution.	Time called _____
12.	Add 25 lbs. of 10% Sodium Dichromate solution (Line D) to 13-1 via 13-1E.	Lbs. added _____ Time added ____
13.	Flush 13-1E to 13-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____

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Date _____
Run No. _____

DECLASSIFIED

II - FIRST DECONTAMINATION (BY-PRODUCT) - FIRST PRECIPITATION (Cont'd.)

14. Add 74 lbs. of Ce-Zr solution (Dolly) to 13-1 via funnel F-13-1E.	Lbs. added _____ Time added _____
15. Flush dolly and funnel F 13-1E. to 13-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____
16. Agitate 13-1 for 10 minutes	Time start _____ Time end _____
17. Add 267 lbs. of 75% Phosphoric Acid (Line F) to 13-1 rapidly via 13-1E.	Lbs. added _____ Time added _____
18. Flush 13-1E to 13-1 with 50 lbs. of process water. (TW 19,310 lbs.)	Lbs. added _____ Time added _____
19. Digest for 1 hour after Phosphoric Acid addition.	Time start _____ Time end _____
20. If 13-1 temperature is not at 30°C, cool 13-1 to 30°C before centrifuging and leave cooling water on.	Time start _____ Time 30°C _____
21. Stop 13-1 agitator and read Wt. Ftr., Temp. and Sp.Gr. when Sp.Gr. meter becomes constant, then start 13-1 agitator.	13-1 agitator off _____ 13-1 Wt.Ftr. _____ Lbs. _____ 13-1 Sp.Gr. _____ Temp. _____ 13-1 agitator on _____

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Date _____

Run No. _____

DECLASSIFIED

III - FIRST DECONTAMINATION (BY-PRODUCT) - CENTRIFUGATION

1. Beckman reading on 13-2. Meter _____ Factor _____

2. Set centrifuge skimmer and bring 13-2 centrifuge to 870 RPM. Time 870 RPM _____

3. Turn on 13-3 air sparger and jacket cooling water. 13-3 air sparger on _____ 13-3 jacket water on _____

4. With 13-2 at 870 RPM and using the "A" jet, jet slurry from 13-1 to 13-2 at the rate of 110 lbs. per minute. Check the rate at intervals to insure proper rate. Shut off 13-1 agitator when the Wt. Ftr. reaches 0.2. Time start _____ Time end _____ Transfer rate _____ Time _____ _____ _____ Avg. _____ _____ 13-1 Agitator off _____

5. When the transfer from 13-1 to 13-2 stops, indicated by the leveling off of the 13-3 Wt. Ftr. trace (do not shut off jet), jet from 13-3 to 13-1 for 30 seconds and empty 13-1 again. Repeat this operation 2 times to flush out 13-1. Shut off jet on completion of the recycling. Time start _____ Time end _____

6. Call 271 Building and request peroxide solution (See page 5.6 Step 6).

7. Add 500 lbs. of water to 13-1 via 13-1E. Lbs. added _____ Time added _____

8. With cooling water on the jacket, circulate through the 13-1 spray (at 60 - 70 psi) for 3 minutes. Hold 13-1 temperature below 50°C. Time start _____ Time end _____

9. Using the "A" jet, jet 13-1 to 13-2. When transfer stops, indicated by the levelling off of the 13-3 Wt. Ftr. trace, shut off the "A" jet. Using the "B" jet, again jet 13-1 to 13-2. Time start _____ Time end _____

10. After 10 minutes skim 13-2 to a 30 gallon heel. PASSING THIS POINT WILL SKIM THE CAKE INTO THE EFFLUENT. Time start _____ Time end _____

11. Plug 13-2 to 10 RPM. Time 10 RPM _____

12. Add cake dissolving acid to 13-1 and start circulation (See page 5.6 steps 1-3 inc.) before starting cake washing.

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Date _____
Run No. _____

DECLASSIFIED

IV - FIRST DECONTAMINATION (BY-PRODUCT) - BY-PRODUCT CAKE WASHING

1.	With 13-2 at 10 RPM, add 85 lbs. of water through the bowl sprays at full pump pressure.	Lbs. added _____ Time added _____
2.	Bring 13-2 to 140 RPM, hold 2 min. then plug to stop. Repeat twice.	Time end _____
3.	Bring 13-2 to 870 RPM, hold 10 min. and skim to a 30 gallon heel. BE CAREFUL NOT TO SKIM PAST THIS POINT INTO THE EFFLUENT.	Time end _____
3A.	Beckman reading on 13-2	Meter _____ Factor _____
4.	Plug 13-2 to a stop	Time stop _____
5.	Call dispatcher and obtain permission to jet 13-3 to 14-1. See page 5.10 steps 1, 2 and 3. (TW 19,900 lbs. in 13-3)	Time called _____ 13-3 Wt Ftr. _____ Lbs. _____ Time start _____ Time end _____

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Date _____
Run No. _____

DECLASSIFIED

V - FIRST DECONTAMINATION (BY PRODUCT) - BY PRODUCT CAKE REMOVAL

1.	Add 2520 lbs. of 60% Nitric Acid (Line G) to 13-1 via 13-1A, and read 13-1 Wt. Ftr.	Lbs. added _____ Time added _____ 13-1 Wt. Ftr. _____ Lbs. _____
2.	Flush 13-1A to 13-1 with 50 lbs. or process water.	Lbs. added _____ Time added _____
3.	Turn on 13-1 agitator	13-1 agitator on _____
4.	With cooling water on the jacket, circulate through 13-1 spray (50-70 lbs. steam pressure) for 20 minutes. HOLD 13-1 TEMPERATURE BELOW 50°C.	13-1 jacket water on _____ Time start _____ Time end _____ Maximum temperature 13-1 _____
5.	Beckman reading on 13-4	Meter _____ Factor _____
6.	Jet slurry in 13-2 to 13-4	Time jetted _____
7.	Call 271 Building and request delivery of one carboy (115 ± 10 lbs.) of 27.5% Peroxide to 13-2C (Special Line).	Time called _____ Time received _____ Lbs. _____
8.	With 13-2 stopped and using the "A" jet, jet 305 lbs. of acid from 13-1 to 13-2.	13-2 Wt. Ftr. _____ Lbs. _____
9.	Bring 13-2 to 10 RPM and add 24 lbs. of 27.5% Peroxide to 13-2 from 13-2C	Lbs. added _____ Time added _____
10.	Bring 13-2 to 140 RPM, hold 2 minutes, then plug to stop. Repeat 4 more times. (Complete operation over 15 min. period.)	Time start _____ Time end _____
11.	Start 13-4 agitator.	13-4 agitator on _____
12.	With 13-2 stopped, jet 13-2 to 13-4.	Time jetted _____
13.	Turn cooling water on 13-4 and hold temperature below 35°C.	13-4 jacket water on _____
14.	Using the "A" jet, jet 315 lbs. of acid from 13-1 to 13-2.	13-2 Wt. Ftr. _____ Lbs. _____

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Date _____
Run No. _____

DECLASSIFIED

V. - FIRST DECONTAMINATION (BY-PRODUCT) BY-PRODUCT CAKE REMOVAL (Cont'd)

15.	Shut off 13-1 agitator	13-1 agitator off _____
16.	Bring 13-2 to 10 RPM and add 24 lbs. of 27.5% Peroxide to 13-2 from 13-2C.	Lbs. added _____ Time added _____
17.	Bring 13-2 to 140 RPM, hold 2 min., then plug to stop. Repeat 4 more times. (Complete operation over 15 min. period.)	Time start _____ Time end _____
18.	With 13-2 stopped, jet 13-2 to 13-4.	Time jetted _____
19.	Using the "A" jet, jet 315 lbs. of acid from 13-1 to 13-2.	13-2 Wt. Ftr. _____ Lbs. _____
20.	Bring 13-2 to 10 RPM and add 24 lbs. of 27.5% Peroxide to 13-2 from 13-2C.	Lbs. added _____ Time added _____
21.	Bring 13-2 to 140 RPM, hold 2 min., then plug to stop. Repeat 4 more times. (Complete operation over 15 minute period.)	Time start _____ Time end _____
22.	With 13-2 stopped, jet 13-2 to 13-4.	Time jetted _____
23.	Using the "A" jet, jet 315 lbs. of acid from 13-1 to 13-2.	13-2 Wt. Ftr. _____ Lbs. _____
24.	Bring 13-2 to 10 RPM and add 24 lbs. of 27.5% Peroxide to 13-2 from 13-2C.	Lbs. added _____ Time added _____
25.	Bring 13-2 to 140 RPM and hold 2 mins. then plug to stop. Repeat 4 more times. (Complete operation over 15 minute period.)	Time start _____ Time end _____
26.	With 13-2 stopped, jet 13-2 to 13-4.	Time jetted _____
27.	Using the "A" jet, jet 315 lbs. of acid from 13-1 to 13-2.	13-2 Wt. Ftr. _____ Lbs. _____

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Date _____
Run No. _____

DECLASSIFIED

V - FIRST DECONTAMINATION (BY-PRODUCT) - BY-PRODUCT CAKE REMOVAL (Cont'd.)

28. Bring 13-2 to 10 RPM and add the remaining 27.5% Peroxide in 13-2C to 13-2 (9 - 29 lbs.) — Lbs. added _____ Time added _____

29. Bring 13-2 to 140 RPM, hold 2 min., then plug to stop. Repeat 4 more times. (Complete operation over 15 minute period.) — Time start _____ Time ended _____

30. With 13-2 stopped, jet 13-2 to 13-4. — Time jetted _____

31. Using the "A" jet, jet 315 lbs. of acid from 13-1 to 13-2. — 13-2 Wt.Ftr. _____ Lbs. _____

32. Bring 13-2 to 140 RPM, hold 2 min., then plug to stop. Repeat 4 more times. (Complete operation over 15 min. period.) — Time start _____ Time end _____

33. With 13-2 stopped jet 13-2 to 13-4. — Time jetted _____

34. Beckman reading on 13-2 — Meter _____ Factor _____

Note: If the reading is greater than on the 10 scale, notify the supervisor and with his approval add the remaining acid in 330 lb. shots until the Beckman reaches this level or the acid is used up.

35. Bring 13-2 to 10 RPM and using the "A" jet, jet the remainder of acid from 13-1 to 13-2 to 13-4. Keep the bowl as empty as possible during this operation. When the 13-1 to 13-2 transfer stops, shut off the "A" jet and plug 13-2 to stop. — Time start _____ Time end _____ Time stop _____

36. Jet 13-2 to 13-4. — Time jetted _____

37. Using the "B" jet, again jet 13-1 to 13-2. When the transfer stops, shut off jet and read 13-2 manometer. — Time start _____ Time end _____ 13-2 Manometer _____ Lbs. _____

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Date _____
Run No. _____

DECLASSIFIED

V - FIRST DECONTAMINATION (BY-PRODUCT) - BY-PRODUCT CAKE REMOVAL (Cont'd)

38.	Jet 13-2 to 13-4.	Time jetted _____
39.	Agitate 13-4 for 30 minutes.	Time start _____ Time end _____
40.	Beckman reading on 13-2.	Meter _____ Factor _____
41.	Shut off 13-4 agitator and read Wt. Ftr., Temp. and Sp.Gr. when Sp. Gr. meter becomes constant, then start 13-4 agitator. (TW approx. 3500 lbs.)	13-4 agitator off _____ 13-4 Wt. Ftr. _____ Lbs. _____ 13-4 Sp.Gr. _____ Temp. _____ 13-4 Agitator on _____
42.	Beckman reading on 13-4.	Meter _____ Factor _____
43.	Call dispatcher, give charge data and request that sample 13-4 BP be taken.	Time called _____ Time sampled _____
44.	When 13-4 temperature reaches 25°C, shut off 13-4 jacket cooling water.	13-4 jacket water off _____
45.	Call dispatcher and obtain permission to jet 13-4 to 15-8. Shut off agitator in 13-4 when Wt. Ftr. reaches 0.2.	Time called _____ Time start _____ Time end _____ 13-4 agitator off _____

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[redacted]

Date _____
Run No. _____

DECLASSIFIED

VI - FIRST DECONTAMINATION (PRODUCT) - REDUCTION AND PRECIPITATION

1.	Beckman reading on 14-1.	Meter _____ Factor _____
2.	Receive effluent from 13-3 into 14-1. (TW in 14-1 approx. 19,300 lbs.) Shut off 13-3 air sparger on completion of transfer.	Time start _____ Time end _____ 14-1 Wt. Ftr. _____ Lbs. _____ 14-1 Sp.Gr. _____ Temp. _____ 13-3 air sparger off _____
3.	Beckman reading on 14-1.	Meter _____ Factor _____
4.	Start 14-1 agitator and adjust temperature to 40-50°C. Do not exceed 50°C until step 8 is completed.	14-1 agitator on _____
5.	Add 1575 lbs. of 14% Ammonium Silico Fluoride solution (Line H) in three portions of 525 lbs. each to 14-1 via 14-1E.	1st Port. _____ Lbs. Time added ___ 2nd Port. _____ Lbs. Time added ___ 3rd Port. _____ Lbs. Time added ___ Total lbs. added _____
6.	Flush 14-1E to 14-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____
7.	Add 1950 lbs. of 20% Iron solution (Line M) to 14-1 via 14-1A.	Lbs. added _____ Time added _____
8.	Flush 14-1A to 14-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____
9.	Heat to 75°C with steam on the sparger and jacket. Maintain temperature with jacket steam until step 15 is completed.	Time start _____ Time 75°C _____ Time end _____
10.	Add 216 lbs. of 24% Bismuth Nitrate solution (Line R) to 14-1 via 14-1E at the rate of 3.5 lbs. per minute.	Time start _____ Time end _____ Lbs. added _____
11.	Flush 14-1E to 14-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____
12.	Digest 14-1 for 30 minutes at 75°C.	Time start _____ Time end _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VI - FIRST DECONTAMINATION (PRODUCT) - REDUCTION AND PRECIPITATION

14. Add 1945 lbs. of 75% Phosphoric Acid (line F) to 14-1 via 14-1A at the rate of 35 lbs. per minute.
    Time start _____ Time end _____
    Lbs. added _____

15. Flush 14-1A to 14-1 with 50 lbs. of process water.
    Lbs. added _____ Time added _____

16. Digest 14-1 for 2 hrs. at 75°C.
    Time start _____ Time end _____

17. Start cooling water on 14-1.
    14-1 Jacket water on _____

18. Stop 14-1 agitator and read Wt. Ftr., Temp. and Sp.Gr. when Sp.Gr. meter becomes constant, then start 14-1 agitator.
    14-1 agitator off _____
    14-1 Wt. Ftr. _____ Lbs. _____
    14-1 Sp.Gr. _____ Temp. _____
    14-1 agitator on _____

19. Cool 14-1 to 50°C and proceed to page 5.12, step 1. Leave cooling water on until 14-1 is empty or at 25°C.
    Time 50°C _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VII - FIRST DECONTAMINATION (PRODUCT) - CENTRIFUGATION

1.	Beckman reading on 14-2.	Meter _____ Factor _____
2.	Supervisor's approval to start centrifugation.	Beckman limit _____ Assay previous run (14-4) _____ Approved by: _____ Time _____ Supervisor
3.	Set the centrifuge skimmer and bring 14-2 centrifuge to 870 RPM.	Time 870 RPM _____
4.	Turn on 14-3 air sparger	14-3 air sparger on _____
5.	With 14-2 at 870 RPM and using the "A" jet, jet slurry from 14-1 to 14-2 at the rate of 140 lbs. per minute. Check the rate at intervals to insure proper rate. Shut off agitator when the liquid level drops below the agitator blades and call the dispatcher and notify him of the coming 14-3-W sample.	Time start _____ Transfer rate _____ Time _____ _____ _____ _____ _____ Avg _____ 14-1 Agitator off _____
6.	Turn on 14-3 jacket cooling water.	14-3 Jacket water on _____
7.	When the transfer from 14-1 to 14-2 stops, indicated by levelling off of the 14-3 Wt. Ftr. trace (do not shut off jet), jet from 14-3 to 14-1 for 30 seconds and empty 14-1 again. Repeat this operation 2 times to flush out 14-1. Shut off jet on completion of the recycling.	Time start _____ Time end _____
8.	Add 500 lbs. of water to 14-1 via 14-1E.	Lbs. added _____ Time added _____
9.	With cooling water on the jacket, circulate through 14-1 spray for 3 minutes. Hold 14-1 temperature below 50°C.	Time start _____ Time end _____
10.	Jet 14-1 to 14-2 using the "a" jet. When transfer stops, indicated by the levelling off of the 14-3 Wt. Ftr. trace, shut off the "A" jet. Using the "B" jet, again jet 14-1 to 14-2.	Time end _____
11.	After 5 minutes skim 14-2 to a 10 gallon heel and plug 14-2 to stop.	Time stop _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VI - FIRST DECONTAMINATION (PRODUCT) - CENTRIFUGATION (Cont'd.)

12.	Shut off the 14-3 air sparger and read the Wt. Ftr., Temp. and Sp.Gr. when the Sp. Gr. meter becomes constant, then start 14-3 air sparger again.	14-3 Air sparger off _____ 14-3 Wt. Ftr. _____ Lbs. _____ 14-3 Sp. Gr. _____ Temp. _____ 14-3 Air sparger on _____
13.	Call dispatchdr give charge data and request that sample 14-3-W be taken.	Time called _____ Time sampled _____
14.	Add cake dissolving acid to 14-1 and start circulation (see page 5.15, step 1-4 inc.) before starting cake washing.	Time added _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VIII - FIRST DECONTAMINATION (PRODUCT) - CAKE WASHING

1. Bring 14-2 to 10 RPM and add 250 lbs. of water through the bowl sprays at full pump pressure.	Lbs. added _____ Time added _____
2. Bring 14-2 to 140 RPM hold 2 minutes and plug to stop. Repeat twice.	Time end _____
3. Read 14-2 Manometer	14-2 Manometer _____ Lbs. _____
4. Bring 14-2 to 870 RPM, hold 5 minutes and skim to a 10 gallon heel.	Time end _____
5. Plug 14-2 to stop.	Time stop _____
6. Bring 14-2 to 10 RPM and add 250 lbs. of water through the bowl sprays at full pump pressure.	Lbs. added _____ Time added _____
7. Bring 14-2 to 140 RPM, hold 2 minutes, then plug to stop. Repeat twice.	Time end _____
8. Bring 14 2 to 870 RPM, hold 5 minutes and skim to a 10 gallon heel.	Time end _____
9. Beckman reading on 14 2.	Meter _____ Factor _____
10. Plug 14 2 to stop.	Time stop _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

IX - FIRST DECONTAMINATION (PRODUCT) - PRODUCT CAKE SOLUTION

1.	Beckman reading on 14-4.	Meter _____ Factor _____
2.	Add 2020 lbs. of 60% Nitric Acid (Line G) to 14-1 via 14-1A.	Lbs. added _____ Time added _____
3.	Flush 14-1A to 14-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____ 14-1 Wt. Ftr. _____ Lbs. _____
4.	With cooling water on the jacket, circulate through 14-1 spray (50-70 lbs. steam pressure) for 20 min. HOLD 14-1 TEMPERATURE BELOW 50°C.	14-1 Jacket water on _____ Time start _____ Time end _____ 14-1 Jacket water off _____
5.	Call dispatcher and obtain permission to jet slurry from 14-2 to 14-4.	Time called _____ Time jetted _____
6.	Using the "A" jet, jet 330 lbs. of acid from 14-1 to 14-2.	14-2 Wt. Ftr. _____ Lbs. _____
7.	Bring 14-2 to 140 RPM, hold 2 minutes, then plug to stop. Repeat twice.	Time start _____ Time end _____
8.	With 14-2 stopped, jet 14-2 to 14-4.	Time jetted _____
9.	Start 14-4 agitator and jacket cooling water	14-4 agitator on _____ 14-4 Jacket water on _____
10.	Using the "A" jet, jet 330 lbs. of acid from 14-1 to 14-2.	14-2 Wt. Ftr. _____ Lbs. _____
11.	Bring 14-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time start _____ Time end _____
12.	With 14-2 stopped, jet 14-2 to 14-4.	Time jetted _____
13.	Using the "A" jet, jet 330 lbs. of acid from 14-1 to 14-2.	14-2 Wt. Ftr. _____ Lbs. _____
14.	Bring 14-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time start _____ Time end _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

IX - FIRST DECONTAMINATION (PRODUCT) - PRODUCT CAKE SOLUTION (Cont'd.)

15. With 14-2 stopped, jet 14-2 to 14-4. — Time jetted _____

16. Using the "A" jet, jet 330 lbs. of acid from 14-1 to 14-2. — 14-2 Wt. Ftr. _____ Lbs. _____

17. Bring 14-2 to 140 RPM, hold 2 min. then plug to stop. Repeat twice. — Time start _____ Time end _____

18. With 14-2 stopped, jet 14-2 to 14-4. — Time jetted _____

19. Beckman reading on 14-2. — Meter _____ Factor _____

Note: If the Beckman reading is greater than on the 10 scale, notify the supervisor and with his approval add the remaining acid in 330 lb. shots until the Beckman reaches this level or the acid is used up.

20. Bring 14-2 to 10 RPM and using the "A" jet, jet the remainder of acid from 14-1 to 14-2 to 14-4. Keep the bowl empty as possible during this operation. When the 14-1 to 14-2 transfer stops, shut off the "A" jet and plug 14-2 to stop. — Time start _____ Time end _____
Time stop _____

21. Jet 14-2 to 14-4. — Time jetted _____

22. Using the "B" jet again, jet 14-1 to 14-2. When the transfer stops, shut off jet and read 14-2. — Time start _____ Time end _____
14-2 Manometer _____ Lbs. _____

23. Jet 14-2 to 14-4. — Time jetted _____

24. Beckman reading on 14-2. — Meter _____ Factor _____

25. Shut off 14-3 air sparger and read Wt. Ftr., Temp. and Sp.Gr. when Sp. Gr. meter becomes constant, then turn on 14-3 air sparger again (TW approx. 28,700 lbs.) — 14-3 Air Sparger off _____
14-3 Wt. Ftr. _____ Lbs. _____
14-3 Sp.Gr. _____ Temp. _____
14-3 Air Sparger on _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

IX - FIRST DECONTAMINATION (PRODUCT) - PRODUCT CAKE SOLUTION (Cont'd.)

26. Agitate 14-4 for 1 hour after the final addition of dissolving acid (Step 23). Keep temperature below 40°C.

Time start _____ Time end _____

27. Stop 14-4 agitator and read Wt. Ftr., Temp. and Sp.Gr. when Sp. Gr. meter becomes constant, then start 14-4 agitator, (TW approx. 2575 lbs.)

14-4 agitator off _____

14-4 Wt. Ftr. _____ Lbs. _____

14-4 Sp. Gr. _____ Temp. _____

14-4 Agitator on _____

28. Beckman reading on 14-4.

Meter _____ Factor _____

29. Call dispatcher, give charge data and request that sample 14-4-P be taken.

Time called _____

Time sampled _____

30. When the dispatcher approves, proceed with the oxidation.

Time approved _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

X - FIRST DECONTAMINATION (PRODUCT) - WASTE FROM 1ST PRODUCT PRECIPITATION

1. Beckman reading on 14-3 — Meter _____ Factor _____

2. When the dispatcher approves, shut off the air sparger and jet first half of 1st product precipitation waste in 14-3 to 15-7. — Time approved _____

14-3 air sparger off _____

Time start _____ Time end _____
14-3 Wt. Ftr _____ Lbs. _____

3. Turn on 14-3 air sparger — 14-3 air sparger on _____

4. When the dispatcher approves, jet the last half of the 1st product precipitation waste from 14-3 to 15-7. — Time approved _____

Time start _____ Time end _____

14-3 Wt. Ftr. _____ Lbs. _____

5. Shut off 14-3 air sparger and jacket cooling water — 14-3 air sparger off _____

14-3 Jacket water off _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

I - SECOND DECONTAMINATION (PRODUCT) - OXIDATION

1. With 14-4 agitator running, add water to 14-4 via 14-4F to give a total weight of 3500 lbs. in 14-4. (14-4 Wt. Ftr. reading of 4.3) Do not add more than 1000 lbs. of water.	14-4 Agitator running _____ Lbs. added _____ Time added _____
2. Adjust 14-4 temperature to 45°C and maintain at this temperature.	Time 45°C _____
3/ Add 130 lbs. of 10% Sodium Bismuthate Slurry (Dolly) to 14-4 via funnel F-14-4-F.	Lbs. added _____ Time added _____
4. Flush dolly and funnel F-14-4-F to 14 4 with 50 lbs. of process water.	Lbs. added _____ Time added _____
5. Add 55 lbs. of 10% Sodium Dichromate solution (Line D) to 14 4 via 14-4-F.	Lbs. added _____ Time added _____
6. Flush 14-4-F to 14-4 with 50 lbs. of process water.	Lbs. added _____ Time added _____
7. Digest 14-4 at 45°C for 1 hour. DO NOT EXCEED 45°C. During this operation proceed to page 6.2, steps 1-4 inc.	Time start _____ Time end _____
8. Cool to 35°C.	Time start _____ Time end _____
9. Leave 14-4 agitator running.	

[redacted]

[redacted]

Date _____

Run No. _____

DECLASSIFIED

II SECOND DECONTAMINATION (BY-PRODUCT) - PRECIPITATION

1. Beckman reading on 16-1. — Meter _____ Factor _____

2. Add 12,060 lbs. of water to 16-1 via 16-1A in four portions of 3015 lbs. each. — 1st Port _____ lbs. time added _____
2nd " _____ " " " _____
3rd " _____ " " " _____
4th " _____ " " " _____
16-1 Wt. Ftr. _____ Lbs. _____

3. Start 16-1 agitator. — 16-1 agitator on _____

4. Heat water in 16-1 to 40°C with jacket steam. At 40°C, shut off steam until solution is received from 14-4.

5. When approved by the dispatcher, receive oxidized solution from 14-4 into 16-1 (TW in 16-1 15,400 lbs.) — Time approved _____
Time start _____ Time end _____

6. Turn off 14-4 agitator. — 14-4 agitator off _____

7. Add 208 lbs. of 75% Phosphoric Acid (Line F) to 16-1 rapidly via 16-1E — Lbs. added _____ Time added _____

8. Flush 16-1E to 16-1 with 50 lbs. of process water. — Lbs. added _____ Time added _____

9. Heat 16-1 to 75°C with jacket steam — Time start _____ Time 75°C _____

10. Digest 16-1 for 1 hour at 75°C . — Time end _____

11. Beckman reading on 16-1 during digestion. — Meter _____ Factor _____

12. Turn cooling water on 16-1. — 16-1 jacket water on _____

13. Stop 16-1 agitator and read Wt. Ftr., Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 16-1 agitator. — 16-1 agitator off _____
16-1 Wt. Ftr. _____ Lbs. _____
16-1 Sp. Gr. _____ Temp. _____
16-1 agitator on _____

14. Cool 16-1 to 50°C and proceed with centrifugation. Leave cooling water on until 16-1 is empty or at 25°C. — Time 50°C _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

III - SECOND DECONTAMINATION (BY-PRODUCT) - CENTRIFUGATION

1. Beckman reading on 16-2.	Meter _____ Factor _____
2. Set centrifuge skimmer and bring 16-2 centrifuge to 870 RPM.	Time 870 RPM _____
3. Turn on 16-3 air sparger and jacket cooling water.	16-3 Air sparger on _____ 16-3 Jacket water on _____
4. With 16-2 at 870 RPM and using the "A" jet, jet slurry from 16-1 to 16-2 at the rate of 140 lbs. per minute Check the rate at intervals to insure the proper rate. Shut off 16-1 agitator when the Wt. Ftr. reaches 0.2.	Time start _____ Time end _____ Transfer rate _____ Time _____ _____ _____ _____ _____ Avg. _____ 16-1 Agitator off _____
5. When the transfer from 16-1 to 16-2 stops, indicated by levelling off of the 16-3 Wt. Ftr. trace (do not shut off jet), jet from 16-3 to 16-1 for 30 seconds and empty 16-1 again. Repeat this operation 2 times to flush out 16-1. Shut off jet on completion of the recycling.	Time start _____ Time end _____
6. Add 500 lbs. of water to 16-1 via 161 E.	Lbs. added _____ Time added _____
7. With cooling water on the jacket, circulate through 16-1 spray at 60 - 70 psi for 3 minutes. Hold 16-1 temperature below 50°C.	Time start _____ Time end _____
8. Using the "A" jet, jet 16-1 to 16-2 when transfer stops, indicated by the levelling off of the 16-3 Wt. Ftr. trace, shut off the "A" jet. Using the "B" jet, again jet 16-1 to 16-2.	Time start _____ Time end _____
9. After 5 minutes skim 16-2 to a 10 gallon heel and plug to stop.	Time stop _____
10. Add cake dissolving acid to 16-1 and start circulation (See page 6.5, steps 3, 4, and 5)	Time added _____ _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

IV - SECOND DECONTAMINATION (BY-PRODUCT) - CAKE WASHING

1. Bring 16-2 to 10 RPM, add 250 lbs. of water through the bowl sprays at full pump pressure.	Lbs. added _____ Time added _____
2. Bring 16-2 to 140 RPM, hold 2 minutes then plug to stop. Repeat twice.	Time end _____
3. Bring 16-2 to 870 RPM, hold 5 minutes, and skim to a 10 gal. heel.	Time end _____
4. Plug 16-2 to stop.	Time stop. _____
5. Bring 16-2 to 10 RPM and add 250 lbs. of water through the bowl sprays at full pump pressure.	Lbs. added _____ Time added _____
6. Bring 16-2 to 140 RPM, hold 2 minutes, then plug to stop. Repeat twice.	Time end _____
7. Bring 16-2 to 870 RPM, hold 5 minutes and skim to a 10 gal. heel.	Time end _____
8. Beckman reading on 16-2	Meter _____ Factor _____
9. Plug 16-2 to stop.	Time stop _____
10. Call dispatcher and obtain permission to jet 16-3 to 17-1. (See page 6.7, steps 1, 2, and 3)	Time called _____ 16-3 Wt. Ftr. _____ Lbs. _____ Time start _____ Time end _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

V - SECOND DECONTAMINATION (BY-PRODUCT) - BY-PRODUCT CAKE SOLUTION

1.	Beckman reading on 16-4.	Meter _____ Factor _____
2.	Jet slurry in 16-2 to 16-4.	Time start _____ Time end _____
3.	Add 1100 lbs. of 60% Nitric Acid (Line G) to 16-1 via 16-1A, and read 16-1 Wt. Ftr.	Lbs. added _____ Time added _____ 16-1 Wt. Ftr. _____ Lbs. _____
4.	Flush 16-1A to 16-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____
5.	With cooling water on the jacket, circulate through 16-1 spray (50-70 lbs. steam pressure) for 20 minutes. HOLD 16-1 TEMPERATURE BELOW 50°C.	16-1 jacket water on _____ Time start _____ Time end _____ Maximum temperature 16-1 _____
6.	With 16-2 stopped and using the "A" jet, jet 330 lbs. of acid from 16-1 to 16-2.	16-2 Wt. Ftr. _____ Lbs. _____
7.	Bring 16-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time start _____ Time end _____
8.	Start 16-4 agitator and turn on 16-4 jacket water	16-4 agitator on _____ 16-4 Jacket water on _____
9.	With 16-2 stopped, jet 16-2 to 16-4.	Time start _____ Time end _____
10.	Using the "A" jet, jet 330 lbs. of acid from 16-1 to 16-2.	16-2 Wt. Ftr. _____ Lbs. _____
11.	Bring 16-2 to 140 RPM, hold 2 mins, then plug to stop. Repeat twice.	Time start _____ Time end _____
12.	With 16-2 stopped, jet 16-2 to 16-4.	Time start _____ Time end _____
13.	Using the "A" jet, jet 330 lbs. of acid from 16-1 to 16-2.	16-2 Wt. Ftr. _____ Lbs. _____
14.	Bring 16-2 to 14- RPM, hold 2 mins., then plug to stop. Repeat twice.	Time start _____ Time end _____
15.	With 16-2 stopped, jet 16-2 to 16-4.	Time start _____ Time end _____
16.	Using the "A" jet, jet 330 lbs. of acid from 16-1 to 16-2.	16-2 Wt. Ftr. _____ Lbs. _____
17.	Bring 16-2 to 140 RPM, hold 2 mins., then plug to stop. Repeat twice.	Time start _____ Time end _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

V - SECOND DECONTAMINATION (BY-PRODUCT) - BY-PRODUCT CAKE SOLUTION (Cont'd.)

18. With 16-2 stopped, jet 16-2 to 16-4. — Time jetted _____

19. Beckman reading on 16.2 — Meter _____ Factor _____

Note: If the Beckman reading is greater than on the 10 scale, notify the supervisor and hold pending his instructions.

20. Bring 16-2 to 10 RIM and using the "A" jet, jet the remainder of acid from 16-1 to 16-2 to 16-4. Keep the bowl empty as possible during this operation. When the 16-1 to 16-2 transfer stops, shut off the "A" jet and plug 16-2 to stop. — Time start _____ Time end _____ Time stop _____

21. Jet 16-2 to 16-4. — Time jetted _____

22. Using the "B" jet, again jet 16-1 to 16-2. When the transfer stops, shut off the jet and read 16-2 manometer. — Time start _____ Time end _____ 16-2 Manometer _____ Lbs. _____

23. Jet 16-2 to 16-4. — Time jetted _____

24. Beckman reading on 16-4. — Meter _____ Factor _____

25. Agitate 16-4 for one hour at 35-40°C. — Time start _____ Time end _____

26. Stop 16-4 agitator and read Wt. Ftr., Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 16-4 agitator. — 16-4 Agitator off _____ 16-4 Wt. Ftr. _____ Lbs. _____ 16-4 Sp. Gr. _____ Temp. _____ 16-4 Agitator on _____

27. Call dispatcher, give charge data and request that sample 16-4-EP be taken. — Time called _____ Time sampled _____

28. When approved by the dispatcher, jet 16-4 to 15-9. — Time approved _____ Time jetted _____

29. Shut off 16-4 jacket cooling water. — 16 4 Jacket water off _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VI - SECOND DECONTAMINATION (PRODUCT) - REDUCTION AND PRECIPITATION

1. Beckman reading on 17-1. Meter _____ Factor _____

2. Receive effluent from 16-3 into 17-1 (TW in 17-1 approx. 16,500 lbs.) Shut off 16-3 air sparger on completion of transfer.
   Time start _____ Time end _____
   17-1 Wt. Ftr. _____ Lbs. _____
   17-1 Sp. Gr. _____ Temp. _____
   16-3 Air sparger off _____

3. Beckman reading on 17-1. Meter _____ Factor _____

4. Start 17-1 agitator and adjust temperature to 40-50°C. DO NOT EXCEED 50°C. until step 6 is completed.
   17-1 agitator on _____

5. Add 1160 lbs. of 14% Ammonium Silico Fluoride solution (Line H) in two portions of 500 lbs. and one of 160 lbs. to 17-1 via 17-1E
   1st Port. _____ Lbs.
   Time added _____
   2nd Port. _____ Lbs.
   Time added _____
   3rd Port. _____ Lbs.
   Time added _____ (Total)

6. Flush 17-1E to 17-1 with 50 lbs. of process water.
   Lbs. added _____ Time added _____

7. Add 1520 lbs. of 20% Iron Solution (Line M) to 17-1 via 17-1A.
   Lbs. added _____ Time added _____

8. Flush 17-1A to 17-1 with 50 lbs. of process water.
   Lbs. added _____ Time added _____

9. Heat 17-1 to 75°C with jacket steam and maintain temperature until step 15 is completed.
   Time start _____ Time 75°C _____

10. Add 168 lbs. of 24% Bismuth Nitrate solution (Line R) to 17-1 via 17-1E at the rate of 3 lbs. per minute.
    Time start _____ Time end _____
    Lbs. added _____

11. Flush 17-1E to 17-1 with 50 lbs. of process water.
    Lbs. added _____ Time added _____

12. Digest 17-1 for 30 minutes at 75°C.
    Time start _____ Time end _____

13. Add 1510 lbs. of 75% Phosphoric Acid (Line F) to 17-1 via 17-1A at a rate of 25 lbs. per minute.
    Time start _____ Time end _____
    Lbs. added _____

14. Flush 17-1A to 17-1 with 50 lbs. of process water.
    Lbs. added _____ Time added _____

Date _____
Run No. _____

DECLASSIFIED

VI SECOND DECONTAMINATION (PRODUCT) - REDUCTION AND PRECIPITATION (Cont'd)

15.	Digest 17-1 for 2 hours at 75°C.	Time start _____ Time end _____
16.	Beckman reading on 17-1 during digestion.	Meter _____ Factor _____
17.	Turn cooling water on 17-1.	17-1 Jacket water on _____
18.	Stop 17-1 agitator, read Wt. Ftr. Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 17-1 agitator.	17-1 Agitator off _____ 17-1 Wt. Ftr. _____-Lbs. _____ 17-1 Sp. Gr. _____ Temp. _____ 17-1 Agitator on _____
19.	Cool 17-1 to 50°C and proceed with centrifugation. Leave cooling water on until 17-1 is empty or at 25°C.	Time 50°C _____

[redacted]

DECONTAMINATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

VI - SECOND DECONTAMINATION (PRODUCT) - CENTRIFUGATION

1.	Beckman reading on 17-2.	Meter _____ Factor _____
2.	Supervisor's approval to start centrifugation.	Beckman limit _____ Assay previous run (17-4) _____% Approved by: _____ Time _____ Supervisor
3.	Set the centrifuge skimmer and bring 17-2 centrifuge to 870 RPM.	Time 870 RPM _____
4.	Turn on 17-3 air sparger.	17-3 Air Sparger on _____
5.	With 17-2 at 870 RPM and using the "A" jet, jet slurry from 17-1 to 17-2 at the rate of 140 lbs. per minute. Check the rate at intervals to insure proper rate. Shut off 17-1 agitator when the liquid level drops below the agitator blades and call the dispatcher and notify him of the coming 17-3-W sample.	Time start _____ Transfer rate _____ Time _____ _____ _____ _____ _____ Avg. _____ 17-1 Agitator off _____
6.	Turn on 17-3 jacket cooling water.	17-3 jacket water on _____
7.	When the transfer from 17-1 to 17-2 stops, indicated by the levelling off of the 17-3 Wt. Ftr. trace (do not shut off jet), jet from 17-3 to 17-1 for 30 seconds and empty 17-1 again. Repeat this operation 2 times to flush out 17-1. Shut off jet on completion of the recycling.	Time start _____ Time end _____
8.	Add 500 lbs. of water to 17-1 via 17-1E.	Lbs. added _____ Time added _____
9.	With cooling water on the jacket, circulate through 17-1 spray for 3 minutes. Hold 17-1 temperature below 50°C.	Time start _____ Time end _____
10.	Jet 17-1 to 17-2 using the "A" jet. When transfer stops, indicated by the levelling off of the 17-3 Wt. Ftr. trace, shut off the "A" jet. Using the "B" jet, again jet 17-1 to 17-2.	Time end _____
11.	After 5 minutes skim 17-2 to a 10 gallon heel and plug 17-2 to stop.	Time stop _____

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Date _____
Run No. _____

DECLASSIFIED

VI. - SECOND DECONTAMINATION (PRODUCT) CENTRIFUGATION (Cont'd)

12.	Shut off the 17-3 air sparger and read the Wt. Ftr., Temp. and Sp. Gr. when the Sp. Gr. meter becomes constant, then start 17-3 air sparger again.	17-3 Air sparger off _____ 17-3 Wt. Ftr. _____Lbs._____ 17-3 Sp. Gr. _____Temp._____ 17-3 Air Sparger on _____
13.	Call dispatcher give charge data and request that sample 17-3 W to be taken.	Time called _____ Time sampled _____
14.	Add cake dissolving acid to 17-1 and start circulation (See page 6.12, steps 1-4 inc.) before starting cake washing.	Time added _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VII -SECOND DECONTAMINATION (PRODUCT) CAKE WASHING

1. Bring 17-2 to 10 RPM, add 250 lbs. of water through the bowl sprays at full pump pressure. — Lbs. added _____ Time added _____
2. Bring 17-2 to 140 RPM, hold 2 minutes then plug to stop. Repeat twice. — Time end _____
3. Read 17-2 Manometer — Manometer _____ Lbs. _____
4. Bring 17-2 to 870 RPM, hold 5 minutes, and skim to a 10 gallon heel. — Time end _____
5. Plug 17-2 to stop. — Time stop. _____
6. Bring 17-2 to 10 RPM and add 250 lbs. of water through the bowl sprays at full pump pressure. — Lbs. added _____ Time added _____
7. Bring 17-2 to 146 RPM , hold 2 minutes, then plug to stop. Repeat twice. — Time end _____
8. Bring 17-2 to 870 RPM, hold 5 minutes and skim to a 10 gallon heel. — Time end _____
9. Beckman reading on 17-2. — Meter _____ Factor _____
10. Plug 17-2 to stop. — Time stop _____

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DECONTAMINATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

VIII - SECOND DECONTAMINATION (PRODUCT) PRODUCT CAKE SOLUTION

1.	Beckman reading on 17-4	Meter _____ Factor _____
2.	Add 1570 lbs. of 60% Nitric Acid (LineG) to 17-1 via 17-1A.	Lbs. added _____ Time added _____
3.	Flush 17-1A to 17-1 with 50 lbs. of process water.	Lbs. added _____ Time added _____ 17-1 Wt. Ftr. _____ Lbs. _____
4.	With cooling water on the jacket, circulate through 17-1 spray (50-70) lbs. steam pressure) for 20 minutes. HOLD 17-1 TEMPERATURE BELOW 50°C.	17-1 jacket water on _____ Time start _____ Time end _____ Maximum temperature _____ 17-1 jacket water off _____
5.	Call dispatcher and request permission to jet slurry from 17-2 to 17-4.	Time called _____ Time jetted _____
6.	Using the "A" jet, jet 330 lbs. of acid from 17-1 to 17-2.	17-2 Wt. Ftr. _____ Lbs. _____
7.	Bring 17-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time end _____
8.	With 17-2 stopped, jet 17-2 to 17-4.	Time jetted _____
9.	Start 17-4 agitator and jacket cooling water.	17-4 agitator on _____ 17-4 jacket water on _____
10.	Using the "A" jet, jet 330 lbs. of acid from 17-1 to 17-2.	17-2 Wt. Ftr. _____ Lbs. _____
11.	Bring 17-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time end _____
12.	With 17-2 stopped, jet 17-2 to 17-4.	Time jetted _____
13.	Using the "A" jet, jet 330 lbs. of acid from 17-1 to 17-2.	17-2 Wt. Ftr. _____ Lbs. _____
14.	Bring 17-2 to 140 RPM, hold 2 mins. then plug to stop. Repeat twice.	Time end _____
15.	With 17-2 stopped, jet 17-2 to 17-4.	Time jetted _____
16.	Using the "A" jet, jet 330 lbs. of acid from 17-1 to 17-2.	17-2 Wt. Ftr. _____ Lbs. _____

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[redacted]

Date _____
Run No. _____

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VIII - SECOND DECONTAMINATION (PRODUCT) PRODUCT CAKE SOLUTION (Cont'd)

19. Bring 17-2 to 140 RPM, hold 2 minutes then plug to stop. Repeat twice. — Time end _____

20. With 17-2 stopped, jet 17-2 to 17-4. — Time jetted _____

21. Beckman reading on 17-2 — Meter _____ Factor _____

Note: If the Beckman reading is greater than on the 10- scale, notify the supervisor and with his approval add the remaining acid in 330 lb shots until the Beckman reaches this level or the acid is used up.

22. Bring 17-2 to 10 RPM and using the "A" jet, jet the remainder of acid from 17-1 to 17-2 to 17-4. Keep the bowl empty as possible during this operation. When the 17-1 to 17-2 transfer stops, shut off the "A" jet and plug 17-2 to stop. — Time start _____ Time end _____ Time stop _____

23. Jet 17-2 to 17-4. — Time jetted _____

24. Using the "B" jet, again jet 17-1 to 17-2. When the transfer stops, shut off jet and read 17-2 manometer. — Time start _____ Time end _____ 17-2 Manometer _____ Lbs. _____

25. Jet 17-2 to 17-4 — Time jetted _____

26. Beckman reading on 17-2. — Meter _____ Factor _____

27. Shut off 17-3 air sparger and read Wt. Ftr. Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 17-3 air sparger again (TW approx. 23740 lbs.) — 17-3 air sparger off _____ 17-3 Wt. Ftr. _____ Lbs. _____ 17-3 Sp. Gr. _____ Temp. _____ 17-3 Air sparger on _____

28. Agitate 17-4 for 1 hour after final addition of dissolving acid (Step 25 above). Keep temperature below 40°C. — Time end _____

29. Beckman reading on 17-4. — Meter _____ Factor _____

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[redacted]

DECONTAMINATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

VIII - SECOND DECONTAMINATION (PRODUCT) PRODUCT CAKE SOLUTION (Cont'd)

30.	Stop 17-4 agitator and read Wt. Ftr., Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 17-4 agitator again. (TW approx. 2020 lbs.)	17-4 Agitator off _____ 17-4 Wt. Ftr. _____ Lbs. _____ 17-4 Sp. Gr. _____ Temp. _____ 17-4 Agitator on _____
31.	Call dispatcher, give charge data and request that sample 17-4-P be taken.	Time called _____ Time sampled _____
32.	When the dispatcher approves, call operator at Section C Bldg. 224 and jet 17-4 to C-4. When notified that the transfer has stopped, put jet on air for 2 minutes, then shut off and check Wt. shipped against Wt. received in 224 Bldg. (3% jetting dilution). If transfer is incomplete, again jet 17-4 to C-4.	Time called _____ Time transfer complete _____ Air blow complete _____ Wt. Shipped 17-4 _____ Wt. Received in C-4 _____
33.	Shut off 17-4 agitator and jacket cooling water.	17-4 agitator off _____ 17-4 Jacket water off. _____

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[redacted]

DECONTAMINATION OPERATIONS

Date _____
Run No. _____

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IX · SECOND DECONTAMINATION (PRODUCT) WASTE FROM 2ND PRODUCT PRECIPITATION

1. Beckman reading on 17-3 — Meter _____ Factor _____

2. When the dispatcher approves, shut off the air sparger and jet first half of 2nd product precipitation waste in 17-3 to 15-6. — Time approved _____ 17-3 Air sparger off _____ Time start _____ Time end _____ 17-3 Wt. Ftr. _____ Lbs. _____

3. Turn on 17-3 air sparger. — 17-3 Air sparger on _____

4. When the dispatcher approves, q jet the last half of the 2nd product precipitation waste in 17-3 to 15-6. — Time approved _____ Time start _____ Time end _____ 17-3 Wt. Ftr. _____ Lbs. _____

5. Shut off 17-3 air sparger and jacket water. — 17-3 Air sparger off _____ 17-3 Jacket water off _____

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CHAPTER VI - DECONTAMINATION

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SECTION C - SEPARATIONS

CHAPTER VII - CONCENTRATION

The extraction and decontamination steps carried out in the Canyon Building yield a dilute product solution containing less than 0.001% of the original fission activity. The main purpose of the operations carried out in the Concentration Building is that of bulk reduction or concentration of this product solution. The bulk after concentration is only about 3.6% of that before the operation and a cross-over from bismuth to lanthanum carrier reduces the weight of carrier associated with the product about 7.5-fold. In addition to this concentration, a secondary purpose of the process is that of increasing the decontamination of the product approximately 100-fold, to a final over-all factor of 10^7 or greater. The operations carried out in the Concentration Building are discussed in this chapter.

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CHAPTER VII - CONCENTRATION

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GENERAL DESCRIPTION OF THE PROCESS

In the Concentration Building, the product is concentrated to the point where it is ready for direct precipitation without carrier in the Isolation Building. In addition, the product is decontaminated further to an over-all factor of 10^7, which permits the concentrated product solution to be handled in the isolation process without any shielding at a distance of a few feet with no radiation hazard.

In order to carry out the two main objectives described above, it is necessary to (1) supplement the removal of two of the major fission activities very nearly eliminated in Canyon Building steps, zirconium and columbium; (2) eliminate as completely as possible the remaining fission activities, namely lanthanum and rare earth components; (3) separate the bismuth phosphate carrier from the product and "cross-over" to a carrier which will co-separate or carry product at lower carrier-to-product weight ratios than bismuth, such as lanthanum fluoride; and (4) convert the relatively acid-insoluble lanthanum fluoride product precipitate by "metathesis" to a highly soluble compound, such as lanthanum hydroxide, which will permit the delivery of a small-volume concentrated product solution to the isolation process.

The second-cycle bismuth phosphate product solution as it arrives from the Canyon Building has a volume of approximately 200 gallons and contains the product in association with a total of 43 lbs. of bismuth phosphate. Its gamma fission activity, after a decontamination of 10^5 in the Canyon Building, is composed essentially of approximately 25% lanthanum and 75% zirconium and columbium. After oxidation in Cell C, a standard "bismuth phosphate by-product" precipitation (without scavengers) is carried out in Cell A. This serves to eliminate the bismuth carrier, which would precipitate later in the fluoride cycle, and most of the zirconium and columbium fission activities. The gross decontamination factor for this step is usually 3.5 to 4 and product losses average 0.2 to 0.3%.

After a supplementary permanganate oxidation of the product effluent, to assure the complete maintenance of the product in the higher valence (VI) state, a "lanthanum fluoride by-product" precipitation is carried out in Cell D. In this step, most of the lanthanum and rare earth fission activities, being fluoride insoluble, are removed. In addition, this step is a convenient point to "recycle" the lanthanum - containing isolation supernatants as a means of recovering the product left in solution (2-3%) when the product peroxide is precipitated at the Isolation Building. The gross decontamination factor for this step is usually 12 to 13 and product losses average 0.7 to 0.8%.

Having carried out two by-product precipitations and separated the bismuth phosphate carrier from the product, the product solution is then ready for a cross-over to carrying by a second carrier, lanthanum. The oxidized product is reduced with oxalate (catalyzed by manganous ion from the permanganate added in Cell D) and a lanthanum fluoride product precipitation carried out in Cell E. Due to the difficulty of separating lanthanum fluoride precipitates,

[redacted]

which are very finely divided and almost colloidal in nature, at least two strikes and centrifugations must be made by returning the first centrifugate to the precipitator and repeating the first strike procedure again. Very little, if any, decontamination is obtained in this step, since more than half of the remaining fission activities (traces of lanthanum, rare earths, zirconium and columbium) are fluoride insoluble. Product waste losses in the effluent usually average less than 1.5%.

The product has then been separated with a relatively small weight of carrier, approximately 5.5 lbs. of lanthanum fluoride. The carrier is but slightly soluble in acid, however. Thus, the lanthanum fluoride product precipitate is transferred to Cell F and metathesized to a lanthanum hydroxide product precipitate by digestion with potassium hydroxide solution. After separation of the lanthanum hydroxide product precipitate by centrifugation, it must then be slurried back into a wash solution which serves to dilute the fluoride ions liberated by the metathesis. This wash solution is made high enough in potassium hydroxide (2%) to prevent colloidal dispersion and leak-through of the lanthanum hydroxide in its second centrifugation. The fluoride ion must be removed to prevent reprecipitation of lanthanum fluoride when the lanthanum hydroxide product precipitate is dissolved in acid. The last step in the concentration process is the solution of lantharum hydroxide and product in enough 60% nitric acid to dissolve the entire cake and produce a small-volume product solution 2N in HNO_3 that is shipped in a stainless steel container by truck to the Isolation Building. Essentially no decontamination is obtained in the metathesis cycle and product losses in the two wastes usually total approximately 1.0%.

The over-all result is a concentration of the product with respect to carrier element of about 7.5, 30 lbs. of bismuth reduced to 4.0 lbs. of lanthanum. The bulk reduction of the product solution is approximately 27, 200 gallons of second-cycle product cake solution reduced to 7.3 gallons of final lanthanum nitrate solution. The decontamination factor obtained in the concentration process is approximately 100, resulting in a final over-all decontamination of the original fission activities present at extraction of greater than 10^7. The residual gamma-emitting fission activities associated with the product in this concentrated solution are composed of more than 50% lanthanum, with the remainder consisting of zirconium, columbium and ruthenium.

CHEMISTRY OF THE STANDARD PROCESS

Bismuth Phosphate By-Product

The chemistry of the concentration process $BiPO_4$ by-product precipitation is the same as for the second decontamination cycle by-product in the Canyon Building (See Chapter VI). The chief function of the precipitation at this point is the removal of bismuth from the product solution in order to prepare the solution for a cross-over to a carrier, such as lanthanum, which is capable of co-precipitating product at lower carrier-to-product ratio. Bismuth oxalate would precipitate in large quantities during the

subsequent reduction step and contribute too much carrier to the final product cake to permit its being dissolved in a sufficiently small volume to deliver to the isolation process.

The decontamination effected by the $BiPO_4$ by-product precipitation, while secondary to the purpose of bismuth elimination, is still relatively important. Essentially no data on the specificity of the cross-over $BiPO_4$ by-product precipitation for fission activity removal are available because of the low levels of activity present in the large volume of solution (References 1, 2). Plant test data show, however, that of the starting solution in the concentration process, approximately 75% of the total gamma activity is composed of zirconium and columbium and 25% of lanthanum, with small amounts of primarily rare earth activities (Reference 3). The decontamination factor of 3.5 to 4, which is usually indicated by plant Beckman meter readings at this step, can be assumed to be effected by the removal of nearly all of the zirconium and columbium present.

Oxidation

As outlined in Chapter VI, the use of $NaBiO_x$ as the primary oxidant and $Na_2Cr_2O_7$ as the holding oxidant constitute the preferred oxidation system. In using these oxidants, 0.01M $NaBiO_x$ in 5 HNO_x solution has been used successfully to oxidize product in 1 hour at 50 °C, making the oxidized solution 0.025N in $Na_2Cr_2O_7$ holding oxidant at the end. In order to provide greater safety from the undesirable precipitation of $BiPO_4$ during oxidation, however, the preferred oxidation method is carried out at 7.5N HNO_x. The $NaBiO_x$ and $Na_2Cr_2O_7$: bismuth carrier ratios are kept the same as in the 5N HNO_x oxidation process, making the minimum recommended concentration 0.013M $NaBiO_x$ and 0.033N $Na_2Cr_2O_7$. In addition, this 7.5N HNO_x oxidation is carried out at 45 °C, rather than 50 °C, to counteract the more rapid decomposition of the $NaBiO_x$ at the higher acidity. The $Na_2Cr_2O_7$ holding oxidant is also preferably added with the $NaBiO_x$ at the start of oxidation so that an oxidizing medium will be present after the decomposition of the $NaBiO_x$ is completed.

The use of 0.01M $KMnO_4$ instead of $NaBiO_x$ as the primary oxidant in 5N HNO_x solution has been successful in the laboratory (Reference 4), but semi-works tests failed to show complete oxidation in the high acid solutions, excessive reduction of the $KMnO_4$ to MnO_2 taking place (Reference 5). The substitution of 0.01M $KMnO_4$ for $Na_2Cr_2O_7$ as the holding oxidant has been successful in semi-works trials, however (Reference 6).

Precipitation

The precipitation of the cross-over $BiPO_4$ by-product differs slightly from the standard by-product in the Canyon Building in that it is carried out at 1.4N rather than at 1.25N HNO_x and on approximately three-fourths of the volume scale. The reasons for this are practical rather than theoretical. At start-up, when large process solution volumes were involved, it was necessary to dilute the 5N oxidized product solution to no lower than 1.4N HNO_x in order to prevent tank overflow in subsequent process steps. As reduced process volumes were developed, the 1.4N HNO_x dilution was retained in order to maintain minimum process volumes in the fluoride cycle. The H_xPO_4 concentration used in the 1.4N HNO_x precipitation solution is 0.11S, adjusted to

maintain the HNO_3/H_3PO_4 molarity ratio at 1.25/0.1. Bismuth solubility under these conditions is approximately 35 mg/l.

Lanthanum Fluoride

Lanthanum fluoride as used in both the by-product and product precipitations has enough in common in these two uses for them to be discussed jointly as to general characteristics. Lanthanum fluoride as normally precipitated is a finely divided, amorphous, hydrated, flocculent mass, which even under the electron microscope shows no definite crystalline structure, but rather a small, ill-defined mass less than 0.01 micron in size (Reference 7). In this form, it is extremely difficult to remove quantitatively from plant solutions in the large volumes in which it is formed. Extensive experimental work has been carried out in the laboratory, semi-works, and plant on the precipitation and separation of lanthanum fluoride. Its sub-microscopic particle size has greatly hindered this work, however, and the majority of the data available on its properties are admittedly confusing and often contradictory.

When precipitated from an oxidized (VI) product solution, lanthanum fluoride does not carry product and has the formula LaF_3. It does carry down fluoride - insoluble fission activities, such as lanthanum, cerium (III), yttrium, and other rare earths (Reference 1).

When precipitated from a reduced (IV) product solution, it carries product, apparently by a double salt formation such as $2\ LaF_3 \cdot PuF_4$. Thus the theoretical minimum carrying ratio is 2 mols of lanthanum/mol of product or 1.17 weight units of lanthanum/weight unit of product. For purposes of simplicity, lanthanum fluoride is referred to henceforth as LaF_3, regardless of its being precipitated in either an oxidized or reduced solution.

Precipitation Methods

There are two major ways in which LaF_3 may be precipitated. "Coformed" LaF_3 is precipitated by adding hydrofluoric acid to a lanthanum - containing solution. "Preformed" LaF_3 is precipitated by adding lanthanum to an hydrofluoric acid-containing solution. Laboratory studies have indicated that coformed LaF_3 flocculates more readily and separates more easily than preformed LaF_3 (Reference 8). Clinton plant experience demonstrated the reverse to be true, however (Reference 8). Process-wise, a choice between the two methods could be made in precipitating the LaF_3 by-product (if no recycle is involved), but the presence of hydrofluoric acid at the time of making the product LaF_3 precipitation (from the by-product) leaves no choice but a preformed LaF_3 strike.

In precipitating the LaF_3 by-product, 0.2M hydrofluoric acid is used. This is a high enough concentration to obtain low LaF_3 solubilities and still not cause too rapid corrosion. When all the lanthanum is added as "fresh" lanthanum, a single preformed strike of approximately 500 mg/l is made. When recycled lanthanum (from isolation supernatants) is used as the sole source of lanthanum, a single coformed strike of approximately 720 mg/l is used.

There is no choice between preformed and coformed LaF_3 in the latter case since it is necessary to add the recycled lanthanum solution before the hydrofluoric acid in order to oxidize the reduced product it contains. No difference in separation efficiency between the two methods, as measured by Beckman meter decontamination calculations, has been observed in the plant. A single strike, rather than a double strike, provides satisfactory separation of LaF_3 at these high lanthanum concentrations.

In precipitating the LaF_3 product, 0.5M hydrofluoric acid is used. Two approximately 125 mg/l lanthanum strikes are carried out by returning the first centrifugation effluent to the precipitator and repeating the first strike procedure. Both strikes are necessarily performed, because of the presence of hydrofluoric acid from the by-product effluent.

Solubilities

In the presence of even 0.1M hydrofluoric acid, LaF_3 solubilities in nitric acid solutions are of the order of 5-10 mg/l. Although the solubility of LaF_3 is affected slightly by the presence of certain ions, such as cerium (IV) and zirconium (Reference 9), the solubility of this carrier compound is not an improtant variable in its use in process. The physico-chemical variables affecting its flocculation and separation characteristics are the determining factors of its efficiency as a carrier and are discussed in more detail later.

In the absence of hydrofluoric acid, LaF_3 has an appreciable solubility in nitric acid. This solubility makes it possible to rework LaF_3 by-product waste slurries by recycling to the product solution prior to the re-oxidation step of the LaF_3 by-product precipitation. The effect of nitric acid concentration on LaF_3 solubility is shown in the following tabulation (Reference 10):

TABLE I

SOLUBILITIES OF LaF_3 IN HNO_3

N HNO_3	Solubility La (mg/l)
1.05	474
2.53	920
3.94	1460
5.06	1420
6.15	1540
7.95	1700
8.09	1660

Oxidation in the Fluoride Cycle

In the absence of a strong oxidizing agent, it is difficult to maintain the product in the higher valence (VI) state during the LaF_3 by-product

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precipitation, due to reduction by ferrous iron entering the solution from hydrofluoric acid corrosion of the equipment surfaces. Although $Na_2Cr_2O_7$ is present in the $BiPO_4$ by-product effluent, it does not prevent the reduction of product. In addition, the recycled product returned to the LaF_3 by-product must be oxidized before the precipitation of the lanthanum. The use of 0.01M $KMnO_4$ has been found satisfactory for reoxidizing all product when the solution is digested for 30 minutes at 75 °C and provides a satisfactory oxidizing medium for maintaining the product at the higher valence (VI) state throughout the precipitation cycle (Reference 11).

It is believed that this treatment results in the deposition of a thin film of MnO_2 on the stainless steel surfaces, thereby inhibiting HF attack. If for any reason this MnO_2 film is destroyed, by extensive mid-operation equipment decontamination, for example, it can be restored by digesting (at 75 °C) and circulating a conditioning solution containing 1% HNO_3 and 0.3% $KMnO_4$, or by making "dummy" chemical runs.

Reduction in the Fluoride Cycle

The reduction of product in the effluent from the by-product LaF_3 separation is necessary to induce the complete carrying of product in the LaF_3 product precipitation. This reduction is accomplished at low temperatures (35 °C) by the use of 0.07M oxalic acid, when catalyzed by the presence of manganous ions. The LaF_3 by-product effluent contains approximately 0.01M $KMnO_4$ and this is rapidly reduced to the manganous state, providing the catalysis for the product reduction. Approximately 0.025M oxalic acid is required for the $KMnO_4$ reduction, leaving a residual excess of 0.045M oxalic acid for product reduction.

Metathesis of Lanthanum Fluoride

Although the LaF_3 product precipitation succeeds in separating the product in combination with a small weight of carrier, this precipitate is not convenient to forward to the isolation process where the product is to be separated from its carrier. Unfortunately the solubility of LaF_3 in HNO_3 is too low to permit the direct solution of this mixed LaF_3 - PuF_4 solid in any reasonable volume of solution (See Table I). This relatively insoluble lanthanum compound is thus converted to a highly soluble compound, lanthanum hydroxide, by metathesizing it with potassium hydroxide.

The conversion of LaF_3 - PuF_4 to the corresponding hydroxides is most simply and preferably carried out by digestion in 15% KOH for 90 minutes at 80 °C. The solid LaF_3 - PuF_4 is converted to the solid $La(OH)_3$ - $Pu(OH)_4$ without passing into solution during the process. The metathesized solid resembles the original LaF_3 - PuF_4 in appearance and characteristics, except for acid solubility. After separation of the metathesis cake, the fluoride anion concentration resulting from both the liberation of fluoride by the metathesis itself and the inclusion of some HF from the Cell E centrifuge heel is lowered by returning the cake to a dilute KOH (2%) wash solution and recentrifuging. The fluoride concentration must be reduced to a minimum to prevent LaF_3 reprecipitating when the $La(OH)_3$ cake is dissolved in acid (Reference 11). The wash is made 2% in KOH to prevent colloidal dispersion

[redacted]

(or peptization) of the $La(OH)_3$, which would result in a leak-through loss during the recentrifugation. The $La(OH)_3$ - $Pu(OH)_4$ cake is dissolved in sufficient HNO_3 to produce a 2N HNO_3 solution of $La(NO_3)_3$ and $Pu(NO_3)_4$.

It is desirable to maintain the iron concentration of the final product solution below 0.01M, in order to obtain low product peroxide solubilities in the isolation process. The straight KOH method of metathesis described above has demonstrated in the plant that it accomplishes a complete metathesis of LaF_3 in that no appreciable losses in HNO_3-insoluble residues have been obtained in the final solution. Also iron concentrations in the final solution have been below the tolerance level. Two other methods of metathesis are available, however, which might be used if either of the above objectives is not obtained by KOH metathesis. The first involves the use of 10% K_2CO_3 in combination with the 15% KOH (KOH-K_2CO_3 metathesis) in the metathesis step. This process is capable of reducing the HNO_3-insoluble residues in the final solution because of more complete metathesis (Reference 12). The metathesis losses are of the order of 3%, however, and the 15% KOH-10% K_2CO_3 must be reworked by the second addition of a small amount of extra lanthanum and recentrifugation. The second process is designed to provide better iron removal and involves solution of the LaF_3 - PuF_4 solid in 40% K_2CO_3 (straight K_2CO_3 method), precipitation of iron sulfide by the addition of $(NH_4)_2S$, and finally precipitation of $La(OH)_3$ - $Pu(OH)_4$ by making the K_2CO_3 solution 15% in KOH (References 11, 12). This process is extremely cumbersome and it has not been deemed necessary to use it in plant practice.

EFFECT OF VARIATIONS IN PROCESS CONDITIONS

Since the time of the separations plant start-up at Hanford, a series of process volume reductions has been developed and applied (References 13, 14). The main objective was a decrease in the process solution volume reaching the LaF_3 product precipitation step so that the product waste losses could be reduced to a low level (less than 1.5%) by increasing the lanthanum carrier concentration. The present standard solution volumes carried through the concentration process are only 60% of the start-up volumes. Other intermediate volumes have been and occasionally still are processed. As a means of identification, the start-up volume is taken as the reference volume and all other process volumes are referred to it as 40%, 60%, 80%, etc., of start-up volume.

Bismuth Phosphate By-Product

The effects of variations in process conditions in this step are the same as those previously described for the same operation in the second decontamination cycle (See Chapter VI). In addition, it has been demonstrated in the plant that variation in the process solution volume from 40% to 100% start-up volume has no effect on either by-product waste loss or decontamination factor (Reference 6).

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Lanthanum Fluoride By-Product

Reoxidation

Reoxidation of either part or all the total product entering the LaF_3 by-product precipitation is accomplished effectively by heating the 0.01M $KMnO_4$ solution at 75 °C for 30 minutes. No attempt has been made to study either optimum temperature or $KMnO_4$ concentration since the standard procedure is satisfactory (Reference 11).

$KMnO_4$ as a reoxidant and holding oxidant has been found to be the most satisfactory of all oxidants tested, including $Na_2Cr_2O_7$ or Pb_3O_4. $NaBiO_3$ is undesirable because of subsequent precipitation of bismuth in the fluoride product step. Various concentrations of $KMnO_4$ holding oxidant have been tried but 0.01M is the minimum which will provide residual oxidant through the by-product precipitation step (Reference 11).

Hydrofluoric Acid Concentration

The concentration of HF used in the LaF_3 by-product precipitation has been varied between 0.2N and 1.0N. The lower concentration, 0.2N HF, is most satisfactory because it causes less corrosion and the life of the holding oxidant is greatly increased. This maintains low by-product waste losses. Some laboratory information indicates higher decontamination factors at higher (0.5N) HF concentrations (Reference 11).

Type of Precipitation

When no recycled lanthanum is used, a preformed LaF_3 by-product precipitation is possible. Whatever part of the lanthanum is supplied by the recycle must be thrown down as a coformed precipitate because of the necessity of reoxidizing the recycled product before precipitation. All of the lanthanum may be supplied in the recycle or it may be supplemented by a preformed precipitate made with lanthanum added from the gallery. Fresh lanthanum is added as a 10% solution of $La(NO_3)_2 \cdot (NH_4NO_3)_2 \cdot 4H_2O$ containing 10% HNO_3 (to prevent hydrolysis) over a period of 20 minutes. Neither the lanthanum concentration nor the rate of addition is critical. No differences in either decontamination factor or by-product waste losses result from the use of any one of these procedures.

Lanthanum Concentration

When the equivalent of slightly more than two Cell E charges of lanthanum are recycled from two isolation supernatants to any single by-product, 720 mg/l (9.0 lbs. total lanthanum) are precipitated in a single by-product strike. When no recycle is returned, 500 mg/l (6.0 lbs. total lanthanum) are precipitated. No differences in either decontamination factor or waste losses result.

In addition to the recycling of the lanthanum from isolation supernatants to a regular production run, the lanthanum may be returned to a synthetic $BiPO_4$ by-product effluent made up in the LaF_3 by-product precipitator. In these "master" recycle runs, as much as from 20 to 31 lbs. of lanthanum have

been recycled into a 40-50% start-up volume solution, producing a lanthanum precipitation concentration of 2300 and 3500 mg/l respectively. In such cases the centrifuge is skimmed to only 30 to 40 gallons. The by-product waste losses for these large cakes range from 2.5 to 3.5%.

Strike Temperature

The standard procedure involves precipitation and digestion at 35 °C. Although a higher temperature would provide better flocculation, the increased corrosion and $KMnO_4$ destruction would tend to cause higher waste losses.

Centrifugation

Since LaF_3 is a finely divided gelatinous material, its separation is extremely difficult. Standard practice requires centrifugation at twice the bowl speed (1740 instead of 870 rev./min.) used in the separation of $BiPO_4$. In addition, as slow a rate of throughput as possible (70 lbs/minute) is used to provide a maximum retention time in the bowl (8 minutes). Either slower centrifugation speed or faster throughput rate would result in lower decontamination factors.

Cake Washing

The product loss in the by-product waste is influenced by the efficiency of the cake-washing procedure. For process volumes from 100% start-up down to 60% volume, the concentration of product in the skimmed centrifuge heel is low enough such that a straight displacement wash from the precipitator through the centrifuge is efficient enough to maintain the waste losses at 0.7 - 0.8%. For process volumes below 60% start-up, however, the centrifuge heel contains enough product that a two-batch slurry wash from the precipitator to the centrifuge is necessary to maintain the waste losses at these levels. A two-batch gallery to centrifuge slurry wash, using the sprays to cut the cake off the bowl walls, results in still lower waste losses (0.5 - 0.6%) but contributes too large a fraction of colloidally dispersed LaF_3 fines to the effluent, causing higher waste losses in the LaF_3 product precipitation step.

The cake wash solution itself is a 6% HNO_3 solution. By adding it to the precipitator first and circulating it through the distributor, undesirable hold-up of oxidized product is eliminated and the HF that it picks up helps to prevent the dissolving of any LaF_3 in the bowl. Water alone would cause peptization of the LaF_3 in the bowl.

The volume of the LaF_3 cake is approximately 1 gallon/pound of lanthanum.

Lanthanum Fluoride Product

Reduction

The concentration of the oxalic acid used for the reduction of product is most likely not the minimum that could be used. Although only 0.025M is required to reduce the 0.01M $KMnO_4$ used in the by-product re-oxidation to the

manganous state, there are varying amounts of $KMnO_4$ returned to the by-product step in the recycled isolation supernatants. Consequently, an initial concentration of 0.07M oxalic acid is used, rather than the 0.06M used in preliminary process studies, providing a final excess of approximately 0.045M oxalic acid. No information on the use of lower concentration is available. Laboratory data indicate that the standard reduction time of 1 hour, while providing very little safety factor, is adequate (Reference 11).

Hydrofluoric Acid Concentration

In carrying out the LaF_3 produce precipitation, it is desirable to use as high a concentration of HF as possible to assure the best precipitation of LaF_3 and still not cause excessive corrosion of process equipment. Here again there are many discrepancies between laboratory and plant information, primarily because the separation of LaF_3 appears to be a flocculation-centrifugation problem specifically influenced by the scale of operation and type of equipment employed.

Laboratory data indicate that product carrying and separation are unaffected in the range of from 0.5 to 0.3M HF, but are decreased at lower concentration (Reference 15). Other laboratory data indicate that product separation efficiency is increased perhaps 30 or 40% by increasing the HF concentration to 1-2N (Reference 16). The latter has not been substantiated in the plant, however, and the corrosion rates are objectionably high at such HF concentrations. The present standard of 0.5N HF is considered to be the safe optimum for product LaF_3 separation and tolerance corrosion rates.

Lanthanum Concentration

Laboratory studies have shown that the lanthanum concentration per strike can be reduced as low as 25 mg/l in the LaF_3 product precipitation without affecting product carrying or separation (Reference 15). These are the results of tests carried out under ideal conditions, however. In actual plant trials tests have been carried out at 33, 50, 80, and 125 mg/l lanthanum concentration (Reference 17). Although there are indications that higher lanthanum concentrations would result in still lower product waste losses, 125 mg/l has been chosen as the practical optimum from both volume reduction and cake size considerations. The concentration of lanthanum is obtained by adding 2.0 lbs. of lanthanum to each of two product strikes in the 60% volume process.

The effect of lanthanum concentration on the product waste losses is shown in the summary of plant data given in the following table:

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TABLE II

Effect of Lanthanum Concentration on LaF_3 Product Waste Losses

No. La Strikes	La Conc. mg/l	% Product Losses in Effluent		
		Cent. #1	Cent. #2	Cent. #3
3	33	20	8	1.5
2	50	12	2	--
2	80	8	1.6	--
2	125	6	1.2	--

Methods of Precipitation

Since HF is already present in the LaF_3 by-product effluent, a preformed LaF_3 product strike is necessary. Standard practice involves adding the required amount of HF to increase the concentration to 0.5N, adding the lanthanum as a 1.5% $La(NO_3)_3 \cdot (NH_4NO_3)_2 \cdot 4H_2O$ salt solution containing 1% HNO_3 (to prevent hydrolysis) over a period of 20 minutes, digesting for 1 hour at 35 °C, and centrifuging. This procedure is repeated for the second strike, which is necessary to obtain more complete separation of the product leaking through the first centrifugation, carried by the incompletely separated LaF_3. Little variation in this procedure, beyond lanthanum strike concentration and number of strikes, has been tried, but such things as adding the lanthanum rapidly without agitation to the heel from the first strike before return of the effluent, etc., have separately appeared to have no appreciable effect on LaF_3 flocculation.

Agitation

Contradictory laboratory information exists that increased agitation during the strike and digestion either hinder or aid flocculation of LaF_3 (Reference 8, 16). No effects have been observed in the plant where sparging instead of mechanical agitation, slow settling periods without agitation, and extended agitation have been tried. At present, mechanical agitation throughout the strike and digestion are considered desirable, to insure uniform precipitation and carrying of product by LaF_3.

Nitric Acid Concentration

No plant information is available on the effect of HNO_3 concentration on the separation and carrying by LaF_3, since the standard precipitation is and has always been carried out at approximately 1.0N HNO_3. Laboratory information shows, however, that higher HNO_3 concentration causes poor separation of LaF_3 (Reference 16). Lower HNO_3 concentrations favor better separation (Reference 18). Neutralization of the HNO_3 in the LaF_3 by-product effluent before

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the product strike has been demonstrated in the laboratory to improve the separation of product-carrying LaF_3. There is some question, however, that the resultant increased salt content would not counter-act this favorable effect in actual plant operation (Reference 18).

Ionic Interferences

Aluminum and magnesium interfere greatly with the carrying of product by LaF_3. Increased concentrations of such salts as NH_4NO_3 and $NaNO_3$ cause poor flocculation or separation (Reference 16), as do increased amounts of chromium and manganese (Reference 5). Calcium does not appear to have any effect (Reference 16).

Temperature

As in the LaF_3 by-product, strike and digestion temperatures higher than the standard 35 °C favor increased flocculation of product-carrying LaF_3 (Reference 16). The effects are not pronounced, however, except for temperatures in the range of 50-75 °C which are objectionable because of accelerated corrosion of equipment by HF.

Centrifugation

The actual separation of the LaF_3 precipitate is the controlling factor in the LaF_3 product precipitation, rather than the carrying of product by LaF_3, since complete carrying is relatively easily achieved. Although the degree of flocculation obtained in the precipitation itself is important, the conditions of centrifugation are none the less important. Standard practice requires centrifugation at top bowl speeds of 1740 rev./min. (approximately 1700 X gravitational/force) with throughput rates as low as practicable, approximately 70 lbs/minute (8 minutes retention time). Lower bowl speeds or higher throughput rates result in increased product-lanthanum leak-through or higher waste losses.

Even under these conditions, however, 5 to 10% of the LaF_3 passes through the centrifuge into the effluent, due to the presence of LaF_3 fines in the precipitation slurry and/or deterioration of the flocculated LaF_3 during the jetting and centrifugation. It is thus necessary to make a second strike in the product precipitation in an effort to reflocculate and redistribute these product-carrying fines, in order to obtain satisfactory separation of product.

Carry-Over of Fines from By-Product

The LaF_3 product precipitation step is not entirely independent of the LaF_3 by-product precipitation. If the separation of the LaF_3 by-product precipitate is incomplete, either because of poor centrifugation or peptization in cake washing, LaF_3 fines carry over into the effluent from which the LaF_3 product precipitation is made. When the reduction step is carried out, a large proportion of the reduced product can then be picked up or carried by these fines. When the regular LaF_3 product strike is made, these fines are not flocculated to a great extent and thus become the source of high product leak-through in the subsequent centrifugations.

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Crystalline Lanthanum Fluoride

In an attempt to eliminate both the carry-over of LaF_3 fine from the by-product and the production of a highly dispersed LaF_3 product carrier, tests have been made with a so-called "crystalline" LaF_3. These were carried out by preparing a reagent slurry of solid sodium lanthanum sulfate crystals. This slurry, when added to both the by-product and product process solutions, was metathesized to LaF_3 by the HF in the solutions, and produced large heavy crystals that were more easily separated than the regular flocculent LaF_3 (Reference 7). The decrease in fines formation or improvement in separation efficiency did not prove to be great enough, however, to justify the use of the more involved preparation.

Cake Washing

It is desirable to wash the LaF_3 product cake to decrease the fluoride, iron, and manganese content of the centrifuge heel passing on to the metathesis step. Standard practice involves adding 500 lbs. of a 6% HNO_3 wash solution to the precipitator, circulating to flush out any LaF_3 hold-up, and jetting to the skimmed centrifuge in one continuous displacement wash. This results in an increase in the product waste loss of only 0.1 to 0.2%.

A two-batch slurry wash, similar to that used in the LaF_3 by-product cake wash, has been used but is much more time consuming and occasionally increases the product waste losses by as much as 1%. Although this results in less MnO_2 appearing in the final metathesized product solution, the presence of the latter does not involve any process disadvantages that would warrant the use of this type of wash.

The wash solution is 6% HNO_3 and is added to the precipitator to pick up fluoride in order to prevent solution or peptization of the LaF_3 product cake.

Metathesis

Conversion of the Product Lanthanum Fluoride to Hydroxide

The conversion of the LaF_3 - PuF_4 precipitate obtained from the LaF_3 product precipitation is normally carried out by digestion in 250 gallons of 15% KOH for 90 minutes at 80 °C. At KOH concentrations lower than 15% or at temperatures lower than 75 °C, the metathesis conversion is less rapid. The volume scale (or lanthanum concentration) apparently is not critical (Reference 11). Although 60-minute digestion has proved satisfactory in operation, the 90-minute heating has been retained to assure the most complete conversion possible.

The standard method of metathesis quoted above is liable to produce appreciable amounts of HNO_3-insoluble material when used in new equipment start-up. The presence of metallic impurities (such as filings, welding slag, etc.) in new equipment either themselves directly contribute to the insolubles or influence the incomplete metathesis of LaF_3 in such a way that objectionable

amounts of product-carrying insoluble solids are obtained in the final $La(OH)_3$ - $Pu(OH)_4$ cake solution.

An alternate method of metathesis may be used in such a case (Reference 19). This method involves the use of 10% K_2CO_3 in company with the 15% KOH in the metathesis proper. This method tends to solubilize the objectionable impurities to the extent that they are discarded in the metathesis waste and a more complete solution of the final cake can be obtained. The metathesis waste loss in the procedure is of the order of 2-5%, however, and it is necessary to rework the waste by adding approximately 30 mg/liter of extra lanthanum and recentrifuging (Reference 11, 12).

Still a third metathesis method may be used, primarily to reduce the iron content of the final product solution. This method, the straight K_2CO_3 - $(NH_4)_2$ S procedure, has been outlined previously in this chapter and does not warrant a more extensive discussion at this point (Reference 11, 12).

The effects of process impurities on the standard metathesis are not very well known. There is some laboratory information available indicating that the presence of chromium has a harmful effect on both the carrying of product and separation of $La(OH)_3$ (Reference 5). This has not been borne out in the plant to date, however. Manganese and iron appear to improve the separation of $La(OH)_3$ after metathesis.

Metathesis Wash

The function of the metathesis wash has been described in the previous section. The tank wash is standard practice rather than a bowl wash because it has been found difficult to slurry the precipitate completely down from the bowl walls and obtain efficient rinsing out of the fluoride ions. The wash liquor itself is made 2% in KOH because this is the minimum concentration of electrolyte which will satisfactorily inhibit peptization and product loss in the subsequent centrifugation.

The major process variable involved in the operation of this step in the plant is the actual removal of the cake from the centrifuge bowl back to the wash tank. It has been found necessary to use a high-pressure water spray (300 lbs/sq.in.) on the two lower baffle sections to strip the cake off the lower walls completely and break up the lumps into a slurry that can be jetted satisfactorily. Without the use of these high-pressure sprays, it is necessary to employ a more extended procedure involving the use of the centrifuge plows and protracted slurrying up and down the walls of the bowl.

Centrifugation

In the centrifugation of the 15% KOH metathesis slurry a throughput rate of 12 to 15 lbs/minute, with the centrifuge operating at top speed, 1800 rev./min., is necessary to obtain the lowest product waste losses. The $La(OH)_3$ - $Pu(OH)_4$ precipitate resembles the LaF_3 - PuF_4 precipitate very closely and is difficult to separate. The through-put rate may be doubled, however, without increasing the waste losses any more than by a factor of 2.

The centrifugation of the 2% KOH wash slurry is also carried out at 1800 rev./min., but the through-put rate of 25 lbs/minute produces satisfactorily low waste losses, even though the rate is twice that of the 15% KOH centrifugation. This is believed to be due to the lower specific gravity of the second KOH solution and the absence of such interfering ions as chromium, which is largely discarded in thr first waste. A two-fold decrease in through-put rate does not decrease the metathesis wash waste losses by more than 0.1%.

Although the volume of the $La(OH)_3$ cake is roughly 1 gallon/pound of lanthanum the distribution of cake is such that a 4.0-lb lanthanum cake can be skimmed to a 3-gallon heel.

Dissolution of Washed Metathesis Cake

A quantity of HNO_3 must be used which is sufficient (1) to neutralize the 2% KOH heel in the centrifuge bowl, (2) to convert $La(OH)_3$ and $Pu(OH)_4$ to the corresponding nitrates, and (3) to make the resulting solution 2.0N in HNO_3. The final solution has a volume of 7.3 gallons and contains approximately 66 gm/l of lanthanum. Since the volume of acid required to effect this solution is small, extra precautions must be taken to insure adequate contact with all of the cake in the bowl. Extensive slurrying and use of the plows are necessary to dissolve the entire cake.

Final HNO_3 concentrations in the range of 1.8 to 2.2N are desirable. If the HNO_3 concentration is below 0.5N, there is danger of hydrolysis or formation of the "abnormal" (polymerized) form of product (See Chapter VIII). If the acidity is greater than 2.2N, it is necessary to adjust downward by neutralization, in order to prevent increases in the product peroxide solubilities in the isolation step.

A small water flush of the centrifuge and transfer lines after cake solution is necessary in order to minimize the hold-up of product in the centrifuge heel, since this heel is recycled to the next charge passing through the metathesis operation. It is desirable that the total volume of the final product solution be kept at a minimum, since the amount of product to be recycled from the isolation process is a direct function of this solution volume.

The product solution usually contains varying amounts of HNO_3-insolubles, largely MnO_2, but also small amounts of Fe, Ni, Cr, La, Bi, Si, Ca, etc. If metathesis has not been complete, the residue will contain LaF_3 and significant amounts of product. In general, the soluble iron content is less than 0.01M, which is tolerance level in the isolation step.

RECOVERY OF PRODUCT FROM WASTES

It has not yet been necessary to recover product from the LaF_3 by-product wastes in the Concentration Process. Reworking of the $BiPO_4$ by-product, LaF_3 product, and metathesis wastes, however, has been necessary occasionally.

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The theories and procedures involved in the reworking of such wastes are outlined at this point. The actual plant procedures are not quoted in detail, since the standard process procedures can be applied with certain revisions if the basis for the reworking procedures is known.

Bismuth Phosphate By-Product

The recovery of product from $BiPO_4$ by-product waste solution of the concentration process may be accomplished by methods similar to those used for the second cycle by-product (See Chapter VI). The causes of high-product waste losses will probably fall into two main categories: (1) the existence of appreciable amounts of the difficulty soluble or "beta" form of $BiPO_4$ in the second-cycle product cake solution from the Canyon Building, or (2) improper execution of the oxidation step in the Concentration Building.

In the first case, the by-product cake will probably be incompletely dissolved when the standard amount of 60% HNO_3 (1400 lbs.) is used. Complete solution of the undissolved $BiPO_4$ should first be effected by the addition of extra HNO_3. Solution should be complete when 2600 lbs. of HNO_3 has been added. The standard 7.5N HNO_3, 0.013M $NaBiO_3$, 0.033N $Na_2Cr_2O_7$ oxidation should then be carried out in the solution tank (by temporary reagent line jumper installation) or, preferably, in the precipitator. The standard procedure for the 100% start-up volume by-product process is then carried out with three equal-weight lanthanum strikes and centrifugation in the LaF_3 product precipitation.

In the case of improper execution of the original oxidation step, solution of the $BiPO_4$ by-product cake will most probably be complete in the regular volume of HNO_3. In this event, a larger volume oxidation process is repeated, preferably in the precipitator, and a normal by-product precipitation also repeated.

For the unusual case of very difficultly soluble by-product cakes requiring more than 2600 lbs. of 60% HNO_3, a special reworking procedure has been developed in the laboratory (Reference 20). This method involves a by-product reprecipitation at as high as 2.5N HNO_3 using 0.3M di-ammonium acid phosphate, to neutralize partially the extra acid at the time of precipitation, and NaOH, to complete the neutralization of the extra acid after precipitation. The resultant high salt concentrations may cause as high as 20% product waste losses in the LaF_3 product precipitation, however.

Lanthanum Fluoride By-Product

A procedure has been developed in the laboratory for reworking the LaF_3 by-product wastes by recycling the waste slurry into the $BiPO_4$ by-product effluent of the succeeding charge prior to the LaF_3 by-product oxidation step (Reference 10). This procedure is based on the solubility of LaF_3 being high enough in the 1.3N HNO_3 process solution, approximately 550 mg/l lanthanum (see Table I), to redissolve completely the LaF_3 precipitate and thus permit product reoxidation as in the recycle process. This procedure is based on the

use of the 100% start-up volume process, however, and it is impossible to dissolve all of the LaF_3 in the 60% start-up volume process.

It is recommended, accordingly, that special synthetic 100% volume $BiPO_4$ by-product effluent (1800 gallons of 1.8N HNO_3) be made up in the LaF_3 by-product precipitator. The waste slurry is then returned to the precipitator, the solution made 0.01M in $KMnO_4$, and the standard LaF_3 by-product oxidation and precipitation carried out. The effluent is processed through the LaF_3 product precipitation and metathesis as a 100% volume "extra" run.

Lanthanum Fluoride Product

If the LaF_3 product waste is between 1.5% and 3%, recentrifugation alone will reduce the loss to 1.5% or lower. Ordinarily a decrease in waste loss by a factor of approximately 2 is obtained by straight recentrifugation. It is usually desirable to proceed with the main LaF_3 product cake removal from the centrifuge, then recentrifuge the waste, and leave the small amount of product LaF_3 removed in the running bowl to be combined with the succeeding charge.

If the waste loss is of the order of 5% to 10%, the use of 100% additional lanthanum in a single strike to form an "extra" run is recommended. A decrease in the waste loss by a factor of approximately 6 may be expected in the procedure. The addition of any such extra lanthanum makes it desirable to form an extra run and the full amount of lanthanum is best used for safety.

Metathesis

If the 15% metathesis waste loss is between 1% and 2%, slow recentrifugation will decrease the loss to less than 1%. If it is higher than 2%, inspection of the waste sample for presence of suspended solids should be made. Recentrifugation should be carried out if suspended slids are present, skimming carefully to a slightly larger centrifuge heel. If no solids are visible, the waste should be digested with 30 mg/l added lanthanum and then recentrifuged.

The same treatment should be applied to the 2% KOH wastes, first making certain that the wash solution is at least 2% KOH. Absence of the KOH will cause peptization of the $La(OH)_3$ and resultant centrifugation leak-through.

EQUIPMENT

The Concentration Building is divided into six operating areas termed Cells A to F inclusive. The building layout and process piping are shown in Figure 60 of Chapter II of this manual. All cell floors except the deep well

of Cell C are at ground level. Cells A to E are contained in a large rectangular canyon as a single unit, being separated only by concrete partitions that extend less than half of the distance to the roof. Gallery tiers run along one side of this canyon. The operating gallery containing the reagent make up tanks is on the third floor so that gravity flow of chemical feeds to the process vessels is obtained. The pipe gallery, sampling labyrinths, and extrances to the centrifuge pits are on the second floor. The first floor contains chemical storage, offices, air conditioning, and change-room facilities. Cell F is a two-story adjunct to the end of the building and includes an operating gallery elevated above the process equipment which is located at ground level.

The radiation levels are low enough so that only 12 inches of concrete shielding are used to separate process equipment from the working galleries. Piping connections are made in a normal manner with standard bolted flanges, without the remotely operated connectors used in the Canyon Building. All piping from the galleries pass into the cells through grouted slots in the main shielding walls and process piping between cells. All of the equipment is readily accessible for repair or inspection. Although operation is controlled remotely, direct rather than remote maintenance is used.

Except for Cell F, the process vessels and instrumentation in the Concentration Building are very nearly identical to those in the Canyon Building. The lower level of activity, however, has made possible a simpler and more normal arrangement. Four of the cells, A, B, D, and E, are essentially identical and contain a precipitator, centrifuge, catch tank and solution tank (two in B). The centrifuges are placed on platforms at second floor level, 13 feet above the cell floors, allowing gravity flow to the catch tanks.

Cell A

Cell A is used for the bismuth phosphate by-product precipitation. Its precipitator (A-1) receives the oxidized product solution from the Cell C oxidation tank (C-4) and includes return lines from the catch tank (A-3) and solution tank (A-4), as well as a discharge to the centrifuge (A-2). The catch tank has discharge lines going to the Cell B precipitator (B-1) and the Cell D precipitator (D-1) for the effluent to pass on to the lanthanum fluoride by-product and the Cell E precipitator (E-1) in the event that the LaF_3 by-product is ever deliberately omitted. The solution tank (A-4) has a discharge out of the cell to the waste neutralization tank (C-8) only. The centrifuge (A-2) has a drain to the catch tank (A-3) and a jet discharge to the solution tank (A-4). The layout of Cell A equipment and piping is shown in Figure 1.

Cell B

Cell B is a spare cell for the bismuth phosphate or LaF_3 by-products or the lanthanum fluoride product. Its precipitator (B-1) has discharge lines

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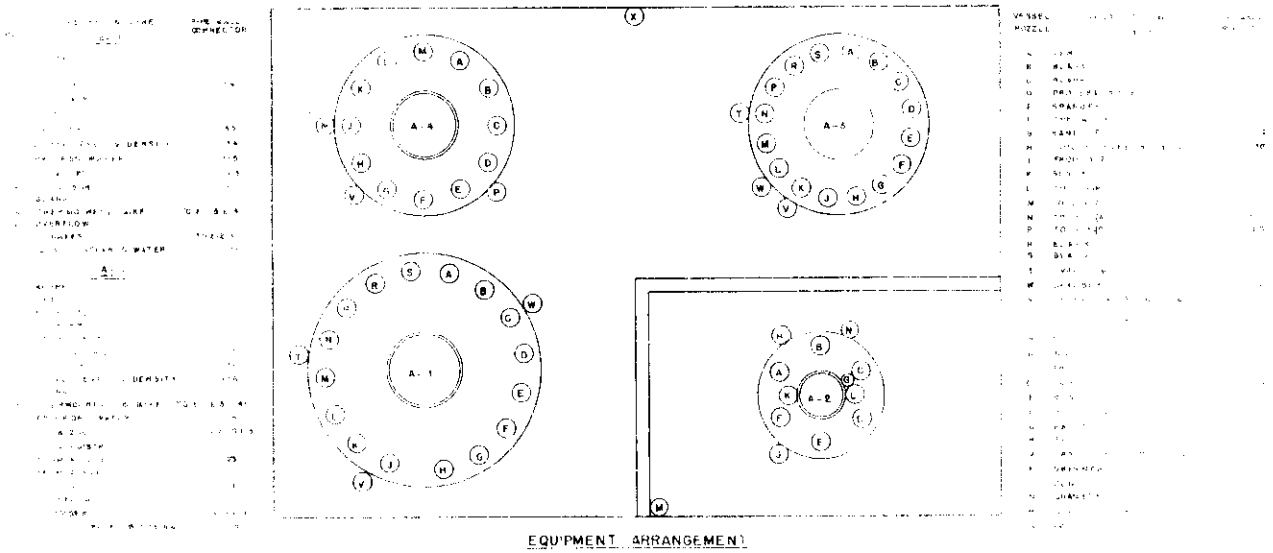
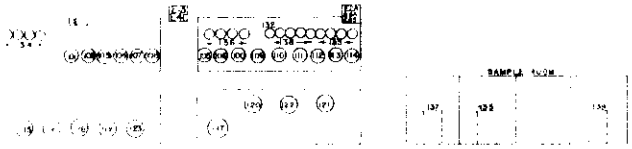
FIGURE

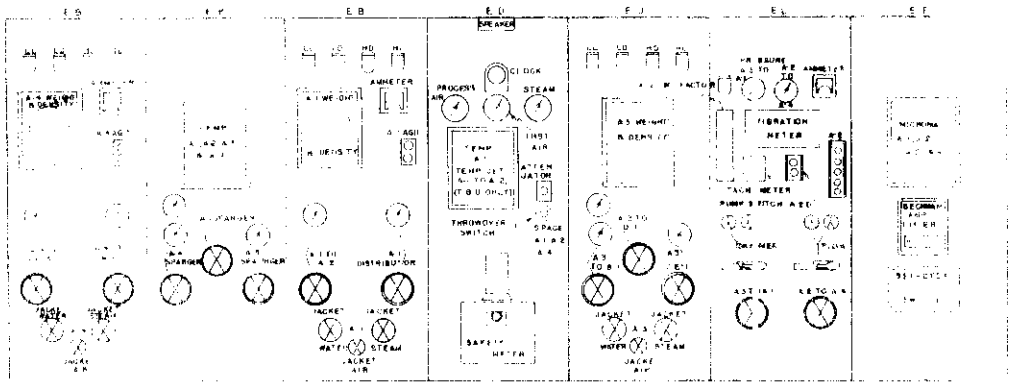
LAYOUT OF CELL A- CONCENTRATION (224) BUILDING



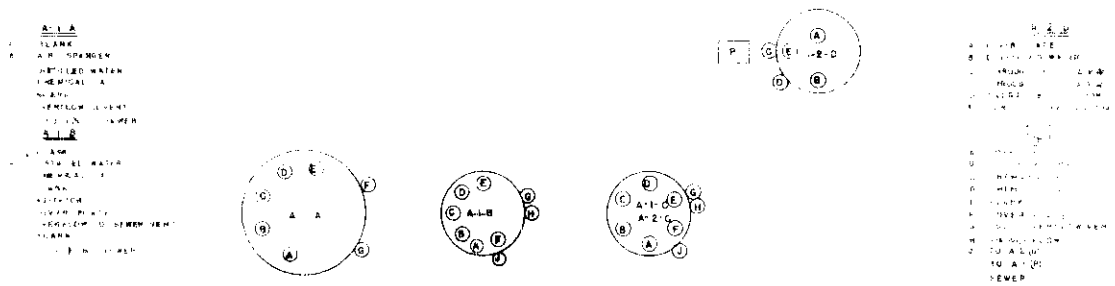
EQUIPMENT ARRANGEMENT



PIPE GALLERY CONNECTORS



PANEL BOARD



CONTROL GALLERY WEIGH TANKS

LEGEND

EQUIPMENT WITHOUT MOTOR DRIVE
EQUIPMENT WITH MOTOR DRIVE
ORDINARY VALVE
BALL INDICATING PRESSURE DOWNSTREAM FROM BALL VALVE
NOZZLE CONNECTION ON EQUIPMENT
FLANGE CONNECTION ON WALL
ELECTRICAL CONNECTOR ON WALL
PUMP
BUBBLER OR AIR SUPPLY TO MANOMETERS
LL LOW PRESSURE LEG -- LIQUID LEVEL SYSTEM
HL HIGH PRESSURE LEG " " "
LD LOW PRESSURE LEG -- DENSITY SYSTEM
HD HIGH PRESSURE LEG " " "
BALL VALVE
" " & CORRESPONDING BALL

REFERENCE DRAWINGS

W-71602 ARRANGEMENT & PIPING 224 BLDG. 3RD FLOOR PLANS A-1
W-71603 " " 224 " END " A-2
W-74626 BASE BOARD ARRANGEMENT & PIPING
W-72917 CELL ARRANGEMENT & PIPING SHEET 1.
W-74672 " " " SHEET 2.
W-75580 FIRST FLOOR POWER PLAN 224 BLDG.
W-74685 ELECTRICAL DETAILS SHEET E.

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from the Cell C oxidation tank (C-4) for receiving oxidized product solution, from the Cell A catch tank (A-3) for receiving bismuth phosphate by-product effluent in the event of a Cell D failure, and from the Cell D catch tank (D-3) for receiving lanthanum fluoride by-product effluent in the event of a Cell E failure. In addition, the precipitator includes return lines from the catch tank (B-3) and solution tank (B-4), as well as a discharge line to the centrifuge (B-2).

The centrifuge (B-2) has a drain line to the catch tank (B-3) and jet discharge lines to the solution tank (B-4) and metathesis tank (F-1), the latter to provide for the use of Cell B as a lanthanum fluoride product cell. The catch tank (B-3) has discharge lines out of the cell to the Cell D precipitator (D-1), to send the effluent into the lanthanum fluoride by-product, and to the waste neutralization tank (C-8), to discard lanthanum fluoride product waste. The solution tank has discharge lines to waste (C-8) and to the Cell D precipitator (D-1). A spare solution tank (F-6) stands isolated from other cell vessels in Cell B, having been originally provided for alternate process work. The layout of Cell B equipment and piping is shown in Figure 2.

Cell C

Cell C contains a large waste neutralization tank (C-8) which can receive discharge from A-4, B-3, B-4, D-4, E-3, F-7, F-8, and C-7. In turn it has discharge lines to the waste settling tank (361) and to the Canyon Building (17-3) for returning high product and high radiation wastes for possible reworking.

A regular-sized solution tank (C-4) is located in a deep sump in the right half of Cell C. This sump is 19 feet lower than the other cells, to permit complete drainage of product solution transfers from the Canyon Building. C-4 can receive product solution from either 17-4 or 19-4 via the pipe tunnel from the Canyon Building. A recovery discharge from the sump tank (C-7) is also provided. In turn, it has discharge lines to A-1, B-1, and D-1, for transferring the product solution after primary oxidation, and to 19-4 in the Canyon Building for returning "hot" solutions for further decontamination.

The deep well sump tank (C-7) catches the floor drainage from all the cells and also the discharge from a floor sump in the deep well itself. It discharges to either the waste neutralization tank (C-8) or to the cooling water sewer.

The layout of Cell C equipment and piping is shown in Figure 3.

Cell D

Cell D is normally the lanthanum fluoride by-product cell. Its precipitator (D-1) can receive oxidized product solution directly from C-4, from the bismuth phosphate by-product catch tank (A-3), which is normal, or from the

[redacted]

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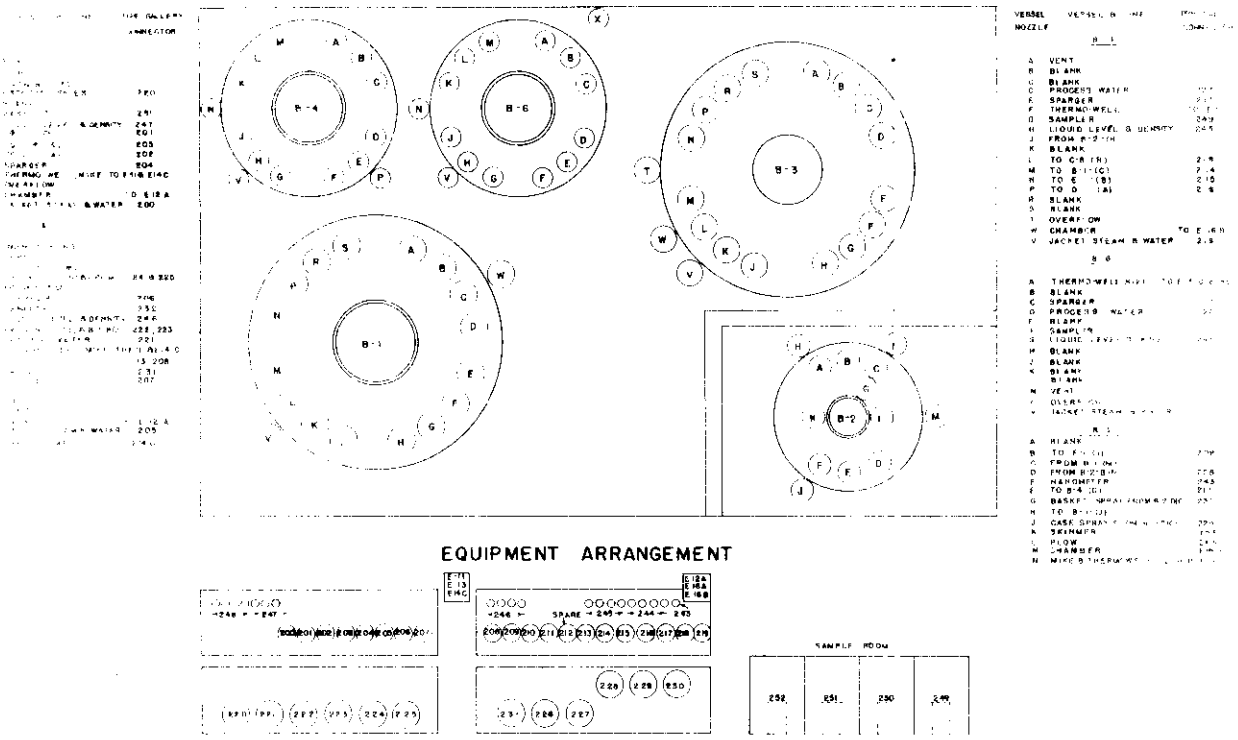
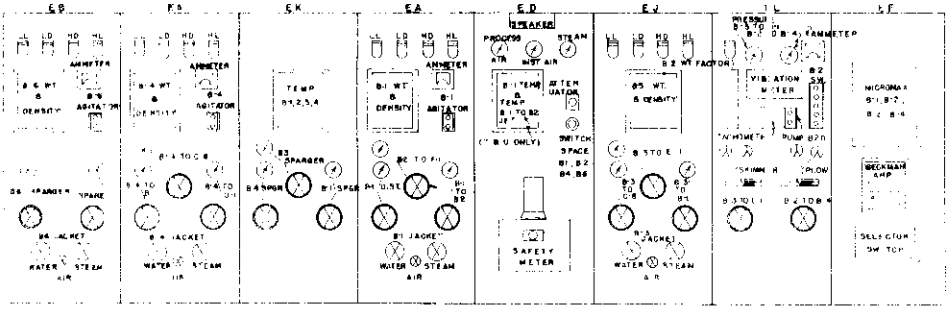
FIGURE 2

LAYOUT OF CELL B - CONCENTRATION (224) BLDG.

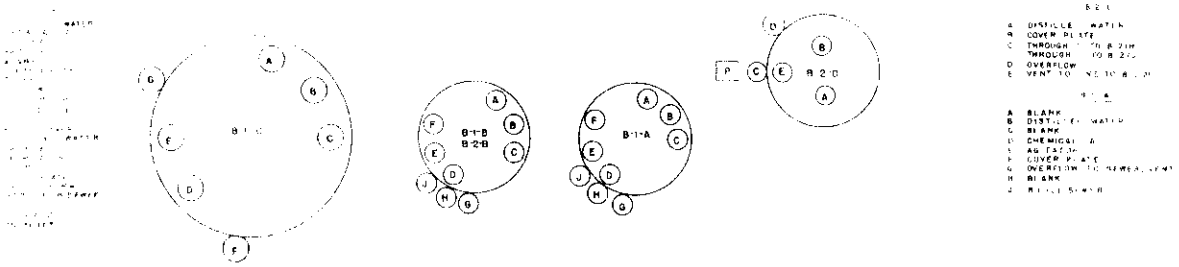


EQUIPMENT ARRANGEMENT

PIPE GALLERY CONNECTORS



PANEL BOARD



CONTROL GALLERY WEIGH TANKS

[redacted]

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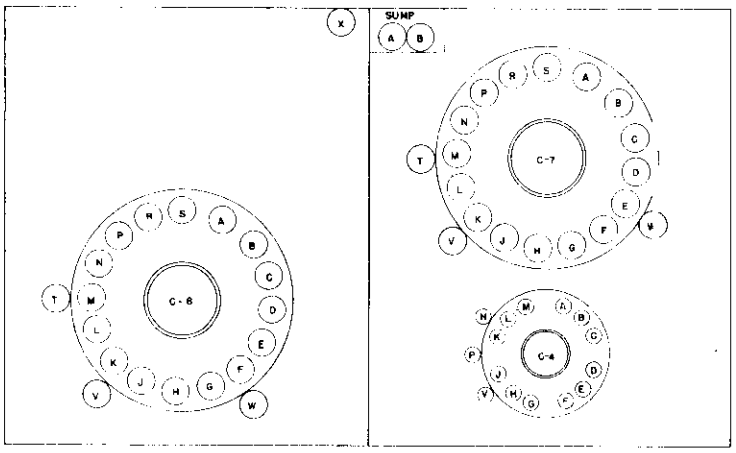
FIGURE 3

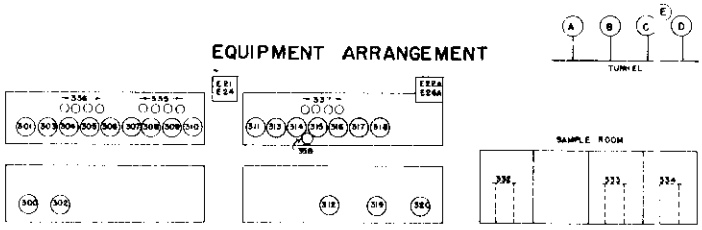
LAYOUT OF CELL C-CONCENTRATION (224) BUILDING

VESSEL NOZZLE	VESSEL & LINE	PIPE GALL. CONNECTOR
	C-8	
A	VENT	
B	FROM C-4-(J)	
C	FROM E-3-(M)	
D	FROM F-3-(M)	
E	FROM F-2-(B)	
F	FROM D-4-(K)	
G	FROM A-4-(K)	
H	SAMPLER	332
J	FROM C-8-A & PROCESS WATER	300,302
K	THERMOCOUPLE, MIKE	E21, E24
L	LIQUID LEVEL & DENSITY	335
M	TO 221-(17-5) VIA TUNNEL(8)	308
N	TO 241 BLDG.	303
P	SPARGER	303
R	FROM B-5-(L)	
S	FROM C-7-(N)	
T	OVERFLOW	
W	CHAMBER	E22A
V	JACKET STEAM & WATER	304
	TUNNEL	
A	FROM 221-17-4 TO C-4-(B)	
B	FROM C-8-(M) TO 221-17-3	
C	FROM 221-19-4 TO C-4-(B)	
D	FROM C-8-(M) TO 221-19-4	
E	DRAIN TO C-4-(F)	
	SUMP	
A	TO C-7-(S)	301
B	MANOMETER	338
X	SPARE MIKE	E24

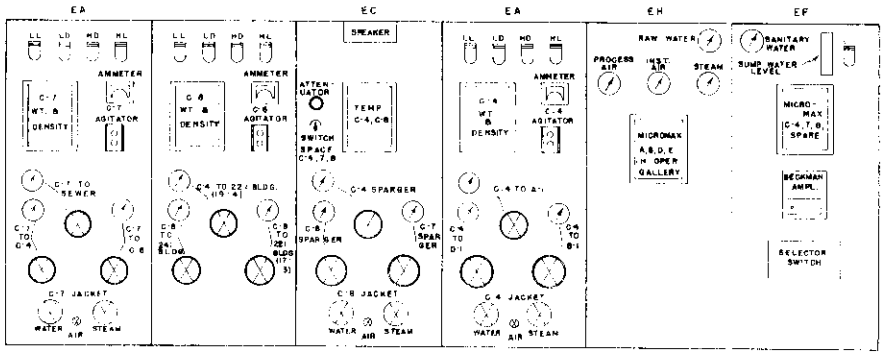


VESSEL NOZZLE	VESSEL & LINE	PIPE GALL. CONNECTOR
	C-7	
A	VENT	
B	BLANK	
C	BLANK	
D	BLANK	
E	THERMOCOUPLE, MIKE	E21, E24
F	SAMPLER	334
G	BLANK	
H	BLANK	
J	224 DRAIN	
K	SPARGER	307
L	LIQUID LEVEL & DENSITY	336
M	TO C-4-(M)	309
N	TO C-8-(S	310
P	TO SEWER	311
R	PROCESS WATER	312
S	FROM SUMP	
T	OVERFLOW	
W	CHAMBER	E22A
V	JACKET STEAM & WATER	305
	C-4	
A	FROM C-4 B (J) & PROCESS WATER	300, 320
B	SPARGER	313
C	SAMPLER	335
D	LIQUID LEVEL & DENSITY	337
E	THERMOCOUPLE, MIKE	E21, E24
F	FROM TUNNEL TROUGH	
G	FROM 221-17-4 VIA TUNNEL (A)	
G	FROM 221-19-4 VIA TUNNEL (C)	
H	TO 221-19-4, VIA TUNNEL (D)	318
J	TO B-1-(E	316
K	TO A-1-(E)	317
L	TO D-1-(R)	314
M	FROM C-7-M, VENT	
N	CHAMBER	
P	OVERFLOW	
V	JACKET STEAM & WATER	315

EQUIPMENT ARRANGEMENT

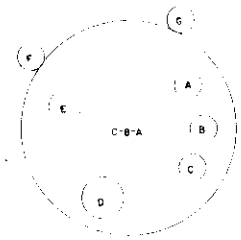


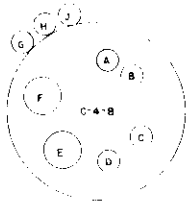
PIPE GALLERY CONNECTORS



PANEL BOARD

C-8-A
- A AIR SPARGER
- B PROCESS WATER
- C CHEMICAL Q
- D BLANK
- E BLANK
- F OVERFLOW TO SEWER, VENT
- G TO C-8-(J) & SEWER





C-4-B
- A BLANK
- B DISTILLED WATER
- C BLANK
- D BLANK
- E AGITATOR
- F BLANK
- G OVERFLOW TO SEWER, VENT
- H BLANK
- J TO C-4-(A) & TO SEWER

CONTROL GALLERY WEIGH TANKS

LEGEND

- EQUIPMENT WITHOUT MOTOR DRIVE
- EQUIPMENT WITH MOTOR DRIVE
- ORDINARY VALVE
- BABB VALVE
- BABB INDICATING PRESSURE DOWNSTREAM FROM BABB VALVE
- NOZZLE CONNECTOR ON EQUIPMENT
- FLANGE CONNECTOR ON WALL
- ELECTRICAL CONNECTOR ON WALL
- PUMP
- BUBBLER OR AIR SUPPLY TO MANOMETER SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, " " "
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, " "

REFERENCE DRAWINGS

W 71802	ARRANGEMENT & PIPING 224 BLDG 330 FLOOR CELLS A TO E	
W 71803	" " " " 224 " 240	A TO E
W 74388	BABB BOARD ARRANGEMENT & PIPING	
W 72938	CELL ARRANGEMENT & PIPING SHEET 1	
W 74674	" " " " " 2 SHEET	
W 72590	FIRST FLOOR POWER PLAN 224 BLDG.	
W 74859	ELECTRICAL DETAILS SHEET 2	

[redacted]

Cell B catch tank (B-3). D-1 also includes return lines from the catch tank (D-3) and solution tank (D-4) and a discharge line to the centrifuge (D-2). In addition, D-1 has a discharge line from the Cell E isolation supernatant reservoir (E-4), for receiving recycled lanthanum product solutions. The centrifuge (D-2 has a drain line to the catch tank (D-3) and a jet discharge line to the solution tank (D-4).

The catch tank (D-3) has discharge lines out of the cell to the Cell E precipitator (E-1) and Cell B precipitator (B-1) for passing on to the lanthanum fluoride product precipitation. The solution tank (D-4) has an out-of-the-cell line to the waste neutralizer (C-8) only. The layout of Cell D equipment and piping is shown in Figure 4.

Cell E

Cell E is normally the lanthanum fluoride product cell. Its precipitator (E-1) can receive oxidized product effluent from either A-3, B-3, or D-3. It possesses a return line from the catch tank (E-3) only, and the usual discharge line to the centrifuge (E-2). The centrifuge has a drain line to the catch tank (E-3) and jet discharge lines to the metathesis tank (F-1) and to the solution tank (E-4). The catch tank has a transfer line out of the cell to the waste neutralizer (C-8) only. The solution tank (E-4) has a recycle transfer line from the Cell F F-10 Tank enclosure and a discharge to the lanthanum fluoride by-product precipitator (D-1).

The layout of Cell E equipment and piping is shown in Figure 5.

Cell F

Cell F is the metathesis cell. All of its equipment is built on a smaller scale than the rest of the cells because of the volume reduction taking place after cross-over. The metathesis tank (F-1) receives lanthanum fluoride product slurries from either B-2 or E-2 centrifuges. It has a discharge line going to either the regular 26-inch centrifuge (F-2) or to the spare (F-22). It can receive recycle solution from the catch tank (F-7) for reworking. The lanthanum hydroxide cake slurry returns from the centrifuges (F-2) and (F-22) also discharge into F-1 for the metathesis wash operation.

The centrifuges (F-2) and (F-22) have drain lines running into a common manifold leading to the primary catch tank (F-9). Discharge jets go to the metathesis tank (F-1) and to the final product solution tank (F-10). The primary catch tank (F-9) discharges into the secondary catch tank (F-7), which in turn discharges back into F-1 and to the waste neutralization tank (C-8). The sump waste tank (F-8) receives the discharges from the Cell F floor sumps and F-10 enclosure sump and has a transfer line to waste (C-8).

The final product solution tank (F-10) is located inside a separately ventilated enclosure and receives product from either centrifuge (F-2) or (F-22). It is a weigh tank and has a bottom drain for unloading the product solution into the transfer container.

The layout of Cell F equipment and piping is shown in Figure 6.

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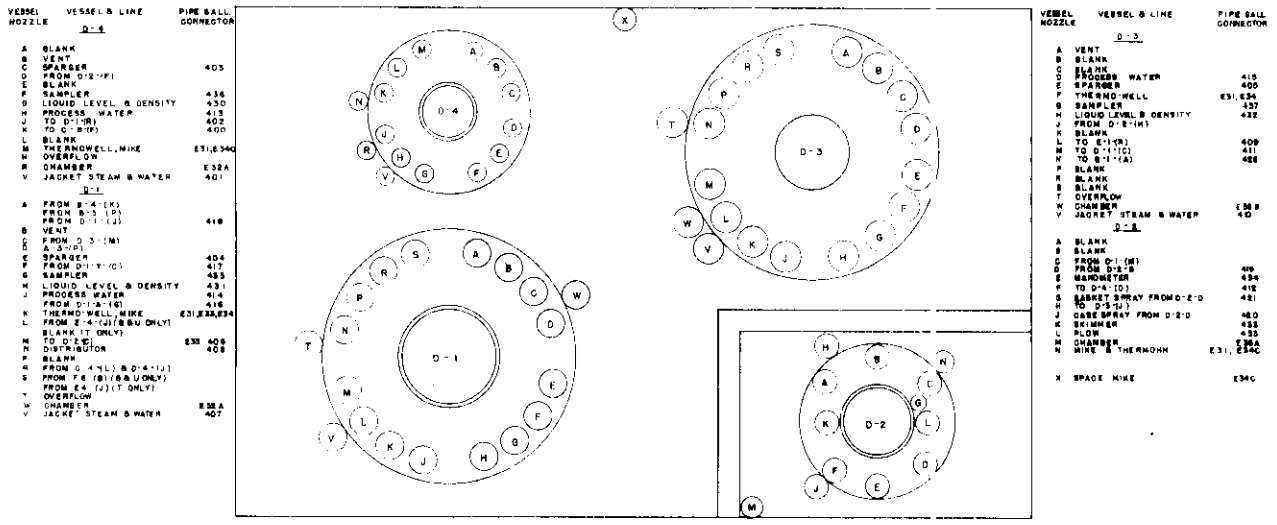
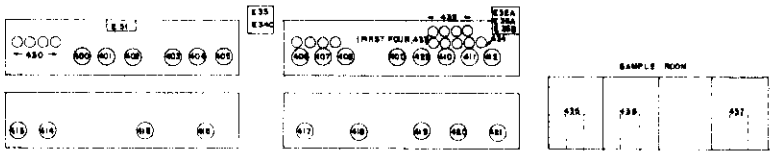
FIGURE 4

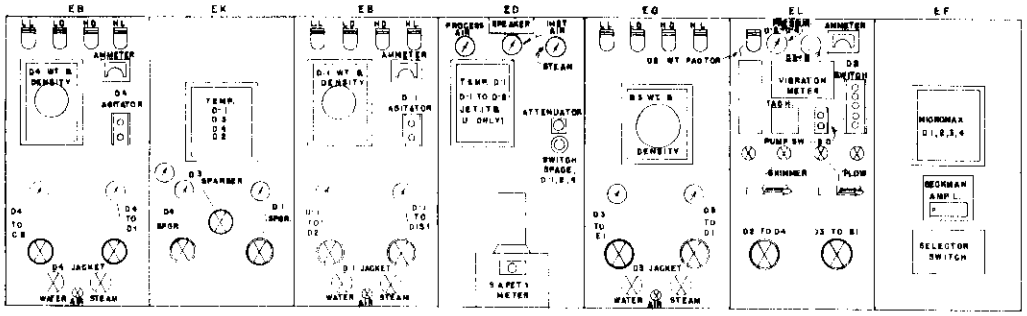
LAYOUT OF CELL D-CONCENTRATION (224) BUILDING



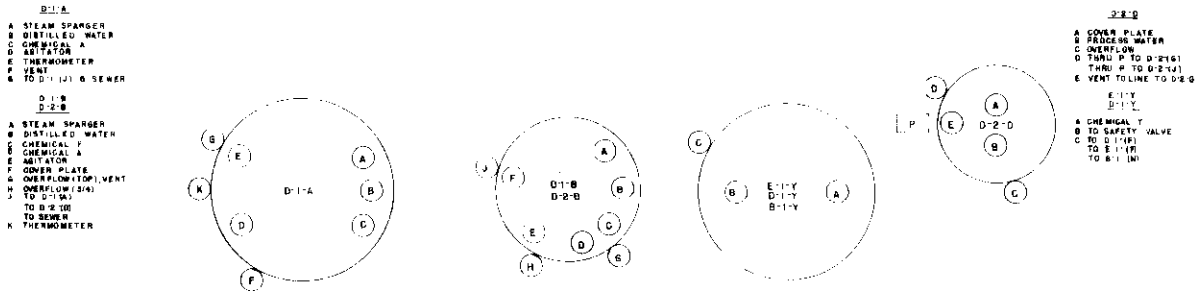
EQUIPMENT ARRANGEMENT



PIPE GALLERY CONNECTORS



PANEL BOARD



CONTROL GALLERY WEIGH TANKS

LEGEND

- EQUIPMENT WITHOUT MOTOR DRIVE
- EQUIPMENT WITH MOTOR DR. E
- ORDINARY VALVE
- GATE VALVE
- GLASS INDICATING PRESSURE DOWNSTREAM FROM GATE VALVE
- NOZZLE CONNECTOR ON EQUIPMENT
- W-40-P FLANGE CONNECTOR ON WALL
- ELECTRICAL CONNECTOR ON WALL
- PUMP
- BUBBLER AIR SUPPLY TO MANOMETER SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, " "
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, " "

REFERENCE DRAWINGS

W-11608	ARRANGEMENT & PIPING, 224 BLDG 3RD FLOOR CELLS A TO E.
W-11610	" " " 224 " 2ND FLOOR " A TO E.
W-74699	GAGE BOARD ARRANGEMENT & PIPING
W-75380	CELL ARRANGEMENT & PIPING SHEET
W-74675	" " " SHEET
W-73890	FIRST FLOOR POWER PLAN 224 BLDG
W-74688	ELECTRICAL DETAIL, SHEET 8
H-2-864	PIPING ALTERATIONS (GENERAL, 224 BLDG.)

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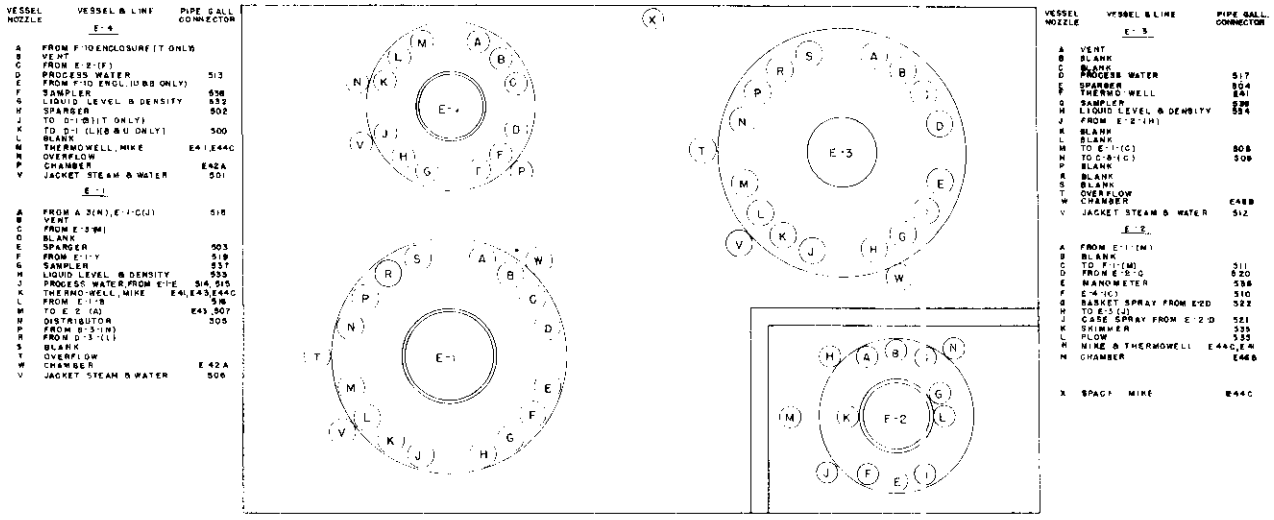
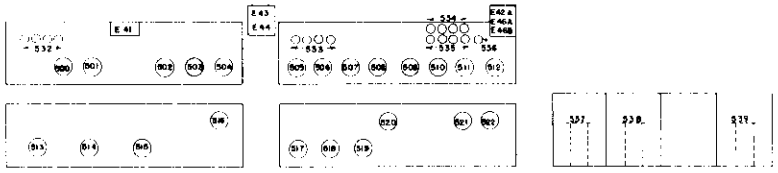
FIGURE 5

LAYOUT OF CELL E-CONCENTRATION (224) BUILDING



EQUIPMENT ARRANGEMENT



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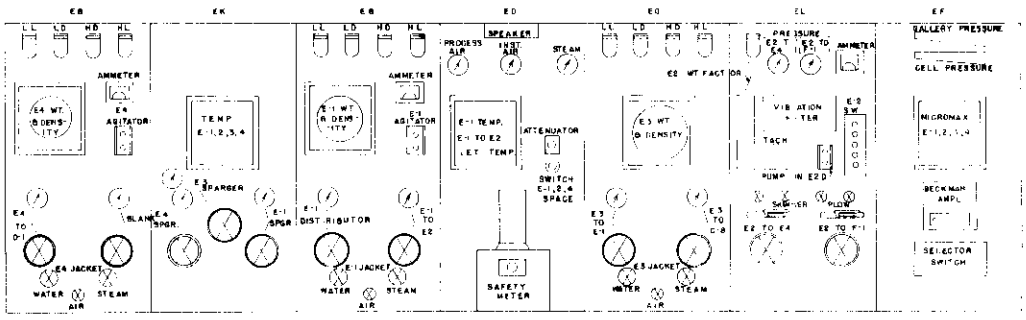
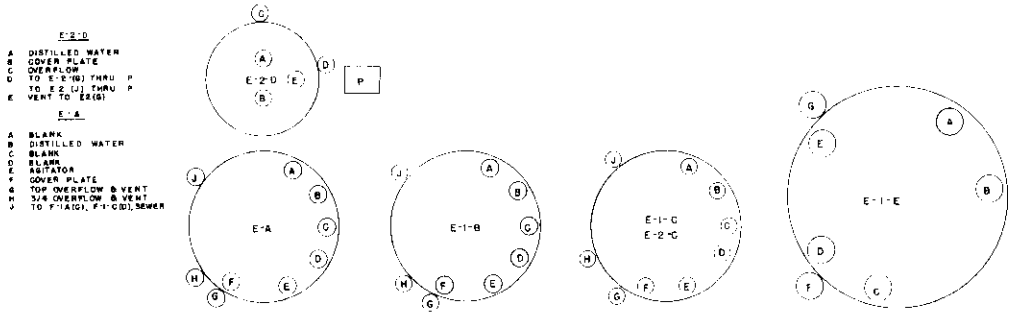
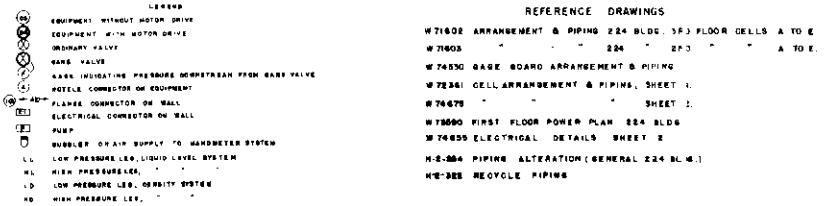
PIPE GALLERY CONNECTORS



PANEL BOARD



CONTROL GALLERY WEIGH TANKS



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HW 10475-C

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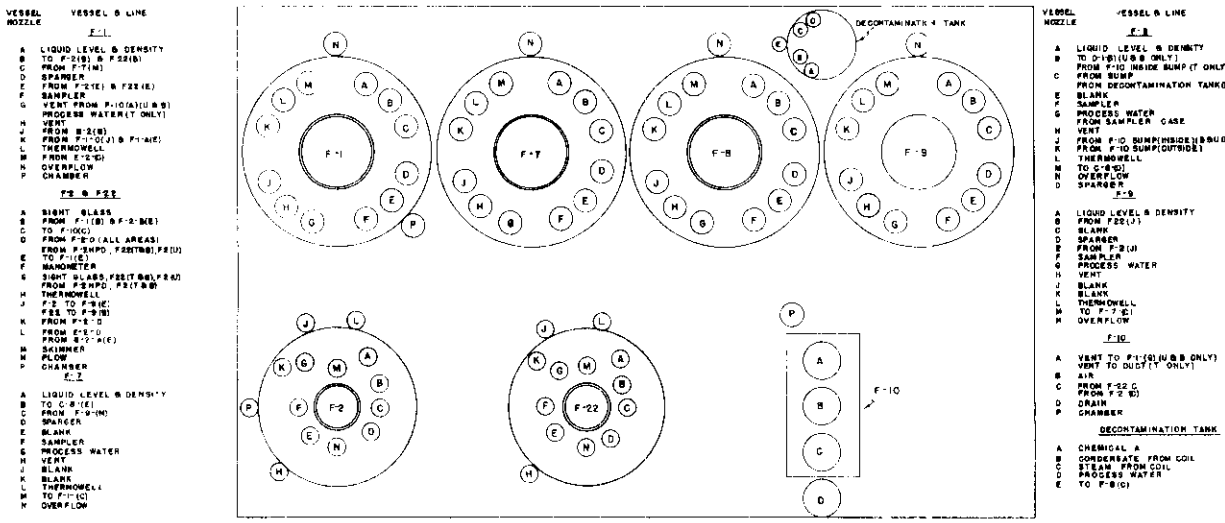
FIGURE 6

LAYOUT OF CELL F - CONCENTRATION(224) BUILDING



EQUIPMENT ARRANGEMENT

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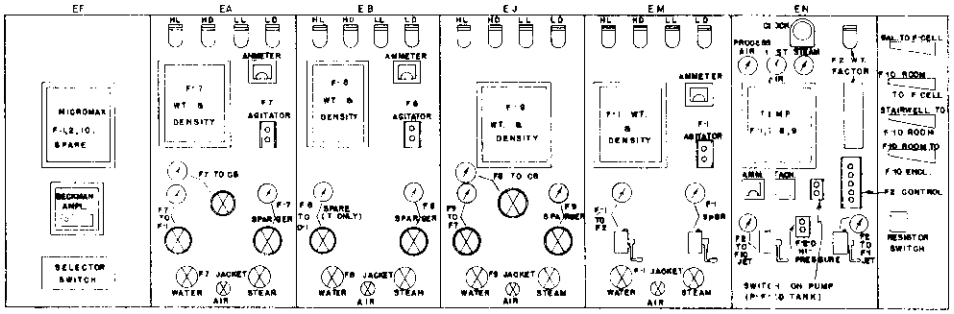
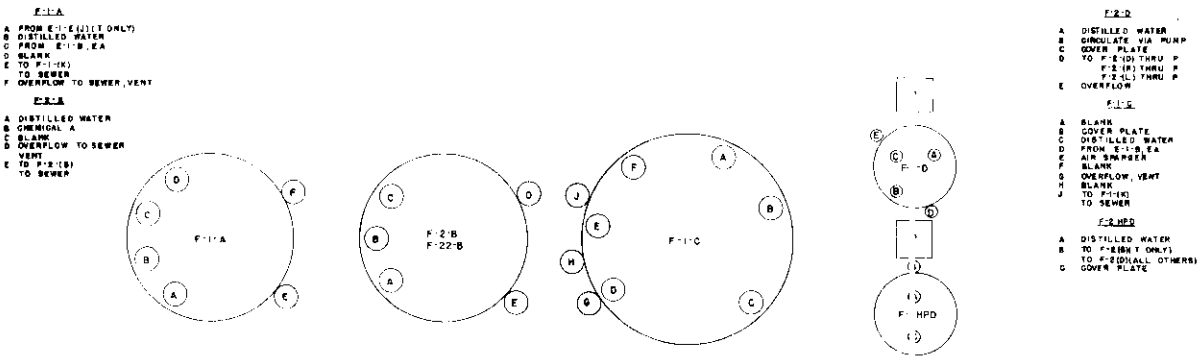


PANEL BOARD



CONTROL GALLERY WEIGH TANKS

LEGEND

EQUIPMENT WITHOUT MOTOR DRIVE
EQUIPMENT WITH MOTOR DRIVE
ORDINARY VALVE
GATE VALVE
GAGE INDICATING PRESSURE DOWNSTREAM FROM GATE VALVE
NOZZLE CONNECTOR ON EQUIPMENT
FLANGE CONNECTOR ON WALL
ELECTRICAL CONNECTOR ON WALL
PUMP
BUBBLER ON AIR SUPPLY TO MANOMETER SYSTEM
LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
HL HIGH PRESSURE LEG, " " "
LD LOW PRESSURE LEG, DENSITY SYSTEM
HD HIGH PRESSURE LEG, " "

REFERENCE DRAWINGS

W-7453-1 GAGE BOARD ARRANGEMENT & PIPING
W-74677 CELL ARRANGEMENT & PIPING SHEET 1
W-74678 " " " SHEET 2
W-75890 FIRST FLOOR PLAN 224 BLDG.
H-2-276 STAND-BY CENTRIFUGE & PIPING
H-2-284 PIPING ALTERATION (GENERAL, 224 BLDG.)
H-2-285 PIPING ALTERATIONS CELL F.
H-2-355 RECYCLE PIPING

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Precipitators A-1, B-1, D-1, E-1

These precipitators are 9 ft. by 9 ft. jacketed, agitated tanks with an overflow capacity of approximately 3700 gallons. They differ from the Canyon Building precipitators in that their jackets are twice as large (approximately 8 ft.), the agitator couplings are located outside of the tank, their overflow lines have water seals, and, as is true of all the Concentration Building equipment, are separately and directly tied into the main (291) ventilation system. Instrumentation and equipment include two thermometers, liquid level and density recorder, circulation distributor, microphone, sampler, sparger, and outside ionization chamber.

Centrifuges A-2, B-2, D-2 and E-2

These are Bird, 40-inch solid bowl machines as illustrated in Figure 9 of Chapter V of this section, except that the special flanges for remotely operated connectors are omitted. They can be operated at either 870 or 1740 rev./min. plugged to a stop, or inched at slow speeds for cake slurrying operations.

The bowl holdup is 60 gallons, giving a holdup time of 5 minutes at a feed rate of 110 lbs./min. The holdup can be reduced to 10 gallons with the skimmer and the cake can be scraped down from the walls by mechanical plows. Both the skimmer and the plows are hydraulically operated from the control gallery.

Instrumentation and equipment include bowl and case sprays operating under 70 lbs./sq.in. pump pressure, resistance thermometer, tachometer, wobble indicator, microphone, liquid level indicator, and outside ionization chamber.

Catch Tanks A-3, B-3, D-3 and E-3

Design of the catch tanks is identical to that of the precipitators, except that agitators and distributors are omitted. Each tank is provided with a resistance thermometer, liquid level and density recorder, sampler, sparger, microphone, and outside ionization chamber.

Solution Tanks A-4, B-4, B-6, C-4, D-4 and E-4

The solution tanks are 4 1/2 ft. by 7 ft. jacketed, agitated tanks with an overflow capacity of approximately 725 gallons. Instrumentation and equipment include resistance thermometer, liquid level and density recorder, sampler, sparger, microphone, and an outside ionization chamber.

Metathesis Tanks F-1, F-7, F-8, and F-9

These four Cell F vessels are 4 ft. by 5 ft. jacketed tanks of approximately 415 gallons overflow capacity, all equipped with agitators, except F-9. Instrumentation on each include resistance thermometer, liquid level and density recorder, sampler, and sparger. F-1 is the only tank equipped with an outside ionization chamber.

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Centrifuges F-2 and F-22

These centrifuges are similar to the other machines used in this process, but are 26-inch instead of the larger 40-inch machines used elsewhere. F-2 is normally used in the process and F-22 is the stand-by. Bowl speeds are 900 and 1800 rev./min. The running holdup is approximately 18 gallons and this holdup can be reduced to 3 gallons or less by skimming. Skimmer and plows, the latter being provided with GX scraper blades, are manually operated by geared extension shafts. Equipment and instrumentation include a two-nozzle high pressure spray (300 lbs./sq.in.) for the two lower baffled sections, a four-nozzle low pressure spray (70 lbs./sq.in.) for all four baffled sections, tachometer, plug control, inch control, liquid level indicator, sight glass sprays, resistance thermometer, and outside ionization chamber.

Product Storage Tank F-10

Final product solution from Centrifuge F-2 is jetted to F-10, a process scale tank 4 inches wide, 3 feet long, and 2 feet 8 inches high, with a capacity of 20 gallons. The solution is drained out of this tank into containers for transfer to the Isolation Building. The tank is suspended inside a glass enclosure with a stainless steel drip pan floor. Any spillage or drainage collects in a small sump in the drip pan floor, from which it is jetted to F-8. The tank is equipped with an air sparger and a direct vent to the ventilation system. The enclosure surrounding the tank is also tied in directly to the ventilation system.

The narrow width of the tank facilitates the escape of radiation from the contents and minimizes the development of undesirable side reactions.

Final Product (PR) Containers

A number of special containers are provided to transfer the final product solution from the Concentration Building to the Isolation Building and to return recycle solutions to the Concentration Building. These containers are described in detail in Chapter VIII (see Vessel Number F-1). The container assembly consists of a removable inner vessel of 15 gallons capacity, possessing a spherically ground closure, and an outer vessel serving as a "carrier" container. The handling of the compound container in the Concentration Building is the same as that described for the Isolation Building.

Waste Tanks C-7 and C-8

These vessels are identical to the jacketed, agitated precipitators previously described, with the exception that they have no distributors. Instrumentation is the same as for the precipitators.

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Scale Tanks

Nitric, phosphoric and hydrofluoric acid, and caustic headers are supplied from Building 271 and are connected to scale tanks metering these chemicals into the cell process vessels. All other chemical reagent solutions used in the process are made up on the control gallery in the Concentration Building.

A list of the scale tanks and their description is given in Table III. Details of their arrangement are shown in the respective cell diagrams, Figures 1 to 6.

The essential materials and their specifications, as used in the reagent solutions make-up, are listed in Report CN-2021.

A-1A is used to add the large volume of dilution water to A-1 for the bismuth phosphate by-product precipitation and also to add the nitric acid for cake solution to A-1. A-1B is used to add phosphoric acid and the water wash to A-1.

C-4B is used to prepare the 6.5% sodium bismuthate-2.5% sodium dichromate solution-slurry oxidation mixture which is added to C-4 for the primary oxidation in the concentration process. These concentration are used because they represent the addition of the required amount of chemicals to the minimum volume of water that will permit agitation during the addition of the bulk of the slurry to the cell process tank. C-8A is used to meter 50% sodium hydroxide into the waste neutralization tank, C-8.

D-1-B is used to prepare the 5% potassium permanganate solution, the 10% lanthanum salt - 10% nitric acid solution, and the 6% nitric acid cake wash solution, all of which are added to D-1 in the lanthanum fluoride by-product step.

B-1-D-1-E-1-Y is used as an anhydrous hydrofluoric acid storage tank, out of which is bled metered amounts of acid to B-1, D-1 and E-1 in the fluoride cycle.

E-1-E is used to prepare the 9.6% oxalic acid solution and the 1.5% lanthanum salt-1% nitric acid solution, which are added to E-1 in the lanthanum fluoride product step. E-1C is used to prepare the 6% nitric acid cake wash solution, which is also added to E-1.

E-A is used to make up the 50% potassium hydroxide solution supplied to the F-1 metathesis via E-2 or F-1C. E-1-B is used to make up the 17% potassium hydroxide which is also routed to the F-1 metathesis wash via F-1C.

F-2B is used to add the 60% nitric acid and water rinse to F-2 Centrifuge for the final cake solution, F-1C is used as an intermediate transfer tank for the potassium hydroxide solutions sent to metathesis from the main control gallery.

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TABLE III

CONCENTRATION (224) BUILDING SCALE TANKS

Operating No.	Nominal Value (gal.)	Diam.	Ht.	Appurtenances				Heaters Supplied				
				Steam Sparger	Air Sparger	Agitator	Steam Condensate	Sanitary Water	H_3PO_4 (73%)	HNO_3 (60%)	NaOH (50%)	HF Anhydrous
A-1 A	400	3'6"	6'		X		X			X		
A-1 B	90	2'6"	2'6"			X	X		X			
A-1C-2C	75	2'6"	2'7"		X		X		X	X		
B-1A	90	2'6"	2'6"			X	X			X		
B-1 B-2B	75	2'6"	2'7"		X		X		X	X		
B-1C	400	3'6"	6'			X	X		X	X		
C-8 A	400	3'6"	6'		X			X			X	
C-4B	75	2'6"	2'7"			X	X					
D-1 A	400	3'6"	6'			X	X			X		
D-1B-2B	90	2'6"	2'6"	X		X	X		X	X		
B-1-D-1-E-1Y	400	3'6"	6'									X
E-A	90	2'6"	2'6"			X	X					
E-1B	90	2'6"	2'6"			X	X			X		
E-1C-2C	75	2'6"	2'7"		X		X			X		
E-1 E	400	3'6"	6'	X		X	X			X		
F-1A	23	1'8"	1'9"				X					
F-2B	23	1'8"	1'9"				X			X		
F-1C	75	2'6"	2'7"		X		X					

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Centrifuge Spray Pumps A-2D, B-2D, D-2D, E-2D, F-2D, F-2HPD

Except for F-2HPD, the above pump systems consist of a 19-gallon head tank and a centrifugal pump delivering water under at least 70 lbs./sq.in. pressure to the centrifuge bowl and case sprays. The bowl sprays are used to cut the cake off the bowl walls in either the cake wash or slurry removal operations. The case sprays can be used to flush off the outside of the bowl and the inside of the case.

F-2HPD is a pump system consisting of a 55-gallon head tank and a high pressure (300 lbs./sq.in.) positive displacement pump used to cut the lanthanum hydroxide cake from the two lower baffled sections from the Metathesis Centrifuges F-2 or F-22.

Steam Condensate System

The sanitary water originally provided as process water proved to contain too many dissolved materials (such as calcium and magnesium) to permit the isolation of satisfactorily pure product in the Isolation Building without extra processing. It was found that relatively pure water (approximately 1 part per million of calcium) could be obtained by condensing process steam.

The facilities provided for condensing process steam include a two-pass condenser operating on 15 lbs./sq.in. steam. Approximately 1500 lbs./hr. of condensate can be produced at an exit temperature of less than 100 °F. A 20,000-gallon wooden storage tank is located on the roof of the building. The condensate is lifted to the storage tank by the steam pressure in the system and is fed to the scale tanks by gravity flow. Automatic control of the condenser by the liquid level in the storage tank is provided.

Samplers

The construction and operation of the samplers is essentially as described in Chapter V. At T Plant, only the deep well tanks, C-7 and C-4, are provided with air lifts. At U and B Plants, all the samplers in Cells A to E are provided with air lifts. The T and U Plant sampler cups are fitted with angle-entry inlets, to furnish maximum mixing of the cup contents during circulation. At B Plant, a simple baffle plate in front of the cup inlet is provided to accomplish the same function.

In Cells A to E, the samplers are placed in wall pits inside closed rooms located on the pipe-gallery (second floor). The sampler rooms themselves are separated from the process equipment area by the 12-inch concrete wall and serve as entrances to the centrifuge platforms. Entrance to the sampler rooms can be made at any time, regardless of the process status within the operating equipment areas.

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In Cell F, the samplers are located on the centrifuge balcony, inside a stainless steel trough hung from the balcony rail, and are approached only when the proper precautions have been taken, such as the wearing of masks and special clothing.

The sampling pipettes used in the Concentration Building are of the bayonet type described in Chapter V.

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REFERENCES

1.	File 3-1493	1-22-45
2.	SE - PC - 18	4-20-45
3.	File 3-1639	2-18-45
4.	SE - PC - 21	5-2-45
5.	File 3-2723	7-2-45
6.	File 3-2803	7-14-45
7.	SE - PC - 44	6-11-45
8.	CN-2010	10-20-44
9.	CN-1214	
10.	SE - PC -3	1-15-45
11.	CN-2021	10-1-44
12.	CN-2053	10-16-44
13.	File 3-2029	4-4-45
14.	File 3-2525	6-5-45
15.	SE - PC - 56	6-19-45
16.	SE - PC - 52	6-19-45
17.	File 3-2507	5-29-45
18.	SE - PC - 65	7-6-45
19.	File 6006	1-3-45
20.	SE - PC - 61	7-2-45

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APPENDIX

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FLOW SHEET

Following this page is given the process flow sheets of the operations carried out in the Concentration Building.

OPERATING LOG FORMS

At the end of this appendix is given a complete set of Operating Log Forms which constitute the detailed operating instructions for Concentration Building operations.

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APPENDIX

CONCENTRATION PROCESS FLOW SHEET

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PRODUCT OXIDATION - BUILDING 224 - CELL C

Oxidation

1) Receive approximately 2125 lbs. (200 gal.) of product cake solution from Tank 17-4, Building 221 into Tank C-4.

2) Make up to 2300 lbs. (220 gal.) by adding water via Scale Tank C-4B.

3) Add 135 lbs. (15 gal.) of 6.5% $NaBiO_3$ - 2.5% $Na_2Cr_2O_7$ oxidation slurry mixture.

4) Agitate for 1 hour at 45 °C.

5) Cool to 35 °C.

6) Jet to Precipitator A-1.

Solution Composition (in C-4)

	lbs.	%	M
$BiPO_4$	43	1.73	21.8 g/l
HNO_3	940	37.5	7.5
$NaBiO_3$	8.8	0.35	0.016 (a)
$Na_2Cr_2O_7$	3.4	0.14	0.0065
H_2O	1495	60	--
Total	2490	(238 gal.)	

Note:
(a) $NaBiO_3$ concentration at start of oxidation; it is completely decomposed during the oxidation step.

$BiPO_4$ BY-PRODUCT PRECIPITATION - BUILDING 224 - CELL A

A. Precipitation

1) Add 8000 lbs. (960 gal.) of water to A-1, start agitator, and heat to 80 °C with jacket and steam sparger.

2) Receive approximately 2550 lbs. (243 gal.) of oxidized solution from C-4 into A-1.

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3) Add rapidly 165 lbs. (12.5 gal.) of H_3PO_4 solution (73.5% H_3PO_4 - 1.2% HNO_3).

4) Adjust temperature to 75 °C and agitate for 1 hour at 75 °C.

5) Cool to 35 °C and jet to centrifuge A-2

Slurry Composition (in A-1)

	lbs.	%	M
HNO_3	942	8.48	1.43
H_3PO_4	118	1.06	0.113 (a)
$BiPO_4$	53	0.48	5.0 g/l (a)
$Na_2Cr_2O_7$	3.4	0.031	0.0012
H_2O	9981	90	---
Total	11,100	(1265 gal.)	

B. Centrifugation

1) Centrifuge at 870 rev./min., 110 lbs./min.

2) Recirculate three 500-lb. batches of effluent from A-3 to A-1 to A-2.

3) Skim A-2 to 10 gal. heel.

4) Add 250 lbs. (30 gal) of water to A-1.

5) Circulate through distributor for 3 minutes.

6) Jet wash water from A-1 to A-2.

7) Skim to 10 gal. heel.

8) Add 250 lbs. (30 gal.) of water to A-2 through sprays.

9) Wash cake by slurrying A-2 three times from 0 to 110 rev./min.

10) Skim A-2 to 10 gal. heel.

11) Jet filtrate and washings to Precipitator D-1.

Filtrate Composition (in A-3)

	lbs.	%	M
HNO_3	942	7.91	1.32
H_3PO_4	118	0.99	0.106
$Na_2Cr_2O_7$	3.4	0.028	0.0011
H_2O	10,837	91	
Total	11,800	(1350 gal.)	

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C. Cake Dissolution

1) Add 1400 lbs. (129 gal.) of 60% HNO_3 to A-1.

2) Circulate through distributor for 10 minutes with agitator running (b).

3) Jet 325 (30 gal.) from A-1 to A-2 and slurry cake.

4) Jet slurry from A-2 to Solution Tank A-4, with agitator running.

5) Repeat (3) and (4) twice.

6) Jet remainder of HNO from A-1 to A-2 to A-4.

7) Agitate A-4 for at least 1 hour after all HNO_3 has been added.

8) Jet solution from A-4 to Waste Neutralizer C-8.

Solution Composition (in A-4)

	lbs.	%	M
$BiPO_4$	53	3.15	41.5 g/l
HNO_3	840	50	10.5
H_2O	787	46.9	--
	1680 (153 gal.)		

Notes:

(a) Corrected for 9.7 lbs. of $BiPO_4$ formed by the decomposition of $NaBiO_3$.

(b) This recirculation is calculated to produce a dilution of approximately 100 lbs. (12 gal) to the dissolving acid.

LaF_3 BY-PRODUCT - BUILDING 224 - CELL D

A. Oxidation and Precipitation

Process I: No recycling of Isolation Supernatants

1) Receive approximately 11,900 lbs. (1370 gal.) of oxidized filtrate from A-3 into D-1.

2) Heat to 75 °C.

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3) Add 390 lbs. (44 gal.) of 5% $KMnO_4$ solution. (a)

4) Agitate for 30 minutes at 75 °C.

5) Cool to 35 °C.

6) Add 53 lbs. of anhydrous HF at rate of 10 lbs./min.

7) Add 220 lbs. (26 gal.) of 10% La salt (b) - 10% HNO_3 solution at rate of 10 lbs./min.

8) Agitate for 1 hour at 35 °C.

9) Jet to Centrifuge D-2.

Slurry Composition (in D-1)

	lbs.	%	M
HNO_3	964	7.60	1.26
H_3PO_4	118	0.92	0.099
$Na_2Cr_2O_7$	3.4	0.027	0.0011
$KMnO_4$	20	0.158	0.010
HF	51	0.40	0.21
LaF_3	8.1	0.059	500 mg/l La
NH_4NO_3	6	0.047	0.006
H_2O	11,530	90.9	
Total	12,700	(1460 gal.)	

Process II: Recycling of Isolation Supernatants (9.0 lbs. of Lanthanum)

1) Receive approximately 11,900 lbs. (1370 gal.) of oxidized filtrate from A-3 into D-1.

2) Heat to 75 °C.

3) Receive the equivalent of 9.0 lbs. of lanthanum from isolation supernatant recycles, approximately 700 lbs. (75 gal.), into D-1 from Recycle Storage Tank E-4.

4) Add 390 lbs. (44 gal.) of 5% $KMnO_4$ solution.

5) Agitate for 30 minutes at 75 °C.

6) Cool to 35 °C.

7) Add 53 lbs. of anhydrous HF at rate of 3 lbs./min.

8) Agitate for 1 hour at 35 °C.

9) Jet to Centrifuge D-2.

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Slurry Composition (in D-1)

	lbs.	%	M
HNO_3	970	7.41	1.22
H_3PO_4	118	0.90	0.096
$Na_2Cr_2O_7$	3.4	0.026	0.0011
$KMnO_4$	20	0.153	0.010
HF	50	0.38	0.20
LaF_3	12.2	0.078	720 mg/l La
H_2O	11,918	91	
Total	13,090	(1510 gal.)	

B. Centrifugation

1) Centrifuge at 1740 rev./min., 70 lbs./min.

2) Reduce D-2 to 870 rev./min. and skim to 10-gal. heel.

3) Add 500 lbs. (60 gal.) of 6% HNO_3 to D-1.

4) Circulate through distributor for 3 minutes.

5) Jet remainder of acid wash to D-2 and slurry.

6) Raise D-2 to 1740 rev./min., hold 10 minutes, reduce to 870 rev./min. and skim to 10-gal. heel.

7) Jet remainder of acid wash to D-2 and slurry.

8) Repeat (6).

9) Jet filtrate and washings for D-3 to Precipitator E-1.

Filtrate Composition (in D-3) (c)

	lbs.	%	M
HNO_3	994	7.35	1.22
H_3PO_4	118	0.87	0.093
$Na_2Cr_2O_7$	3.4	0.025	0.0010
$KMnO_4$	20	0.15	0.0098 (d)
HF	51	0.38	0.198
NH_4NO_3	6	0.044	0.006
H_2O	12,308	91.1	
Total	13,500	(1550 gal.)	

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C. Cake Removal

1) Slurry cake from D-2 with 2000 lbs. (240 gal.) of water, added via bowl sprays and jetted continuously to D-4.

2) Add 250 lbs. (30 gal.) of water to D-2, slurry from 0 to 110 rev./min. and jet to D-4.

3) Repeat (2) twice.

4) Add 300 lbs. of water (36 gal.) to D-2 via bowl sprays, jetting continuously to D-4.

5) Jet by-product waste slurry from D-4 to Waste Neutralizer C-8.

Waste Slurry Composition (in D-4) (c)

	lbs.	%	M
LaF_3	8.1	0.25	1860 mg/l La (e)
HNO_3	trace		
HF	trace		
MnO_2	present		
H_2O	3193	99.8	
Total	3200	(384 gal.)	

Notes:

(a) The 5% $KMnO_4$ reagent solution is prepared by heating to 75 °C to hasten its solution.

(b) $La(NO_3)_3 \cdot 2NH_4NO_3 \cdot 4 H_2O$.

(c) Calculated for regular process with no recycling.

(d) Calculated for no decomposition of the $KMnO_4$, which does take place to an unknown extent to contribute MnO_2 to the by-product cake.

(e) If a 9.0 lb. lanthanum recycle is used, the waste slurry will contain 12.2 lbs. of LaF_3.

LaF_3 PRODUCT PRECIPITATION - BUILDING 224 - CELL E

A. Reduction and Initial Precipitation

1) Receive approximately 13,700 lbs. (1575 gal.) of oxidized filtrate from D-3 into E-1.

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2) Adjust temperature to 35 °C.

3) Add 1435 lbs. (170 gal.) of 9.6% $H_2C_2O_4 \cdot 2H_2O$ solution. (a)

4) Agitate for 1 hour at 35 °C.

5) Add 120 lbs. of anhydrous HF at rate of 10 lbs./min.

6) Add 500 lbs. (59 gal.) of 1.5% La salt - 1% HNO_3 solution at rate of 25 lbs./min. (b)

7) Agitate for 1 hour at 35 °C.

8) Jet to Centrifuge E-2.

Slurry Composition (in E-1)

	lbs.	%	M
HNO_3	970	6.10	1.02
H_3PO_4	118	0.745	0.08
$NaNO_3$	2.2	0.014	--
KNO_3	12.5	0.079	--
$Cr(NO_3)_3$	5.5	0.035	--
HF	170	1.07	0.565
LaF_3	2.72	0.016	133 mg/l La
$H_2C_2O_4 \cdot 2H_2O$	98	0.62	0.051
$Mn(NO_3)_2$	23	0.145	0.008
NH_4NO_3	8	0.050	--
H_2O	14,440	90.9	
Total	15,850	(1810 gal.)	

B. First Centrifugation and Final Precipitation

1) Centrifuge at 1750 rev./min. 70 lbs./min.

2) Reduce E-2 to 870 rev./min. and skim to 10-gal. heel.

3) Jet filtrate from E-3 to E-1.

4) Add 500 lbs. (59 gal.) of 1.5% La salt - 1% HNO_3 solution at rate of 25 lbs./min. (b)

5) Agitate for 1 hour at 35 °C.

6) Jet to Centrifuge E-2.

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Slurry Composition (in E-1)

	lbs.	%	M
HNO_3	975	5.76	0.96
H_3PO_4	118	0.697	0.075
$NaNO_3$	2.2	0.013	--
KNO_3	12.5	0.074	--
$Cr(NO_3)_3$	5.5	0.033	--
HF	169	1.00	0.525
LaF_3	2.72	0.015	125 mg/l La
$H_2C_2O_4 \cdot 2H_2O$	98	0.58	0.048
$Mn(NO_3)_2$	25	0.136	0.008
NH_4NO_3	8	0.047	--
H_2O	15,486	91.5	
Total	16,900	(1930 gal.)	

C. Final Centrifugation

1) Centrifuge at 1740 rev./min. 70 lbs./min.

2) Reduce E-2 to 870 rev./min. and skim to 10-gal. heel.

3) Add 500 lbs. (60 gal.) of 6% HNO_3 to E-1.

4) Circulate acid wash through distributor for 3 minutes.

5) Raise E-2 to 1740 rev./min. and jet acid wash from E-1 to E-2.

6) Reduce E-2 to 870 rev./min. and skim to 10-gal. heel.

7) Proceed with E-2 cake removal to F-1 (see Part D).

8) Jet waste filtrate and washings from E-3 to C-8.

Filtrate Composition (in E-3)

	lbs.	%	M
HNO_3	1005	5.62	0.93
H_3PO_4	118	0.660	0.070
$NaNO_3$	2.2	0.012	--
KNO_3	12.5	0.070	--
$Cr(NO_3)_3$	5.5	0.031	
HF	169	0.945	0.492
$H_2C_2O_4 \cdot 2H_2O$	98	0.55	0.045
$Mn(NO_3)_2$	23	0.130	0.008
NH_4NO_3	8	0.045	--
H_2O	16,459	92.0	--
Total	17,900	(2006 gal.)	

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D. Cake Removal

1) Slurry cake from E-2 with 700 lbs. (84 gal.) of water, added via bowl sprays and jetted continuously to F-1.

2) Add 361 lbs. (29 gal.) of hot (60-85°C) 50% KOH to E-2, slurry from 0 to 110 rev./min., and jet to F-1.

3) Repeat (2).

4) Add 500 lbs. (60 gal.) of water to E-2 via bowl sprays, jetting continuously to F-1, making total weight in F-1 up to 2400 lbs.

Notes:

(a) The 9.6% $H_2C_2O_4 \cdot H_2O$ solution is prepared by heating to 65 °C to increase its solubility.

(b) $La(NO_3)_3 \cdot 2\ NH_4NO_3 \cdot 4H_2O$.

KOH METATHESIS - BUILDING 224 - CELL F

A. Metathesis

1) Receive approximately 2400 lbs. (250 gal.) of LaF_3 - KOH slurry from E-2 into F-1.

2) Heat to 80 °C and agitate for 90 minutes.

3) Cool to 35 °C.

4) Jet to Centrifuge F-2.

Slurry Composition (in F-1)

	lbs.	%	M
$La(OH)_3$	5.43	0.21	1920 mg/l La
KOH	362	15.0	3.07
KF	5.05	0.21	0.041
H_2O	2028	84.6	
Total	2400	(252 gal.)	

B. First Centrifugation

1) Centrifuge in F-2 at 1800 rev./min. at rate of 12-15 lbs./min., with effluent running to Catch Tank F-9.

2) Skim F-2 to 5-gal. heel.

3) Jet effluent from F-9 to F-7 to Waste Neutralizer 3-8.

Solution Composition (in F-7)

	lbs.	%	M
KOH	356	14.5	2.90
KF	4.9	0.20	0.039
H_2O	2099	85.3	--
Total	2460	(263 gal.)	

C. Cake Removal and Wash

1) Slurry cake from F-2 to F-1 with 450 lbs. (54 gal.) of water added via low pressure (70 lbs./sq.in.) bowl sprays, jetting continuously to F-1.

2) Repeat (1) with 450 lbs. (54 gal.) of water added via high pressure (300 lbs./sq.in.) bowl sprays.

3) Make F-1 weight up to 1600 lbs. (192 gal.) by adding water to F-2 via low pressure sprays and jetting continuously to F-1.

4) Add 250 lbs. (26 gal.) of 17% KOH solution.

5) Make F-1 weight up to 2100 lbs. (247 gal.) by adding water.

6) Adjust F-1 temperature to 35 °C and agitate for 1 hour.

7) Jet to Centrifuge F-2.

Slurry Composition (in F-1)

	lbs.	%	M
$La(OH)_3$	5.43	0.24	1960 mg/l La
KF	42.5	2.03	0.37
H_2O	2052	97.8	--
Total	2100	(247 gal.)	

D. Wash Centrifugation

1) Centrifuge in F-2 at 1800 rev./min. at rate of 25 lbs./min., with effluent running to Catch Tank F-9.

2) Skim F-2 to 3-gal. heel.

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3) Jet effluent from F-9 to F-7 to Waste Neutralizer C-8.

Solution Composition (in F-9)

	lbs.	%	M
KOH	41.9	1.90	0.35
H_2O	2158	98.1	--
Total	2200	(259 gal.)	

E. Cake Dissolution

1) Add 24 lbs. (2.1 gal.) of 60% HNO_3 to F-2.(a)

2) Slurry F-2 from 0 to 160 rev./min. for 30 minutes.

3) Cut cake from F-2 bowl wall with plows.

4) Rotate F-2 at 100 rev./min. for 30 minutes with plows three-fourths in.

5) Jet from F-2 to Receiving Tank F-10.

6) Add sufficient water to F-2 to make F-10 final weight 72 lbs. and slurry from 0 to 160 rev./min.

7) Jet wash water from F-2 to F-10.

8) Flush F-2 bowl back to F-1 with 100 lbs. (12 gal.) of water, added via bowl sprays.

9) Drop F-10 solution into container and transfer to Building 231.

Solution Composition (in F-10)

	lbs.	%	M
$La(NO_3)_3$	9.35	12.2	66 g/l La
HNO_3	7.64	10.6	2.0
KNO_3	1.07	1.49	0.175
$Pu(NO_3)_4$	1.35	1.88	10.9 g/l Pu (b)
H_2O	52.7	74.0	
Total	72	(7.3 gal.)	

Notes:

(a) The amount of 60% HNO_3 needed for cake solution may vary slightly due to variation in the total alkalinity of the F-2 heel.

(b) Based of the processing of 300 g. of product/charge.

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CONCENTRATION OPERATIONS

Date _____
Run No. _____

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I - PHOSPHATE BY-PRODUCT - OXIDATION

1.	Beckman on C-4.	Meter _____ Factor _____
2.	Obtain supervisor's approval to receive product cake solution from Building 221.	Approved by _____ Time _____ Supervisor
3.	Start C-4 agitator	C-4 agitator on _____
4.	Receive product cake solution (Approx. 2200 lbs.) from tank 17-4 in Bldg. 221 following notification by the 221 Bldg. dispatcher.	Time received _____
5.	When C-4 Wt.Ftr. stops rising, stop C-4 agitator, read Wt. Ftr. when constant, and notify Bldg. 221 dispatcher of weight received.	C-4 Agitator off _____ C-4 Wt. Ftr. _____ Lbs. _____ Time 221 Dis'pr. called _____
6.	Start agitator in C-4	C-4 agitator on _____
7.	Beckman on C-4.	Meter _____ Factor _____
8.	With supervisor's approval make C-4 up to total weight of 2300 lbs.. by adding water to C-4 via weigh tank C-4B. Agitate C-4 for 10 min.	Lbs. water added _____
9.	Stop C-4 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-4 agitator off _____ C-4 Wt. Ftr. _____ Lbs. _____ C-4 Sp. Gr. _____ Temp. _____
10.	Start C-4 agitator	C-4 agitator on _____
11.	When supervisor approves, adjust C-4 temperature to 45°C. (Notify Cell A to start Items 1 through 3, p. 7.3)	Time OK to proceed _____ Time 45°C _____
12.	Make up 135 lbs. of compound oxidizing mixture in C-4-B.	
	a. Add 120 lbs. water	Lbs. water add _____ Time _____
	b. Turn on agitator.	Time agitator on _____
	c. Add 4 lbs sodium dichromate	Lbs. sodium dichromate add Time
	d. Add 11 lbs. sodium bismuthate	Lbs. sodium bismuthate add Time
	e. Agitate 10 minutes	Lbs. Total _____
	f. Take sample to lab for approval	Time sampled _____ Sampled by _____
	g. Leave agitator on until slurry has been added to C-4.	Time OK'd by Lab _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

I - PHOSPHATE BY-PRODUCT OXIDATION (cont'd)

13.	Add the oxidation mixture to C-4 while agitating C-4B.	Lbs. add _____ Time _____
14.	Flush C-4B to C-4 with approx. 50 lbs. water by leaving C-4B to C-4 line open and adding successive portions of water to flush solid from bottom of C-4B tank.	Lbs. add _____ Time _____
15.	Agitate C-4 for 1 hr. holding the temperature at 45°C. DO NOT EXCEED 45°C.	Time start _____ Time end _____
16.	Cool C-4 to 35°C. (Start items 4 through 6 page 7.3).	Time 35°C _____
17.	Beckman on C-4.	Meter _____ Factor _____
18.	Stop C-4 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-4 agitator off _____ C-4 Wt. Ftr. _____ Lbs. _____ C-4 Sp. Gr. _____ Temp. _____
19.	Start C-4 agitator	C-4 agitator on _____
20.	Jet C-4 to [redacted] 1, upon supervisor's approval .	17-4-P assay _____ T.C. Approved by _____ Time _____ Supervisor Time jet on _____
21.	When C-4 Wt. Ftr. pen reaches 0.2 stop C-4 agitator.	C-4 agitator off _____
22.	When jet gasses, stop jet.	Time jet off _____
23.	Beckman on C-4	Meter _____ Factor _____

[redacted]

CONCENTRATION OPERATIONS

Date _____ Run No. _____

DECLASSIFIED

II - PHOSPHATE BY-PRODUCT PRECIPITATION

1.	Beckman on A-1	Meter _____ Factor _____
2.	Start A-1 Agitator.	A-1 Agitator on _____
3.	Add 8000 lbs. of water to A-1 via weigh tank A-1A in three portions	Lbs. 1st port. _____ Time _____ Lbs. 2nd port. _____ Time _____ Lbs. 3rd port. _____ Time _____ Lbs. total _____
4.	Use jacket steam and steam sparger to bring A-1 temp to 80°C.	Steam on _____ Time 80°C _____
5.	Stop A-1 steam sparger	A-1 sparger off _____
6.	Hold water at 80°C until step 9 is started.	
7.	Five minutes before receiving C-4-0 solution (item 9), stop A-1 agitator and read Wt. Ftr. when constant.	A-1 agitator off _____ A-1 Wt. Ftr. _____ Lbs. _____
8.	Start A-1 agitator	A-1 agitator on _____
9.	Receive approx. 2500 lbs. of oxidized solution from C-4 into A-1.	Time received. _____
10.	Stop A-1 agitator and read Wt. Ftr. when reading is constant.	A-1 agitator off _____ A-1 Wt. Ftr. _____ Lbs. _____ A-1 Sp. Gr. _____ Temp _____ Lbs. received into A-1 from C-4 (item 10 minus item 7).
11.	Start A-1 agitator.	A-1 agitator on _____
12.	Obtain supervisor's approval that C-4-0 solution has been completely transferred to A-1.	Approved by _____ Time _____ Supervisor
13.	Beckman on A-1.	Meter _____ Factor _____
14.	Add 165 lbs. of 75% Phosphoric Acid rapidly to A-1 via weight tank A-1B (Line F)	Time add _____ Lbs. _____
15.	Flush A-1B to A-1 with 50 lbs. of water.	Time add _____ Lbs. add _____
16.	Adjust A-1 temperature to 75°C	Time 75°C _____
17.	Agitate A-1 for 1 hr. at 75°C.	Time start _____ Time end _____
18.	Cool A-1 to 35°C and stop cooling water	Time start _____ Time 35°C _____

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[redacted]

CONCENTRATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

III - PHOSPHATE BY-PRODUCT CENTRIFUGING

1. Bring A-2 to 870 RPM. — Time 870 RPM _____

2. Beckman on A-2 — Meter _____ Factor _____

3. Beckman on A-3. — Meter _____ Factor _____

4. Start A-3 air sparger — A-3 sparger on _____

5. Transfer slurry from A-1 to A-2 at the rate of 110 lbs per min. Check the transfer rate at 30 minute intervals, recording data in table.

Time	Jet Press	A-3 Wt.	Lbs/min.
A		0	----

6. When the Wt.Ftr. pen on A-1 reaches 0.2, stop the agitator. — A-1 agitator off _____

7. Just as jet from A-1 to A-2 first gasses, indicating A-1 is empty (do not shut off jet), jet from A-3 to A-1 for 30 sec. and empty A-1 again. Repeat this operation two more times to flush out A-1. — Time jet gasses _____; Time 1st flush _____; Time 2nd flush _____; Time 3rd flush _____

8. When the jet from A-1 to A-2 gasses, raise jet steam pressure to full. Stop jet when A-2 ammeter and tachometer and jet Thermohm indicate A-1 is empty. — Time full pressure _____; P.S.I. _____; Time jet off _____

9. Five minutes after item 8, skim A-2 to a 10 gal. heel. — Time end skimming _____

10. Add 250 lbs. of water to A-1 via weight tank A-1B. — Lbs. added _____ Time _____

11. Circulate through A-1 distributor at 50 psi for 3 min. with cooling water on jacket — Time start _____ Time end _____; P.S.I. _____

12. Stop A-1 cooling water. — A-1 water off _____

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[redacted]

CONCENTRATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

III - PHOSPHATE BY-PRODUCT CENTRIFUGING (Cont'd)

13.	Jet wash water from A-1 to A-2 at full jet pressure. Stop jet when A-2 ammeter and tachometer and jet Thermohm indicate A-1 empty.	Time jet on _____ Time jet off _____
14.	Five minutes after item 13, skim A-2 to 10 gal. heel.	Time end _____
15.	Plug A-2 to 10 RPM.	Time 10 RPM _____
16.	With A-2 at 10 RPM add 250 lbs. of water via A-2-D at 70 psi or more. Use bowl spray.	Time add _____ Lbs. added _____ Pressure _____
17.	Bring A-2 to 110 RPM and hold 2 min. Plug to a stop. Repeat twice.	Time first slurrying _____ Time second _____ Time third _____
18.	Bring A-2 to 870 RPM, hold 5 min., and skim to a 10 gal. heel.	Time start _____ Time end _____
19.	Stop A-3 sparger and read Wt. Ftr. and Sp. Gr. when constant	A-3 sparger off _____ A-3 Wt. Ftr. _____ Lbs. _____ A-3 Sp.Gr. _____ Temp. _____
20.	Start A-3 air sparger.	A-3 sparger on _____
21.	Beckman on A-2 and A-3	A-2 Meter _____ Factor _____ A-3 Meter _____ Factor _____

Note: If A-3 cannot be jetted to D-1 at this point, proceed with A-2 cake removal, page 7.6.

22.	With supervisor's approval, jet A-3 to D-1 (Item 1 through 7 on p. 7.6 may be started while jetting).	Time OK to proceed _____ Time start jet _____
23.	When A-3 Wt. Ftr. pen reaches 0.3, stop A-3 sparger.	A-3 sparger off _____
24.	When jet gases, stop jet.	Time stop jet _____
25.	Beckman on A-1, A-2 and A-3.	A-1 Meter _____ Factor _____ A-2 Meter _____ Factor _____ A-3 Meter _____ Factor _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

IV - PHOSPHATE BY-PRODUCT CAKE REMOVAL

1.	Beckman on A-4.	Meter _____ Factor _____
2.	Plug A-2 to stop and take Wt. Ftr. reading.	Time stop _____ A-2 Wt. Ftr. _____ Lbs. _____
3.	Jet slurry from A-2 to A-4, until A-2 manometer reaches minimum	Time end _____ A-2 Wt. Ftr. _____ Lbs. _____
4.	Start A-1 agitator	A-1 agitator on _____
5.	Add 1,400 lbs. of 60% Nitric Acid to A-1 via weigh tank A-1A (Line A)	Time add _____ Lbs. Add _____
6.	Start cooling water and circulate through A-1 distributor at 50 psi for 10 min. Hold temperature below 40°C.	Time start _____ Time end _____ psi _____ A-1 Max. Temp. _____
7.	Stop A-1 agitator and cooling water	A-1 agitator off _____
8.	Jet 325 lbs. from A-1 to A-2.	A-1 water off _____ A-2 Wt. Ftr. _____ Lbs. _____
9.	Bring A-2 to 110 RPM. Hold 2 min. Plug to stop. Repeat twice.	Time first slurrying _____ Time second _____ Time third _____
10.	Start A-4 agitator and cooling water.	A-4 agitator on _____ A-4 water on _____
11.	With A-2 stopped, jet A-2 to A-4.	Time jet _____
12.	Jet 325 lbs. from A-1 to A-2.	A-2 Wt. Ftr. _____ Lbs. _____
13.	Bring A-2 to 110 RPM. Hold 2 min. Plug to stop. Repeat twice.	Time first slurrying _____ Time second _____ Time third _____
14.	With A-2 stopped, jet A-2 to A-4.	Time jet _____
15.	Jet 325 lbs. from A-1 to A-2.	A-2 Wt. Ftr. _____ Lbs. _____

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CONCENTRATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

IV - PHOSPHATE BY-PRODUCT CAKE REMOVAL (Cont'd)

16. Bring A-2 to 110 RPM. Hold 2 min. Plug to a stop. Repeat twice.
Time first slurrying _____
Time second _____
Time third _____

17. With A-2 stopped, jet A-2 to A-4
Time jet _____

(Note: Hold A-4 temperature at 40-50°C during items 18 through 26)

18. Jet the remaining acid from A-1 to A-2 at full jet pressure. As A-2 weight exceeds 400 lbs., jet the excess acid above 400 lbs. directly from A-2 to A-4.
A-2 Wt. Ftr. _____ Lbs. _____
A-1 jet pressure _____

19. Bring A-2 to 110 RPM. Hold 2 min. Plug to stop. Repeat twice.
Time first slurrying _____
Time second _____
Time third _____

20. With A-2 stopped, jet A-2 to A-4.
Time jet _____

21. Beckman on A-2.
Meter _____ Factor _____

22. Note: If Beckman reading is higher than limit, add two 250 lb. portions of 60% nitric acid to A-2 via A-2C. Repeat items 19 and 20 after each acid addition. Notify Cell C supervisor that extra acid has been used in A-2 cake removal. (Approx. 500 lbs.)
A-2 Beckman limit 20 X10 -14
Total lbs. extra nitric acid. _____
Final A-2 Beckman Meter X10

23. Agitate A-4 for 1 hour at 40-50°C.
Time start _____ Time end _____
Max. Temp. _____ Min. Temp. _____

24. Beckman on A-4.
Meter _____ Factor _____

[redacted]

[redacted]

CONCENTRATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

IV - PHOSPHATE BY - PRODUCT CAKE REMOVAL (cont'd)

25.	Stop A-4 agitator and read Wt. Ftr. and Sp. Gr. when constant	A-4 agitator off _____ A-4 Wt. Ftr. _____Lbs._____ A-4 Sp. Gr. _____Temp._____
26.	Start A-4 agitator.	A-4 agitator on _____
27.	Start full A-4 cooling water.	A-4 water on _____
28.	Take sample A-4 BP.	Time sampled _____
29.	Stop A-4 cooling water when temperature reaches minimum.	A-4 water off _____
30.	With supervisor's approval, jet A-4 to C-8.	Time OK to proceed _____ Time start jet _____
31.	When A-4 Wt. Ftr. pen reaches 0.2 stop A-4 agitator.	A-4 agitator off _____
32.	When jet passes, stop jet.	Time stop jet _____
33.	Beckman on A-4.	Meter _____Factor _____

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[redacted]

Date _____
Run No. _____

DECLASSIFIED

E-4 RECYCLE RECORD

A. E-4 SAMPLING

ITEM	SYMBOL	FORMULA	READING OR CALCULATION	UNITS
1. E-4 Wt. Ftr.	***	* * * * * *		* *
2. Total Weight	W	* * * * * *		Lbs.
3. E-4 Specific Gravity	SG	* * * * * *		* *
4. Time E-4-RC Sampled	* * *	* * * * * *		* *

B. LANTHANUM INVENTORY

1. Lanthanum Assay	La	* * * * * *		g/l
2. Total Wt. of La in E-4	M	$M=\frac{(W)\ (La)\ X10^{-3}}{(SG)}$		lbs.
3. Calc. Wt. of Solution to jet to D-1 (See note)	R	$R=\frac{9.0 \times W}{M}$		lbs.

Note: If the jetting of 9.0 lbs. of lanthanum from E-4 will exceed the product limit in D-1, tailor between 6,0 and 0.9 lbs. (Formula B-3)as required.

C. E-4 INVENTORY AFTER JETTING TO D-1

1. Time Jetted to D-1	* * *	* * * * * *		* *
2. E-4 Wt. Ftr. After jetting	* * *	* * * * * *		* *
3. Total Wt. Remaining in E-4	Z	* * * * * *		Lbs.
4. Net Wt. Jetted to D-1	RC	RC = W-Z		Lbs.

Signed by: --
Supervisor

[redacted]

[redacted]

CONCENTRATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

Note: Pages 8.1 and 8.2 to be used instead of pages 8.1A and 8.2A when no recycle is added.

I - FLUORIDE BY-PRODUCT OXIDATION AND PRECIPITATION

1. Beckman on D-1 — Meter _____ Factor _____

2. Start D-1 agitator — D-1 agitator on _____

3. Receive approximately 12,000 lbs. of solution from A-3 into D-1. Start jacket steam to bring D-1 temp. to 75°C while receiving solution. (Proceed with item 6 while items 3 and 4 in progress.) — Time start jetting _____; Time steam on _____; Time end jetting _____

4. Finish adjusting D-1 to 75°C. — Time 75°C _____

5. Beckman on D-1 — Meter _____ Factor _____

6. Make up 390 lbs of 5% potassium permanganate in D-1-B
   a. Add 315 lbs. of water — Lbs. water add _____ Time _____
   b. Start agitator — Time agitator on _____
   c. Heat to 40°C with steam sparger — Time 40°C _____
   d. Over 10 to 15 minutes add 20 lbs. of potassium permanganate. — Time start _____ Time end _____; Lbs. add _____
   e. Continue heating and agitate for 30 minutes or until solution is complete. DO NOT EXCEED 75°C. — Time end _____
   f. Add water to make 390 lb total. — Lbs. water add _____; Lbs. total _____
   g. Take sample to lab for approval — Sampled by _____; Time OK'd by lab _____

7. With supervisor's approval, add the 5% permanganate solution from D-1B to D-1. — Time OK to proceed _____; Lbs. add _____ Time _____

8. Flush D-1B to D-1 with 50 lbs. of water. — Lbs. add _____ Time _____

9. Agitate D-1 for 30 min. at 75°C — Time start _____ Time end _____

10. Cool D-1 to 35°C (Proceed with item 14, p 8.2 while item 10 is in progress). — Time start _____ Time 35°C _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

I - FLUORIDE BY-PRODUCT OXIDATION AND PRECIPITATION (Cont'd)

11.	Stop D-1 agitator and read Wt. Ftr. and Sp. Gr. when constant.	D-1 agitator off _____ D-1 Wt. Ftr. _____Lbs._____ D-1 Sp. Gr. _____Temp._____
12.	Start D-1 agitator	D-1 Agitator on _____
13.	Add 53 lbs. of hydrofluoric acid to D-1 via D-1Y at approx. 10 lbs./min. Blow the line with air for 5 min. following the acid addition.	Time start _____Time end _____ Lbs. start _____Lbs. end _____ Lbs. add _____ Time line blown _____
14.	Make up 221 lbs. of approximately 10% Lanthanum salt solution in D-1-B	
	a. Add 160 lbs. of water	Lbs. water add _____Time _____
	b. Start agitator	Time agitator on _____
	c. Add 37 lbs. of 60% nitric acid (Line A)	Lbs. nitric added _____ Time added _____
	d. Add 25 lbs. Lanthanum salt	Lbs. Lanthanum salt added Time Lbs. total _____
	e. Agitate until dissolved	Time end _____
	f. Take sample to lab for approval	Sampled by _____ Time OK'd by lab _____
15.	Add the above 10% Lanthanum salt solution to D-1 at 10 lbs per min.	Time start _____Time end _____ Lbs. add _____
16.	Flush D-1B to D-1 with 100 lbs. of water.	Lbs. added _____Time added _____
17.	Agitate D-1 for one hour at 35°C DO NOT EXCEED 35°C. (Proceed with item 10, page 8.3)	Time start _____Time end _____
18.	Beckman on D-1.	Meter _____Factor _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

Note: Pages 8.1A and 8.2A to be used instead of pages 8.1 and 8.2 when recycle is added.

I - FLUORIDE BY-PRODUCT (PRODUCTION RUN RECYCLE) OXIDATION AND PRECIPITATION

1. Beckman on D-1 — Meter _____ Factor _____

2. Start D-1 agitator — D-1 agitator on _____

3. Receive approx. 12,000 lbs. of solution from A-3 into D-1. Start jacket steam to bring D-1 temp. to 75°C while receiving solution. (Proceed with item 7 while items 3, 4, and 5 in progress.)
   - Time start jetting _____
   - Time steam on _____
   - Time end jetting _____

4. With supervisor's approval, receive recycle solution into D-1 from E-4
   Note: Care must be taken to receive as closely as possible the amount calculated by supervisor.
   - Time OK to proceed _____
   - Time received _____

5. Finish adjusting D-1 to 75°C — Time 75°C _____

6. Beckman on D-1. — Meter _____ Factor _____

7. Make up 390 lbs of 5% potassium permanganate in D-1B.
   a. Add 315 lbs. of water — Lbs. water add _____ Time _____
   b. Start agitator — Time agitator on _____
   c. Heat to 40°C with steam sparger — Time 40°C _____
   d. Over 10 to 15 minutes add 20 lbs of potassium permanganate — Time start _____ Time end _____ Lbs. add _____
   e. Continue heating and agitate 30 minutes or until solution is complete. DO NOT EXCEED 75°C. — Time end _____
   f. Add water to make 390 lb. total — Lbs. water add _____ Time _____ Lbs. total _____
   g. Take sample to lab for approval. — Sampled by _____ Time OK'd by lab _____

8. With supervisor's approval, add the 5% permanganate solution from D-1B to D-1.
   - Time OK to proceed _____
   - Lbs. add _____ Time _____

9. Flush D-1B to D-1 with 50 lbs. of water. — Lbs. add _____ Time _____

10. Agitate D-1 for 30 min. at 75°C. — Time start _____ Time end _____

11. Cool D-1 to 35°C. — Time start _____ Time 35°C _____

[redacted]

[redacted]

CONCENTRATION OPERATIONS

Date _____
Run No. _____

DECLASSIFIED

I - FLUORIDE BY-PRODUCT OXIDATION AND PRECIPITATION (Cont'd.)

12. Stop D-1 agitator and read Wt. Ftr. and Sp. Gr. when constant.

D-1 agitator off _____

D-1 Wt. Ftr. _____Lbs. _____

D-1 Sp. Gr. _____Temp. _____

13. Start D-1 agitator

D-1 agitator on _____

14. Add 53 lbs. hydrofluoric acid to D-1 via D-1-Y at the rate of 3 lb per minute. Blow the line with air for 5 minutes following the acid addition.

Time start ____Time end _____

Lbs. start ____Lbs. end_____

Lbs. add _____

Time line blown_____

15. Agitate D-1 for one hour at 35°C
DO NOT EXCEED 35°C
(Proceed with item 10, page 8.3)

Time start _____Time end _____

16. Beckman on D-1

Meter _____Factor _____

[redacted]

[redacted]

Date _____

Run No. _____

DECLASSIFIED

II - FLUORIDE BY-PRODUCT CENTRIFUGING

1. Bring D-2 to 1740 RPM — Time 1740 RPM _____

2. Beckman on D-2 — Meter _____ Factor _____

3. Beckman on D-3 — Meter _____ Factor _____

4. Start D-3 cooling water and air sparger. — D-3 water on _____ D-3 sparger on _____

5. Transfer slurry from D-1 to D-2 at the rate of 70 lbs. per min. Check the transfer rate at 30 minute intervals, recording data in table. (Notify Cell E operator to start item 5, page 8.7, while centrifuging)

Time	Jet Press	D-3 Wt.	Lbs/Min.
		0	-----

6. Stop D-1 agitator when Wt. Ftr. pen reaches 0.2. — D-1 agitator off _____

7. Just as jet from D-1 to D-2 first gases, raise jet steam pressure to full. Stop jet when D-2 ammeter and jet Thermohm indicate D-1 is empty. — Time jet gases _____ Time full pressure _____ P. S. I. _____ Time stop jet _____

8. Slow D-2 to 870 RPM; plug to 650 RPM and inch. — Time 870 RPM _____ Time 650 RPM _____

9. Five min. after completing item 8, skim to 10 gal. Leave D-2 at 870 RPM. — Time end _____

10. Make up 500 lbs. of 6% Nitric Acid in D-1-B.
    a. Add 450 lbs. water — Lbs. water added ____ Time _____
    b. Start agitator — Time agitator on _____
    c. Add 50 lbs. of 60% Nitric Acid (Line A) — Lbs. Nitric added ____ Time _____
    d. Take sample to lab for approval — Sampled by _____ Lbs. total _____ Time OK'd by lab. _____

11. Add the 500 lbs. of 6% nitric acid to D-1 via D-1B. — Time add _____ Lbs. add _____

11A. Bring D-2 to 1740 RPM — Time 1740 RPM _____

12. Circulate thru D-1 distributor at 50 psi for 3 min. with cooling water on jacket. — Time start _____ Time end _____ P. S. I. _____

13. Stop D-1 cooling water. — D-1 water off _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

II - FLUORIDE BY-PRODUCT CENTRIFUGING (Cont'd)

14. With D-2 at 1740 RPM, jet all of nitric wash from D-1 to D-2 at full jet pressure.
Time start ____ Time end ____
Jet pressure _____

15. Slow D-2 to 870 RPM; plug to 650 RPM and inch.
Time 870 RPM _____
Time 650 RPM _____

16. Five min. after completing item 15, skim D-2 to 10 gal. Leave D-2 at 870 RPM.
Time end _____

17. Stop D-3 sparger and read Sp. Gr. and Wt. Ftr. when constant.
Time sparger off _____
Wt. Ftr. _____ Lbs. _____
Sp. Gr. _____ Temp. _____

18. Start D-3 air sparger.
Time sparger on _____

19. Beckman on D-2 and D-3.
D-2 Meter _____ Factor _____
D-3 Meter _____ Factor _____

20. Obtain supervisor's approval that D-3 Beckman reading is satisfactory
D-3 Beckman limit _____
(Less than 1/5 of item 5, page 8.1A or item 6, page 8.1A)
Approved by _____ Time _____
Supervisor

21. Stop D-3 cooling water when temperature falls to a minimum if D-3 to E-1 movement is held up.
D-3 water off _____
D-3 Minimum temp. _____

NOTE: If D-3 can not be jetted to E-1 at this point proceed with D-2 cake removal, page 8.5.

22. With supervisor's approval, jet D-3 to E-1.
Time OK to proceed _____
Time start _____

23. When D-3 Wt. Ftr. pen reaches 0.2, stop D-3 sparger.
D-3 sparger off _____

24. When jet from D-3 to E-1 gasses, shut it off.
Time off _____

25. Stop D-3 cooling water, if still on.
Time water off _____

26. Beckman on D-1 and D-3.
D-1 Meter _____ Factor _____
D-3 Meter _____ Factor _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

II - FLUORIDE BY-PRODUCT CENTRIFUGING (Cont'd)

14.	Plug D-2 to stop.	D-2 stopped _____
15.	Jet 250 lbs. from D-1 to D-2	D-2 Wt. Ftr. _____ Lbs. _____ Time _____
16.	Bring D-2 to 110 RPM, hold 2 min. Plug to stop. Repeat twice.	Time start _____ Time end _____
17.	Bring D-2 to 1740 RPM, hold 10 min.	Time start _____ Time end _____
18.	Slow D-2 to 870 RPM; Plug to 650 RPM and inch	Time 870 RPM _____ Time 650 RPM _____
19.	Five minutes after completing item 18, skim to 10 gal.	Time end _____
20.	Plug D-2 to stop.	D-2 stopped _____
21.	Jet remainder of acid from D-1 to D-2 at full jet pressure. (When jet gasses, stop jet.)	D-2 Wt. Ftr. _____ Lbs. _____ P.S.I. _____ Time _____

NOTE: If D-2 weight should exceed 400 lbs. stop jet. Carry out third slurrying with remainder.

22.	Bring D-2 to 110 RPM, hold 2 min. Plug to stop. Repeat twice.	Time start _____ Time end _____
23.	Bring D-2 to 1740 RPM, hold 10 min.	Time start _____ Time end _____
24.	Slow D-2 to 870 RPM; Plug to 650 RPM and inch .	Time 870 RPM _____ Time 650 RPM _____
25.	Five minutes after item 24 above, skim D-2 to 10 gals. Leave D-2 at 870 RPM.	Time end _____

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[redacted]

Date _____
Run No. _____

DECLASSIFIED

II - FLUORIDE BY-PRODUCT CENTRIFUGING (Cont'd)

26.	Stop D-3 sparger and read Sp. Gr. and Wt. Ftr. when constant.	Time sparger off _____ Wt. Ftr. _____ Lbs. _____ Sp. Gr. _____ Temp. _____
27.	Start D-3 air sparger.	Time sparger on _____
28.	Beckman on D-3 and D-2.	D-2 Meter _____ Factor _____ D-3 Meter _____ Factor _____
29.	Obtain supervisor's approval that D-3 Beckman reading is satisfactory.	D-3 Beckman limit _____ (Less than 1/5 of item 5, page 8.1 or item 6, page 8.1A) Approved by _____ Time _____ Supervisor
30.	Stop D-3 cooling water when temperature falls to a minimum if D-3 to E-1 movement is held up.	D-3 Water off _____ D-3 Minimum temp. _____
NOTE:	If D-3 can not be jetted to E-1 at this point proceed with D-2 cake removal, page 8.5.	
31.	With supervisor's approval, jet D-3 to E-1.	Time OK to proceed _____ Time start _____
32.	When D-3 Wt. Ftr. pen reaches 0.2, stop D-3 sparger.	D-3 sparger off _____ _____
33.	When jet from D-3 to E-1 gasses, shut it off.	Time off _____
34.	Stop D-3 cooling water, if still on.	Time water off _____
35.	Beckman on D-1 and D-3.	D-1 Meter _____ Factor _____ D-1 Meter _____ Factor _____

Date _____
Run No. _____

DECLASSIFIED

III - FLUORIDE BY-PRODUCT CAKE REMOVAL

1.	Start D-4 agitator and cooling water.	D-4 agitator on _____ D-4 cooling water on _____
2.	Beckman on D-4.	Meter _____ Factor _____
3.	Plug D-2 to stop and read Wt. Ftr.	D-2 stopped _____ D-2 Wt. Ftr. _____ Lbs. _____
4.	Bring D-2 to 10 RPM, start D-2 to D-4 jet, and add approx. 2000 lbs. of water from D-2D through bowl sprays at not less than 70 psi (If D-2 manometer exceeds 3.0, stop sprays temporarily until bowl is empty.) Stop jet at end	D-2 at 10 RPM _____ Time start jet _____ Time stop jet _____ Pump pressure _____ Lbs. water added _____
5.	Plug D-2 to stop.	Time D-2 stopped _____
6.	With D-2 stopped add 250 lbs. of water from D-2D through Bowl sprays.	D-2 Wt. Ftr. _____ Lbs. _____ Time add _____
7.	Bring D-2 to 110 RPM, hold 2 min. plug to stop. Repeat once.	Time first slurrying _____ Time second _____
8.	Bring D-2 to 110 RPM and hold 2 min. Start D-2 to D-4 jet. Plug D-2 to stop. When jet gasses, stop jet.	Time jet on _____ Time D-2 stopped _____ Time jet off _____
9.	With D-2 stopped add 250 lbs. of water from D-2D through bowl sprays.	D-2 Wt. Ftr. _____ Lbs. _____ Time add _____
10.	Bring D-2 to 110 RPM and hold 2 min. Plug to stop. Repeat once.	Time first slurrying _____ Time second _____
11.	Bring D-2 to 110 RPM and hold 2 min. Start D-2 to D-4 jet. Plug D-2 to stop. When jet gasses, stop jet.	Time jet on _____ Time D-2 stopped _____ Time jet off _____
12.	With D-2 stopped add 250 lbs. of water from D-2D through bowl sprays.	D-2 Wt. Ftr. _____ Lbs. _____ Time add _____
13.	Bring D-2 to 110 RPM and hold 2 min. Plug to stop. Repeat once.	Time first slurrying _____ Time second _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

III - FLUORIDE BY-PRODUCT CAKE REMOVAL (Cont'd.)

14.	Bring D-2 to 110 RPM and hold 2 min. Start D-2 to D-4 jet. Plug D-2 to stop. When jet gases,. stop jet.	Time jet on _____ Time D-2 stopped _____ Time jet off _____
15.	With D-2 stopped, start D-2 to D-4 jet and add 300 lbs. of water to D-2 from D-2D through bowl sprays at not less than 70 psi. When jet gases stop jet.	Time start jet _____ Pump pressure _____ Lbs. water add _____ Time stop jet. _____
16.	Beckman on D-2.	Meter _____ Factor _____
17.	Note: If Beckman reading is higher than limit, repeat items 9 through 15 once more. Notify Cell C supervisor that extra water (approximately 800 lbs.) has been used in D-2 cake removal.	D-2 Beckman limit 20 X10 -14 Total lbs. extra water _____ Final D-2 Beckman Meter. Factor
18.	Beckman on D-4.	Meter _____ Factor _____
19.	Stop D-4 agitator and read Wt. Ftr. and Sp. Gr. when constant.	D-4 Agitator off _____ D-4 Wt. Ftr. _____ Lbs. _____ D-4 Sp. Gr. _____ Temp. _____
20.	Start D-4 agitator	D-4 agitator on _____
21.	Take sample D-4 BP.	Time sampled _____
22.	Stop D-4 cooling water.	D-4 Water off _____
23.	With supervisor's approval, jet D-4 to C8, stopping agitator when Wt. Ftr. pen reaches 0.2.	Time OK to proceed _____ Time start _____ Time agitator off _____
24.	Just as D-4 to C-3 jet gasses (heard over speaker), stop jet.	Time jet off _____
25.	Beckman on D-4.	Meter _____ Factor _____

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[redacted]

Date _____
Run No. _____

DECLASSIFIED

IV - FLUORIDE PRODUCT REDUCTION AND FIRST PRECIPITATION

1. Beckman on E-1. — Meter_____ Factor_____

2. Start E-1 agitator. — Time start_____

3. Receive approximately 14,000 lbs. of filtrate from D-3 into E-1 (Start adjusting E-1 temperature to 35°C while receiving solution.) — Time start_____ Time end_____

4. Beckman on E-1. — Meter_____ Factor_____

5. Make up 1435 lbs. of 9.6% oxalic acid solution in E-1-E.
   a. Add 1180 lbs. of water — Lbs. water added_____ Time_____
   b. Start agitator — Time agitator on _____
   c. Add 138 lbs. of oxalic acid — Lbs. oxalic acid added- Time_____
   d. Start sparger and heat to 65°C. — Time sparger on_____ Time 65°C_____
   e. Make up to 1435 lbs. with water — Lbs. water make-up_____ Lbs. Total_____
   f. Take sample to lab for approval — Sampled by_____ Time OK'd by lab_____

6. With supervisor's approval, add the 9.6% oxalic acid solution to E-1 via E-1E, while adjusting E-1 temperature to 35°C. — Time add_____ Lbs. add_____

7. Flush E-1E to E-1 with 100 lbs. of water. — Time add_____ Lbs. add_____

8. Agitate E-1 for 1 hour. When E-1 temperature reaches 35°C, hold at 35°C. (Proceed with item 9 while 8 in progress.) — Time start_____ Time end_____ Time 35°C _____

9. Make up 1,000 lbs. of approximately 1½% lanthanum salt solution.
   a. Add 967 lbs. of water — Lbs. water added_____ Time_____
   b. Start agitator. — Time agitator on _____
   c. Add 17 lbs. of 60% nitric acid (line A). — Lbs. nitric added_____ Time_____
   d. Add 16 lbs.-8oz. of lanthanum salt. — Lbs. lanthanum salt added_____ Time_____ Lbs. total _____
   e. Agitate until dissolved. — Time dissolved _____
   f. Take sample to lab for approval — Sampled by_____ Time OK'd by lab_____

10. Stop E-1 agitator and read Wt. Ftr. and Sp. Gr. when constant. — E-1 agitator off _____ E-1 Wt. Ftr._____ Lbs._____ E-1 Sp. Gr._____ Temp._____

11. Start E-1 agitator. — E-1 agitator on _____

11A Take sample E 1-R — Time sampled_____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

IV - FLUORIDE PRODUCT REDUCTION AND FIRST PRECIPITATION (Cont'd)

12. Add 120 lbs. of Hydrofluoric Acid to E-1 via E-1-Y at approx. 10 lbs./min. (Use cooling water as necessary to maintain E-1 temperature at 35°C) Blow line out with air for 5 minutes after the acid addition.

Time start_____ Time end_____

Lbs. start_____ Lbs. end_____

Lbs. added_____

Time line blown_____

13. Add 500 lbs. of 1½ Lanthanum salt solution to E-1 via E-1E at 25 lbs. per min.

Time start_____ Time end_____

Lbs. start_____ Lbs. end_____

Lbs. add_____

14. Agitate 1 hour at 35°C. DO NOT EXCEED 35°C.

Time start_____ Time end_____

15. Beckman on E-1.

Meter_____ Factor_____

16. Start E-1 cooling water.

E-1 water on _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

V - FLUORIDE PRODUCT FIRST CENTRIFUGING

1. Beckman on E-3. — Meter _____ Factor _____

2. Beckman on E-2 at 30 RPM. — Meter _____ Factor _____

3. Obtain supervisor's approval to start centrifuging — E-2 Beckman limit 5 x 10 - 14
F-1-PS Assay, Previous Run _____ %
Approved by _____ Time _____
Supervisor

4. Bring E-2 to 1740 RPM. — Time 1740 RPM _____

5. Start E-3 cooling water and air sparger. — Water on _____ Sparger on _____

6. Transfer slurry from E-1 to E-2 at the rate of 70 lbs. per min Check the transfer rate at 30 min. intervals, recording data in table.

Time	Jet Press	E-3 Wt.	lbs/min.
		0	

7. Stop E-1 agitator when Wt. Ftr. pen reaches 0.2. — E-1 agitator off _____

8. Stop E-1 cooling water. — E-1 water off _____

9. Just as jet from E-1 to E-2 first gases, raise jet steam pressure to full. Stop jet when E-2 ammeter and jet Thermohm indicate E-1 is empty. — Time jet gasses _____
Time full pressure _____
P.S.I. _____

10. Slow E-2 to 870 RPM; Plug to 650 RPM and inch. — Time 870 RPM _____
Time 650 RPM _____

11. Five minutes after item 10 above, skim E-2 to 10 gals. Leave E-2 at 870 RPM. — Time end _____

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Date _____
Run No. _____

DECLASSIFIED

VI - FLUORIDE PRODUCT SECOND PRECIPITATION

1.	Start E-1 agitator.	E-1 agitator on _____
2.	Beckman on E-1.	Meter_____Factor_____
3.	Jet E-3 to E-1. Start adjusting E-1 temp. to 35°C, while receiving the solution. When E-3 Wt. Ftr. pen reaches 0.2, stop E-3 sparger.	Time start jet_____ Time stop sparger_____
4.	When jet gases, stop jet and E-3 cooling water.	Time stop jet _____ E-3 water off _____
5.	Add 500 lbs. of 1½% Lanthanum salt solution to E-1 via E-1E at 25 lbs. per min. while adjusting E-1 temperature to 35°C.	Time start_____Time end_____ Lbs. add_____
6.	Flush E-1E to E-1 with 100 lbs. of water.	Time add_____Lbs.add_____
7.	Agitate E-1 for 1 hour. When E-1 temperature reaches 35°C, hold at 35°C. (Proceed with item 11, page 8.11)	Time start_____Time end_____ Time 35°C _____
8.	Start E-1 cooling water.	Time water on _____
9.	Beckman on E-1.	Meter_____Factor_____
10.	Stop E-1 Agitator and read Wt. Ftr. and Sp. Gr. when constant.	E-1 Agitator off _____ E-1 Wt. Ftr._____Lbs._____ E-1 Sp. Gr. _____Temp._____
11.	Start E-1 agitator.	E-1 agitator on _____

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[redacted]

Date _____
Run No. _____

DECLASSIFIED

VII - FLUORIDE PRODUCT SECOND CENTRIFUGING

1. Bring E-2 to 1740 RPM. — Time 1740 RPM _____

2. Start E-3 cooling water and air sparger. — E-3 water on _____ Sparger _____

3. Transfer slurry from E-1 to E-2 at the rate of 70 lbs. per min. Check the transfer rate at 30 minute intervals, recording data in table. (Proceed with item 1, page 9.4, while centrifuging.) and also item 5, page 8.13C)

Time	Jet Press	E-3 Wt.	lbs./min.
		0	

4. Stop agitator on E-1 when Wt. Ftr. pen reaches 0.2. — Time off _____

5. Just as jet from E-1 to E-2 first gasses, raise jet steam pressure to full. Stop jet and E-1 cooling water when E-2 ammeter and jet Thermohm indicate E-1 is empty.
   Time jet gasses _____
   Time full pressure _____
   P.S.I. _____
   Time jet off _____
   Time water off _____

6. Slow E-2 to 870 RPM; Plug to 650 RPM and inch.
   Time 870 RPM _____
   Time 650 RPM _____

7. Five min. after completing item 6, skim to 10 gal. Leave E-2 at 870 RPM. — Time end _____

8. Stop E-3 sparger and read Sp. Gr. and Wt. Ftr. when constant.
   Time sparger off _____
   Wt. Ftr. _____ Lbs. _____
   Sp. Gr. _____ Temp. _____

9. Start E-3 air sparger — Time sparger on _____

10. Take sample E-3 W — Time sampled _____

11. Make up 500 lbs. of 6% nitric acid in E-1-C.
    a. Add 540 lbs. water — Lbs. water added _____ Time _____
    b. Start sparger — Time sparger on _____
    c. Add slowly 50 lbs. of 60% nitric acid (Line A) — Lbs. nitric acid added _____ Time _____ Lbs. total _____
    d. Take sample to lab for approval — Sampled by _____ Time OK'd by lab _____

12. Add the 500 lbs. of 6% Nitric to E-1 via E-1C. — Time added _____ Lbs. added _____

13. Bring E-2 to 1740 RPM — Time 1740 RPM _____

[redacted]

Date_____
Run No. _____

DECLASSIFIED

VII - FLUORIDE PRODUCT SECOND CENTRIFUGING (Cont'd.)

14.	Circulate through E-1 distributor 50 psi for 3 min. with cooling water on jacket.	Time start_____Time end_____ P.S.I._____
15.	Stop E-1 cooling water.	E-1 water off _____
16.	With E-2 at 1740 RPM, jet all of nitric wash from E-1 to E-2 at full jet pressure	Jet pressure _____
17.	Slow E-2 to 870 RPM; Plug to 650 RPM and inch.	Time 870 RPM_____ Time 650 RPM_____
18.	Five minutes after item 27 above, skim E-2 to 10 gals. Leave E-2 at 870 RPM.	Time end_____
19.	Stop E-3 sparger and read Sp.Gr. and Wt. Ftr. when constant.	Time sparger off _____ Wt. Ftr._____Lbs._____ Sp. Gr._____Temp._____
20.	Start E-3 air sparger	Time sparger on _____
21.	Stop E-3 cooling water when temperature reaches a minimum.	E-3 water off _____ Minimum temp._____
22.	Beckman on E-1, E-2, and E-3.	E-1 Meter_____Factor_____ E-2 Meter_____Factor_____ E-3 Meter_____Factor_____
23.	With supervisor's approval, jet waste from E-3 to C-8.	Time OK to jet_____ Time start_____
24.	When E-3 Wt. Ftr. pen reads 0.2, stop E-3 sparger.	E-3 sparger off _____
25.	When E-3 to C-8 jet gases, stop jet.	Jet off _____
26.	Beckman on E-3.	Meter_____Factor_____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VIII - FLUORIDE PRODUCT PRODUCT CAKE REMOVAL

1. With supervisor's approval, plug E-2 tp stop and read Wt. Ftr.
   Time OK to proceed _____
   Time E-2 stopped _____
   E-2 Wt. Ftr. _____ Lbs. _____

2. Bring E-2 to 10 RPM, start E-2 to F-1 jet and add 700 lbs. of water from E-2D through bowl sprays at not less than 70 psi. (If manometer exceeds 3.0, stop sprays temporarily until bowl is empty) Plug E-2 to Stop.
   E-2 at 10 RPM _____
   Time start jet _____
   Pump pressure _____
   Lbs. water added _____
   E-2 stopped _____

3. Stop E-2 jet when E-2 is empty.
   Time stop jet _____

4. Beckman on E-2 at 30 RPM.
   E-2 Meter _____ Factor _____

5. Make up 722 lbs. of 50% Potassium Hydroxide in E-A.
   a. Add 297 lbs. of water
      Lbs. water add _____ Time _____
   b Start agitator
      Time agitator on _____
   c. Add 425 lbs. of Potassium Hydroxide.
      Lbs. Potassium Hydroxide add _____
      Lbs. total _____
   d. Continue agitation until solid is dissolved
      Time dissolved _____
      Temperature _____
   e. Take sample to lab for approval
      Sampled by _____
      Time OK'd by lab _____

6. Transfer 50% Potassium Hydroxide from E-A to E-2C using the E-A pump.
   Time transferred _____

7. With E-2 stopped, add 361 lbs. of hot 50% Potassium Hydroxide to E-2 from E-2-C.
   Lbs. Potassium Hydroxide add _____
   Time _____
   Temp. Potassium Hydroxide _____

8. Bring E-2 to 110 RPM and hold 2 min. Plug to stop. Repeat three times.
   Time first slurrying ___ Time 2nd ___
   Time 3rd _____ Time 4th _____

9. Bring E-2 to 110 RPM and hold 2 min. Start E-2 to F-1 jet. Plug to stop.
   Time jet on _____
   E-2 stopped _____

10. Stop E-2 jet when E-2 is empty.
    Time stopped _____

11. Beckman on E-2 at 30 RPM.
    Meter _____ Factor _____

12. With E-2 stopped, add 361 lbs. of hot 50% Potassium Hydroxide to E-2 from E-2C
    Lbs. Potassium Hydroxide add _____
    Time _____
    Temp. Potassium Hydroxide _____

[redacted]

[redacted]

Date_____
Run No. _____

DECLASSIFIED

VIII - FLUORIDE PRODUCT PRODUCT CAKE REMOVAL (Cont'd)

13.	Bring E-2 to 110 RPM and hold 2 min. Plug to stop. Repeat three times.	Time first slurrying__ Time 2nd____ Time 3rd_____ Time 4th_____
14.	Bring E-2 to 110 RPM and hold 2 min. Start E-2 to F-1 jet. Plug to stop.	Time jet on _____ E-2 stopped_____
15.	Stop E-2 jet when E-2 is empty.	Time stopped_____
16.	Beckman on E-2 at 30 RPM.	Meter_____ Factor_____
17.	Flush E-2-C to E-2 with 200 lbs. of water.	Lbs. water add_____ Time_____
18.	Bring E-2 to 110 RPM and hold 2 min. Start E-2 to F-1 jet. Plug to stop.	Time jet on _____ E-2 stopped_____
19.	Stop E-2 jet when E-2 is empty.	Time stopped_____
20.	With E-2 at 10 RPM and with the E-2 to F-1 jet on add sufficient water from E-2-D through the bowl sprays to E-2 to F-1 to bring the total weight in F-1 to 2400 lbs. (approx. 500 lbs.) When jet goes stop jet.	Lbs. water add_____ Pump pressure_____ Time jet off_____
21.	Beckman on E-2 at 30 RPM.	Meter_____ Factor_____
22.	Obtain supervisor's approval that E-2 Beckman reading is satisfactory.	E-2 Beckman limit 20×10^{-14} Approved by _____ Time_____ Supervisor

[redacted]

Date_____
Run No. _____

DECLASSIFIED

I - METATHESIS

1.	Read F-1 Wt. Ftr.	F-1 Wt. Ftr._____Lbs._____
2.	Beckman on F-1.	F-1 Meter_____Factor_____
3.	Obtain supervisor's approval that F-1 Beckman in satisfactory.	F-1 Beckman Limit 20×10^{-14} Approved by_____Time_____ Supervisor
4.	Start agitator in F-1.	Time F-1 agitator on_____
5.	Receive sufficient weight of slurry from E-2 into F-1 to bring F-1 total weight to 2400 lbs.	Time start_____Time end_____
6.	Beckman on F-1.	Meter_____Factor_____
7.	Stop F-1 agitator and read Wt. Ftr. and Sp. Gr. when constant	F-1 agitator off _____ Wt. Ftr._____Lbs._____ Sp. Gr._____Temp._____
8.	Start F-1 agitator.	F-1 agitator on _____
9.	Take sample F-1-PS	Time sampled_____
10.	Adjust F-1 temp. to 80°C and agitate 90 minutes at 80°C.	Time 80°C _____ Time start_____Time end_____
11.	Cool F-1 to 35°C and leave cooling water on.	Time start_____Time 35°C _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

II - METATHESIS CENTRIFUGING

1. Beckman on F-2.
   Meter _____ Factor _____
2. Obtain supervisor's approval to start centrifuging.
   F-2 Beckman Limit 20×10^{-14}
   Approved by: _____ Time _____
   Supervisor
3. Bring F-2 to 1800 RPM.
   Time 1800 RPM _____
4. Start F-9 sparger and cooling water.
   F-9 sparger on _____
   Water on _____
5. Transfer slurry from F-1 to F-2 at the rate of 12 lbs. per min. Check the transfer rate at 30 minute intervals, recording data in table.

Time	Jet Press	F-9 Wt.	lbs./min.
		0	

6. Stop F-1 agitator when F-1 Wt. Ftr. pen reaches 0.8 and start F-1 air sparger.
   F-1 agitator off _____
   F-1 sparger on _____
7. When F-1 Wt. Ftr. pen reaches zero, stop F-1 air sparger.
   F-1 sparger off _____
8. When the jet from F-1 to F-2 first gasses, raise jet steam pressure to full. Stop jet when ammeter and jet therometer indicate F-1 is empty.
   Time full pressure _____
   P. S. I. _____
   Time stop jet _____
9. After 5 min., at 1800 RPM, skim F-2 to 5 gal. Stop cooling water on F-1.
   Time and skim _____ - _____
   F-1 water off _____
10. Slow F-2 to 900 RPM.
    F-2 at 900 RPM. _____
11. Beckman on F-2.
    F-2 Meter _____ Factor _____
12. Start F-7 agitator and jet F-9 to F-7.
    F-7 agitator on _____
    Time start jet _____
13. When F-9 Wt. Ftr., pen reaches 0.2, stop F-9 sparger and cooling water.
    F-9 sparger off _____
    F-9 water off _____
14. When F-9 to F-7 jet gasses, stop jet.
    Time stop jet _____
15. Stop F-7 agitator and read F-7 Wt. Ftr. and Sp. Gr.
    F-7 agitator off _____
    F-7 Wt. Ftr. _____ Lbs. _____
    F-7 Sp. Gr. _____ Temp _____
16. Start F-7 agitator
    F-7 agitator on _____
17. Take sample F-7 WS.
    Time sampled _____

[redacted]

Date_____
Run No. _____

DECLASSIFIED

III - FIRST CAKE REMOVAL

1.	Start F-1 agitator and cooling water.	F-1 agitator on _____ F-1 cooling water_____
2.	Plug F-2 to stop and read manometer.	F-2 stopped_____ F-2 Manometer_____Lbs._____
3.	Start F-2 to F-1 jet and bring F-2 to 10 RPM.	Time start jet_____ F-2 at 10 RPM _____
4.	Add 450 lbs. of water from F-2D through bowl sprays (low pressure) at not less than 70 psi. Shut off pump temporarily if F-2 Wt. Ftr. manometer exceeds 1.0.	Lbs. water added_____ Pump pressure_____
5.	With F-2 at 10 RPM and with F-2 to F-1 jet on, add 450 lbs. of water through high pressure bowl sprays. (Pressure must be at least 275 psi) Shut off pump temporarily if F-2 Wt. Ftr. manometer exceeds 1.0.	Lbs. water add_____ Pump pressure_____
6.	With F-2 at 10 RPM and with F-2 to F-1 jet on, add water to F-2 via F-2D and (low pressure) bowl sprays until F-1 Wt. Ftr. shows approx. 1600 lbs. in F-1. Shut off pump temporarily if F-2 Wt. Ftr. manometer exceeds 1.0.	Time end_____ F-1 Wt. Ftr _____Lbs._____
7.	Stop F-2	Time F-2 stopped_____
8.	When jet gasses, stop jet.	Time stop jet_____
9.	Stop F-1 cooling water.	F-1 cooling water off_____
10.	Beckman on F-2	Meter_____Factor_____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

III - FIRST CAKE REMOVAL

1.	Start F-1 agitator and cooling water.	F-1 agitator on _____ F-1 water on _____
2.	Plug F-2 to 10 RPM and start F-2 to F-1 jet. Pump 400 lbs. of water from F-2-D through F-2 bowl spray.	Time F-2 at 10 RPM _____ Time jet on _____ Lbs. water add _____
3.	With F-2 to F-1 jet off and F-2 at 10 RPM, plow cake from bowl wall for approx. 10 minutes.	Time jet off _____ Plows in _____
4.	Move plows to "Out" position.	Time out _____
5.	With F-2 at 10 RPM and 100 lbs. of water to F-2 via F-2-D and F-2 bowl spray.	Lbs. water add _____ Time water add _____
6.	Bring F-2 to 100 RPM and hold with "inch button" at 100 RPM.	Time F-2 at 100 RPM _____
7.	Set plows three-fourths of the full distance to bowl wall and hold for 30 minutes at 100 RPM.	Plows in _____ Time start _____ Time end _____
8.	Stop F-2, move plows out, and jet slurry from F-2 to F-1.	Time F-2 stop _____ Plows out _____ Time jet off _____
9.	With F-2 at 10 RPM add 50 lbs. of water to F-2 via F-2-D and F-2 bowl spray.	Lbs. water add _____ Time water add _____
10.	Bring F-2 to 100 RPM and hold with "inch button" at 100 RPM.	Time F-2 at 100 RPM _____
11.	Set plows three-fourths of the full distance to bowl wall and hold for 15 minutes at 100 RPM.	Plows in _____ Time start _____ Time end _____
12.	Stop F-2, move plows out, and jet slurry from F-2 to F-1.	Plows out _____ Time F-2 stop _____ Time jet off _____
13.	With F-2 at 10 RPM add 50 lbs. of water to F-2 via F-2-D and F-2 bowl spray.	Lbs. water add _____ Time water add _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

III - FIRST CAKE REMOVAL (Cont'd)

14.	Bring F-2 to 100 RPM and hold with "inch button" at 100 RPM.	Time F-2 at 100 RPM _____
15.	Set plows three-fourths of the full distance to bowl wall and hold for 15 minutes at 100 RPM.	Plows in _____ Time start _____ Time end _____
16.	Stop F-2, move plows out, and jet slurry from F-2 to F-1.	Plows out _____ Time F-2 stop _____ Time jet off _____
17.	With F-2 at 10 RPM add 50 lbs. of water to F-2 via F-2-D and F-2 bowl spray.	Lbs. water add _____ Time water add _____
18.	Bring F-2 to 100 RPM and hold with "inch button" at 100 RPM.	Time F-2 at 100 RPM _____
19.	Set plows three-fourths of the full distance to bowl wall and hold for 15 min. at 100 RPM.	Plows in _____ Time start _____ Time end _____
20.	Stop F-2, move plows out, and jet slurry from F-2 to F-1.	Plows out _____ Time F-2 stop _____ Time jet off _____
21.	With F-2 at 10 RPM and with F-2 to F-1 jet on, add water to F-2 via F-2-D and F-2 bowl spray until F-1 Wt. Ftr. shows approx. 1600 lbs. in F-1.	Time jet on _____ Time end _____ F-1 Wt. Ftr. _____ Lbs. _____
22.	Stop F-2.	F-2 stopped _____
23.	When jet gasses, stop jet.	Time jet off _____
24.	Beckman on F-2	F-2 Meter _____ Factor _____

[redacted]

Date_____
Run No. _____

DECLASSIFIED

IV - METATHESIS WASH

1.	Make up 250 lbs. of 17% Potassium Hydroxide in E-1B.	
	a. Add 200 lbs. of water	Lbs. water add____Time_____
	b. Start agitator	Time agitator on _____
	c. Add 50 lbs. of Potassium Hydroxide	Lbs. Potassium Hydroxide add_____ Time _____
	d. Continue agitation until solid is dissolved.	Lbs. Total_____Time_____ Time dissolved_____
	e. Take sample to lab for approval	Sampled by_____ Time OK'd by lab_____
2.	Add the 250 lbs. of 17% Potassium Hydroxide from E-1B to F-1 via F-1C	Lbs. add_____Time add_____
3.	Wash E-1B to F-1C to F-1 with 100 lbs. of water	Lbs. add_____ Time add_____
4.	Stop F-1 agitator and add sufficient water to make total weight in F-1 up to 2100 lbs. as determined by Wt. Ftr.	Time agitator off_____ Lbs. water add_____ F-1 Wt. Ftr. _____Lbs._____ F-1 Sp. Gr._____Temp._____
5.	Start F-1 agitator	F-1 agitator on _____
6.	Adjust temperature of F-1 to 35°C and agitate for one hour.	Time at 35°C _____ Time end_____
7.	Start F-1 cooling water	F-1 water on _____

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V - METATHESIS WASH CENTRIFUGING

1. Bring F-2 to 1800 RPM. — Time 1800 RPM_____

2. Beckman on F-2. — Meter_____Factor_____

3. Start F-9 sparger. — F-9 sparger on _____

4. Transfer slurry from F-1 to F-2 at the rate of 25 lbs. per min. Check the transfer rate at 30 minute intervals, recording data in table.

Time	Jet Press	F-9Wt.	Lbs./min.
		0	

5. Stop F-1 agitator when F-1 Wt. Ftr. pen reaches 0.8 and start F-1 air sparger. — F-1 agitator off _____
F-1 sparger on _____

6. When F-1 Wt. Ftr. pen reaches zero, stop F-1 air sparger. — F-1 sparger off _____

7. When the jet from F-1 to F-2 first gasses, raise jet steam pressure to full. Stop jet when ammeter and jet thermometer indicate F-1 is empty. — Time full pressure_____
P. S. I. _____
Time stop jet _____

8. After 5 min. at 1800 TPM, skim F-2 to 3 gal. Stop cooling water on F-1. — Time end skimming _____

9. Beckman on F-1 and F-2. — F-1 Meter_____Factor_____
F-2 Meter_____Factor_____

10. Slow F-2 to 900 RPM. — Time 900 RPM._____

11. Stop F-9 sparger and read Wt. Ftr. and Sp. Gr. when constant. — F-9 sparger off _____
F-9 Wt. Ftr._____Lbs._____
F-9 Sp. Gr._____Temp._____

12. Start F-9 sparger. — F-9 sparger on _____

13. Take sample F-9 WS — Time sampled_____

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VI - FINAL CAKE REMOVAL

1.	Plug F-2 to stop and add 24 lbs. 60% Nitric Acid to F-2 via F-2B. (Line A)	F-2 stopped _____ Time add _____ Lbs. add _____ F-2 Wt. Ftr. _____ Lbs. _____
2.	Bring F-2 to 160 RPM, hold 2 min. Plug to stop. Repeat continuously for thirty min.	Time start _____ Time end _____
3.	Rotate F-2 slowly (approx. 10 RPM) and cut cake off bowl wall with plow. Plow for 10 minutes)	Plows in _____ Time end _____
4.	Bring F-2 to 100 RPM and hold at 100 RPM for 30 min., with plows set approx. three-fourths in. Withdraw plow and plug F-2 to stop.	Plows in _____ Plows out _____ Time end _____ F-2 stopped _____ F-2 Wt. Ftr. _____ Lbs. _____
5.	Check F-10 system (drain valve closed) and carry out item 1, page 9.7	PR -Can in loading position _____ F-10 Wt. _____ F-10 valve closed _____
6.	Beckman on F-10.	F-10 Meter _____ Factor _____
7.	Obtain supervisor's approval that F-10 Beckman is satisfactory.	F-10 Beckman limit 20 x 10^{-14} _____ Approved by _____ Time _____ Supervisor
8.	Jet F-2 to F-10.	Time jetted _____
9.	Read weight in F-10.	F-10 Wt. _____
10.	Add to F-2 sufficient water via F-2B to bring F-10 weight to 72 lbs.	Lbs. water added _____
11.	Bring F-2 to 160 RPM, hold 2 min. Plug to stop. Repeat three times.	Time first slurrying _____ Time second " _____ Time third " _____ Time fourth " _____
12.	Jet F-2 to F-10.	Time jetted _____
13.	Read weight in F-10.	F-10 Weight _____
14.	Beckman on F-2.	F-2 Meter _____ Factor _____
15.	Obtain supervisor's approval that F-2 Beckman is satisfactory.	F-2 Beckman limit 20 x10-- _____ Approve by _____ Time _____ Supervisor
16.	Beckman on F-10.	F-10 Meter _____ Factor _____
17.	Obtain supervisor's approval that F-10 Beckman is satisfactory.	Calculated Beckman minimum x10 _____ Approved by _____ Time _____ Supervisor
18.	With F-2 at 10 RPM, add 100 lbs. of water via F-2D (low pressure sprays) Just after sprays start, begin jetting F-2 to F-1. Keep bowl empty during the entire water wash by intermittently stopping water to the sprays. Leave water in F-1 for the next charge.	Time start _____ Time end _____ F-1 Wt. Ftr. _____ Lbs. _____
19.	Plug F-2 to stop.	F-2 stopped _____
20.	Beckman on F-2.	Meter _____ Factor _____

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VII - SHIPMENT OF PRODUCT

1.	With supervisor's approval, swing container out of carrier and into F-10 enclosure with chain hoist. Raise container until F-10 drain spout is at maximum depth inside open charging port. Close doors of F-10 enclosure.	F-1-PS Assay_____T.C. Approved by_____Time_____ Supervisor
2.	Record wt. of F-10.	F-10 lbs._____
3.	Open F-10 valve gently and slowly empty F-10 into container (Approx. 10 lbs/min.).	Time start_____ Time end_____
4.	Record weight of F-10 and lbs. of solution delivered.	F-10 lbs._____ Lbs. delivered_____
5.	Close F-10 valve and let spout drain into container for 20 min.	Valve closed_____ Time drained_____
6.	Carefully lower container away from F-10 spout and bring to a position just inside enclosure. Replace drip pan under spout.	
7.	With supervisor's approval, swing cover plate over container charging hole, hold suspended, and rise top of can inside curbing with 100 cc of 60% nitric acid.	Time OK to proceed_____ Acid rinse volume_____ Time rinsed._____
8.	Rinse twice with 100 cc portions of water.	Time rinsed_____
9.	Make "Sandy" survey of container.	Time completed_____
10.	If "Sandy" survey is satisfactory, close and tighten container cover, remove from enclosure, and make portable Beckman survey of container.	Time removed_____ Beckman reading (side)_____ (bottom)_____
11.	Place container in carrier and remove to truck.	Time completed_____
12.	Beckman on F-10.	F-10 Meter_____Factor_____
13.	Obtain supervisor's approval that F-10 Beckman is satisfactory.	F-10 Beckman limit 20 X 10^{-14} Approved by_____Time_____ Supervisor

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VIII - DISPOSAL OF F-7 WS AND F-9 WS

1.	With the approval of the supervisor, jet F-7 to C-8. C-8 should be empty or contain neutralized wastes before jetting starts.)	Time OK to proceed _____ Time start _____
2.	Stop F-7 agitator when F-7 Wt. Ftr. reaches 0.2.	F-7 agitator off _____
3.	When F-7 to C-8 jet gasses, stop jet.	Time stop jet _____
4.	With supervisor's approval, start F-7 agitator and jet F-9 to F-7.	Time OK to proceed _____ F-7 agitator on _____ Time start jet _____
5.	When F-9 Wt. Ftr. pen reaches 0.2, stop F-9 sparger.	F-9 sparger off _____
6.	When F-9 to F-7 jet gasses stop jet.	Time stop jet _____
7.	With the approval of the supervisor, jet F-7 to C-8. (C-8 should be empty or contain neutralized wastes before jetting starts.)	Time OK to proceed _____ Time start _____
8.	When F-7 Wt. Ftr. pen reaches 0.2, stop F-7 agitator.	F-7 agitator off _____
9.	When F-7 to C-8 jet gasses, stop jet.	Time stop jet _____

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CHAPTER VII - CONCENTRATION

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SECTION C - SEPARATIONS

CHAPTER VIII - PRODUCT ISOLATION

In the extraction, decontamination, and concentration processes described in the foregoing chapters the product is separated from the uranium and the quantity of fission elements associated with the product is reduced by a factor about 10^7 through the use of carriers.

The final step in the separation process consists in the separation or isolation of the plutonium as its peroxide from the final carrier used during the concentration process, followed by purification, concentration, and shipment as plutonium nitrate. This chapter describes the equipment and the process used in this product isolation step.

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CHAPTER VIII - PRODUCT ISOLATION

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ISOLATION PROCESS BASIS

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Purpose of the Isolation Process

Throughout its processing in the Canyon and Concentration Buildings, plutonium is not identified in any way as a pure compound. In these operations plutonium is always associated with large amounts of carriers, so that it may be decontaminated with respect to fission products and be recovered from large volumes of solutions. Following final processing in the Concentration Building, the plutonium is delivered to the Isolation Building in a relatively small volume of solution per batch (about 8 gallons or 30 liters) which contains a relatively small amount of carrier (about 1 kg of La). The purpose of subsequent operations in the Isolation Building is the separation of plutonium from the carrier and the preparation of its nitrate salt having a minimum purity of 95% as the final product.

Reasons for the Selection of the Plutonium Peroxide Isolation Process

The separation of plutonium from lanthanum and other ions involves the direct precipitation of a plutonium compound for the first time in the process. To obtain the desired separation from carrier and impurities, the plutonium compound selected must satisfy certain conditions. This compound must have a low solubility in the precipitation medium and the mechanical separation from the supernatant solution must be simple. Purification from other cations (La, Fe, Zr, etc.) should be obtained. Ease of dissolution of the precipitated compound is also an important consideration.

Of the known compounds of plutonium, plutonium peroxide most closely approximates the limitations established for the compound to be chosen. Plutonium peroxide exhibits a low solubility in the reaction medium anticipated, it is easily redissolved with HNO_3 to give the nitrate, and it may be readily separated from its supernatant by decantation or filtration. Further precipitation of the peroxide yields high purification from other cations since those cations when present do not precipitate as peroxides, nor are they carried appreciably by plutonium peroxide. For these reasons plutonium peroxide has been selected for isolation purposes.

From a knowledge of the chemistry of plutonium peroxide and of the variables that influence its precipitation, a chemical process for the isolation of plutonium and suitable equipment in which to carry out this process have been developed. The nature of the physical equipment employed has been determined not only by the chemistry of the process, but by the physiological activity of trace amounts of plutonium which has necessitated remote control operations and elaborate ventilation systems for the purpose of safe operation.

Chemistry of Plutonium Peroxide

Plutonium forms a green, insoluble peroxide in dilute acid solutions in the presence of H_2O_2. (Ref. 1-8) This compound has a relatively low solubility

in the precipitation medium, in dilute acid solution and in water. In this respect plutonium is similar to uranium, thorium and zirconium.

Formula of Plutonium Peroxide

Valence of Plutonium (Ref. 9-12) Considerable effort has been devoted to the determination of the exact formula of plutonium peroxide. If the compound is analogous to uranyl peroxide UO_4, the valence of plutonium is 6 and the formula is of the type PuO_4. If the peroxide is analogous to thorium peroxide (Th_2O_7), the valence is 4 and the formula is of the type Pu_2O_7. There is strong evidence to indicate that the valence of plutonium in the peroxide is 4 (Pu(IV)). Addition of H_2O_2 to known solutions of Pu(IV) rapidly precipitates a green peroxide. The addition of H_2O_2 to known solutions of Pu(VI) and Pu(III) results in a slow precipitation of the same green compound. Hydrogen peroxide is capable of reducing Pu(VI) to Pu(IV) and oxidizing Pu(III) to Pu(IV) so that in the above cases it is the Pu(IV) formed that precipitates as the peroxide.

Other information concerning the valence of plutonium in the peroxide has been obtained in titration studies. When plutonium peroxide is dissolved in KI solution and the iodine liberated is titrated with thiosulfate, an end point is obtained at about three equivalents of iodine per atom of plutonium. At this point the solution has the typical amber color of a Pu(IV) solution. On further standing another equivalent of iodine is liberated and the solution has the blue color of Pu(III). The extra equivalent of iodine is liberated as a result of the reduction of Pu(IV) to Pu(III). Similar evidence has been obtained by titration with stannous salts.

Peroxidic Oxygen Content (Ref. 11, 13-16) The determination of the equivalents of peroxidic oxygen present per atom of plutonium has been studied by titration techniques employing Ce^{+4}, MnO_4^{-1}, SO_2, Sn^{+2} and I^{-1}. The most consistent results have been obtained with Sn^{+2} and I^{-1}. When either thorium or plutonium peroxides are precipitated from excess H_2O_2 and subjected to five or six water washes, on titration with I^{-1} or Sn^{+2} about 3.2-3.3 peroxide equivalents/gram atom of Th or Pu are found. This odd number does not fit into the formula of Th_2O_7 for thorium or any simple formula for plutonium. It is believed that excess peroxide has been precipitated either as H_2O_2 of crystallization or by occlusion and has not been removed by the washings. However, when either Th or Pu peroxide is precipitated with an excess of Th or Pu present, the number of peroxide equivalents/gram atom of Th or Pu is about 2.95. Under these conditions the formula Th_2O_7 is satisfied and the formula for plutonium peroxide is presumably Pu_2O_7.

Influence of Sulfate Ion (Ref. 10, 14, 17, 18) When plutonium peroxide is precipitated in the presence of sulfate ion, the precipitate obtained is darker green, of larger particle size and of lower solubility than that precipitated in the absence of sulfate. Both types of precipitate contain the same number of equivalents of peroxidic oxygen/gram atom of Pu. Some sulfate is precipitated with the peroxide and cannot be removed by extensive washing. Analyses have indicated mol ratios of Pu/SO_4 of 2.6-3.0.

Thorium peroxide precipitated in the presence of sulfate is known to have the formula, $Th_2O_6 \cdot SO_4$. Plutonium peroxide precipitated in the presence of

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sulfate may then have the formula, $Pu_2O_6 \cdot SO_4$, although the sulfate found in such precipitates is less than that required by the formula.

Most Probable Formula. Interpretation of the above data would seem to indicate that the most probable formula for plutonium peroxide is $Pu_2O_6 \cdot X \cdot nH_2O$ where X may be SO_4^{-2}, $(NO_3^{-1})_2$, or O^{-2}. The simplest formula for the peroxide may be Pu_2O_7.

Factors Influencing the Precipitation of Plutonium Peroxide

A large number of factors influence the precipitation of plutonium peroxide and result in changes in the physical characteristics of the precipitate as well as its solubility. These factors include acidity, temperature, H_2O_2 concentration, time of digestion, and the concentration of various ions. Certain conditions are optimum for the production of one set of desired characteristics, while other conditions are optimum for other characteristics. It is difficult to separate the consideration of these factors since in most studies several of these factors have been varied simultaneously. In the following the relationship between these factors is discussed.

Acidity. (Ref. 19-21) Plutonium peroxide may be precipitated over a wide range of acidity, from a few thousandths normal to several normal hydrogen ion. Little is known regarding the peroxide compound at very low acidities owing to the hydrolytic behavior of plutonium at these acidities. Plutonium peroxide may be precipitated from several different acids, HNO_3, HCl, H_2SO_4, $HClO_4$ and $HC_2H_3O_2$ having been studied. However, most studies have dealt with HNO_3, particularly 1-2N HNO_3. Above 3N HNO_3 the solubility is markedly increased. Precipitation from 2N HNO_3 in the presence of 0.25M $(NH_4)_2SO_4$ gives rise to larger sized particles with better settling characteristics than are obtained at lower acidities. The solubility of the peroxide under these conditions is only slightly greater than in 1N HNO_3-0.2N H_2SO_4 solutions.

Sulfate Ion. (Ref. 17,20,22-25) Early studies of the influence of sulfate ion on plutonium peroxide formation indicated that sulfate has a very marked effect on the physical properties and solubility of the peroxide. That sulfate has been incorporated into the molecule has already been mentioned. In the presence of sulfate the solubility of the plutonium peroxide is initially lower and decreases more rapidly on standing, although the physical characteristics are such that the precipitate forms, agglomerates and settles more rapidly and is more filterable. The optimum sulfate concentration is in the range 0.1-0.25M.

H_2O_2 Concentration. (Ref. 13,26,27) Plutonium peroxide may be precipitated from solutions containing a few tenths percent up to 10 or 20% by weight of H_2O_2. As the H_2O_2 concentration is increased, lower solubilities are obtained. The decrease in solubility on standing is more rapid at higher H_2O_2 concentrations. In the presence of 1.0 N HNO_3-0.2N H_2SO_4, the solubility obtained with 4% H_2O_2 is about the same as that found with 10% H_2O_2. In 2N HNO_3 - 0.25M $(NH_4)_2SO_4$ solutions the solubility with 10% H_2O_2 is considerably lower than with 4% H_2O_2.

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Plutonium Concentration. (Ref. 26) The concentration of plutonium also influences the solubility of the plutonium peroxide precipitated. In solutions 1.0N in HNO_3 and 0.2N in H_2SO_4 there is practically no difference in solubility whether the original concentration was 1.0 or 3.3 gm Pu/liter (equivalent to 30 and 250 gm/ton of uranium, respectively). However, when 2N HNO_3 - 0.25M $(NH_4)_2SO_4$ solutions are employed the solubility obtained when the original plutonium concentration was 1.0 gm/liter is three times as great as that when 8.3 gm Pu/liter was originally present.

Temperature. (Ref. 12,13,20,25,27) Variations in temperature markedly affect the precipitation of the peroxide. Precipitations at temperatures above 20 °C are advantageous with respect to particle size and hence to filterability and settling. At 60 °C in the presence of sulfate easily filterable precipitates may be obtained with no appreciable increase in solubility over that found at 10-25 °C. At 80 °C, higher solubilities are obtained. Temperatures above 20-25 °C are not practical in the presence of ferric iron owing to the marked catalytic decomposition of H_2O_2 by Fe^{+3} at high temperatures. Hydrogen peroxide is quite stable, however, in the presence of a stainless steel surface (25-12 Cb stabilized).

Digestion. (Ref. 12,19,20,22,23) The precipitation of plutonium peroxide is not complete on the first addition of H_2O_2 to plutonium solutions. The solubility of the peroxide continues to decrease for several hours at 20° C and may continue to decrease for as long as 24 hours, but the rate of decrease is usually low after the first 2 or 3 hours. In the presence of sulfate the digestion period for complete precipitation is shortened. Similarly, in 2N HNO_3 the required digestion period is shorter than in 1.0N HNO_3. In the absence of Fe the digestion time is reduced at temperatures higher than 20 °C. Long standing (24 hours) in the presence of Fe^{+3} may result in increasing solubilities as a result of H_2O_2 decomposition. High concentrations (about 0.5 M) of various salts such as $La(NO_3)_3$, $Al(NO_3)_3$ and NH_4NO_3 tend to prolong the digestion period required to achieve low solubility of product in the supernatant solution.

Ionic Interference. (Ref. 11,12,18,19,24,25,27-31) The presence of other ions in solution when plutonium peroxide is precipitated may affect the solubility of the peroxide. From studies on purified plutonium preparations, the solubility of the peroxide was found to be about 20 mg Pu/liter in 10% H_2O_2 - 1.0N HNO_3 - 0.2N H_2SO_4, or 10% H_2O_2 - 2N HNO_3 - 0.25M $(NH_4)_2SO_4$ solutions. Lower solubilities have occasionally been obtained.

The solubility of the plutonium peroxide at 60 °C increases rapidly with increasing concentration of Fe^{+3}. The increase is 2-fold at 0.001M Fe, 4-fold at 0.002M Fe and over 100-fold at 0.005M Fe when precipitated from 10% H_2O_2 - 1.0N HNO_3 - 0.2N H_2SO_4 solutions and digested 2 hours. At 10 - 20 °C the increase in solubility with 0.01M Fe may be only 2 or 3-fold. In 10% H_2O_2 - 2N HNO_3 - 0.25M $(NH_4)_2SO_4$ at 20 °C the presence of 0.01M Fe approximately doubles the solubility found in 10% H_2O_2 - 1.0N HNO_3 - 0.2N H_2SO_4 solutions. The action of iron involves the catalytic decomposition of H_2O_2. Plutonium peroxide in the presence of Fe^{+3} does not seem to dissolve until the H_2O_2 concentration is appreciably reduced.

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Zirconium ion also increases the solubility of plutonium peroxide. Zirconium acts, presumably, by forming a complex with plutonium and H_2O_2, possibly a mixed peroxide. If zirconium is present during the precipitation of plutonium peroxide from 10% H_2O_2 - 1.0N HNO_3 - 0.2N H_2SO_4 solutions, the solubility of the peroxide is increased 2-fold at 0.001M Zr, 4-fold at 0.01M Zr, and about 100-fold at 0.1M Zr. The effect of Zr is somewhat greater at 10 °C than at 60 °C and in 2N HNO_3 than in 1.0N HNO_3. This effect of Zr can be duplicated by the addition of Zr to previously formed plutonium peroxide followed by a period of digestion.

When iron and zirconium are both present, the solubility of the peroxide seems to be controlled by the ion present in highest concentration. At low Fe and Zr concentrations the effect is probably additive.

Iron and zirconium are the only known cations that markedly affect the solubility of plutonium peroxide. Their action is exerted at relatively low concentrations. Other ions, such as La^{+3}, Bi^{+3}, Mn^{+2}, Cr^{+3}, may be present to the extent of several tenths molar concentration with only a very slight, if any, increase in solubility. Lower solubilities are actually found in the presence of Bi^{+3}, possibly due to coprecipitation of some bismuth.

Stability

Plutonium peroxide is stable (Ref. 27, 32) for several hours in the absence of excess H_2O_2 in dilute acids (1.0N). After 24 hours, however, the peroxide shows evidence of dissolution probably as a result of decomposition of the peroxide linkage in the molecule. In water, the peroxide is stable for 48 hours or longer in the absence of H_2O_2. In more concentrated acids, the peroxide slowly dissolves at room temperature.

Dissolution of Plutonium Peroxide

Plutonium peroxide dissolves in the presence of concentrated acids with the liberation of oxygen (Ref. 10, 11, 15). Heating is usually employed to hasten the dissolution. The mechanism of dissolution probably involves the formation of H_2O_2 with its subsequent decomposition to water and oxygen.

Dissolution may also be accomplished by reagents that react with the peroxidic oxygen in the compound. These reagents include SO_2, NO_2, I^{-1} and Sn^{+2}, all of which react by reducing the peroxidic oxygen; and MnO_4^{-1}, $Cr_2O_7^{-2}$ and Ce^{+4}, which react by oxidizing the peroxidic oxygen. Simultaneous reduction to Pu(III) or oxidation to Pu(VI) may occur, but this is not a prerequisite to the dissolving action of these substances.

Properties of Plutonium Nitrate Solutions

Densities and freezing and melting points of concentrated solutions of plutonium nitrate have been determined (Ref. 20, 32) at several concentrations. Table I records the density versus product concentration of several $Pu(NO_3)_4$ solutions prepared by the evaporation of dilute HNO_3 solutions of plutonium.

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TABLE I

Density of Plutonium Nitrate Solutions at 25 °C

	$Pu(NO_3)_4$ Concentration						
%	9.1	14.9	24.2	28.0	39.8	46.4	58.0
Pu, gm/l	57	108	191	226	347	427	578
Density	1.30	1.48	1.62	1.66	1.79	1.87	2.03

The solutions are quite viscous at room temperature when the $Pu(NO_3)_4$ concentration was greater than 40%.

The freezing and melting points of concentrated $Pu(NO_3)_4$ solutions also prepared by evaporation are recorded in Table II. The solutions were slowly cooled by immersion in a solid CO_2 - acetone bath. The large divergence between freezing and melting points indicates that considerable supercooling occurred in these experiments. Furthermore, insufficient time may have been allowed for establishing equilibrium conditions.

TABLE II

Freezing and Melting Points of Plutonium Nitrate Solutions

$Pu(NO_3)_4$ %	Freezing Point °C	First Evidence of Melting °C	Complete Melting °C	Remarks
24	-62	-28	-18	Sharp, definite freezing point
30	-63	-44	-19	Gradual increase in viscosity on cooling to a syrupy consistency until crystallization began
42	-70	-53	-26	Same as 30% $Pu(NO_3)_4$
49	-79	-57	-38	Freezing occurred only after several hours at -79° at which time a hard glass was produced

Only limited solubility data on plutonium nitrate solutions are available. The solubility of plutonium nitrate in high concentrations of HNO_3 is at least 2.5M (about 60% $Pu(NO_3)_4$). The HNO_3 concentration in the above case is not known, but it is probably less than 10M, since a 30% $Pu(NO_3)_4$ solution (similarly prepared by evaporation) was found to be 11M in HNO_3.

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PLUTONIUM PEROXIDE ISOLATION PROCESS

The process employed in the Isolation Building was developed by selecting those conditions which gave the most favorable results with the process solution received from the Concentration Building. This solution, known as the F-10-P solution, is obtained from the dissolution of the metathesized LaF_3. A typical F-10-P solution has a nominal volume of 8 gallons (30.3 liters) and contains the following approximate concentrations of substances, 0.4M La, 0.004-0.01M Fe, 1.8-2.2M HNO_3, 0.1-0.2M K, 0.03M Pu, and variable but low concentrations of Zr, Ca, Mn, together with some suspended MnO_2.

Two peroxide precipitations are required to obtain a product purity of 95% or better; therefore, the isolation process consists of a first and second cycle. Figures 1 and 2 are schematic diagrams illustrating the movement of product and or recycle materials in the process cells. A schematic diagram of the process cell equipment is shown in Figure 3.

First Cycle

First Filtration

The initial treatment of the F-10-P solution consists of filtration through a Type E Filtros plate in Tank N-1 upon which a pre-coat of H_2O-elutriated filter aid (Hyflo-Super-Cel) has been laid. With each new F-10-P solution a small additional quantity (1 g./l) of filter aid is added. The filtered solution is transferred to the first precipitating vessel, P-1, and a filter wash of one 500 ml. portion of 1.0N HNO_3 is added to the filtrate. The separation of various insoluble materials, including MnO_2 is the purpose of the filtration.

Prereduction

A variable portion, as high as 60%, of the plutonium present in the F-10-P solution is in the oxidized form, Pu(VI). Since Pu(VI) does not precipitate with H_2O_2, and since Pu(VI) is only slowly reduced to Pu(IV) by H_2O_2 under the precipitation conditions, an apparent high solubility may result from the presence of Pu(VI). Reduction of the Pu(VI) to Pu(IV) or Pu(III) is required before hydrogen peroxide addition. Rapid reoxidation of Pu(III) to Pu(IV) occurs in the presence of H_2O_2 (particularly when sulfate ion is also present), hence any Pu(III) formed during reduction of Pu(VI) is not harmful to the process. Prereduction of the solution in P-1 is carried out using 0.05M $(NH_4)_2SO_3$ in the presence of 0.020M $(NH_4)_2SO_4$. The reduction is complete within a few minutes at room temperature; however, in practice a 15-minute period is used. Prereduction may be obtained with 0.1M hydroxylamine sulfate, but this requires at least 30 minutes at room temperature, as well as a higher concentration of reagent. Hydrogen peroxide also may be employed for the prereduction. Substantially complete reduction is obtained if the solution contains 1% H_2O_2 by weight and is heated to 50 °C for 1 hour. The use of hydrogen peroxide for reduction of the plutonium has several disadvantages. In addition to heating and the longer period of time required, gas

FIGURE 1

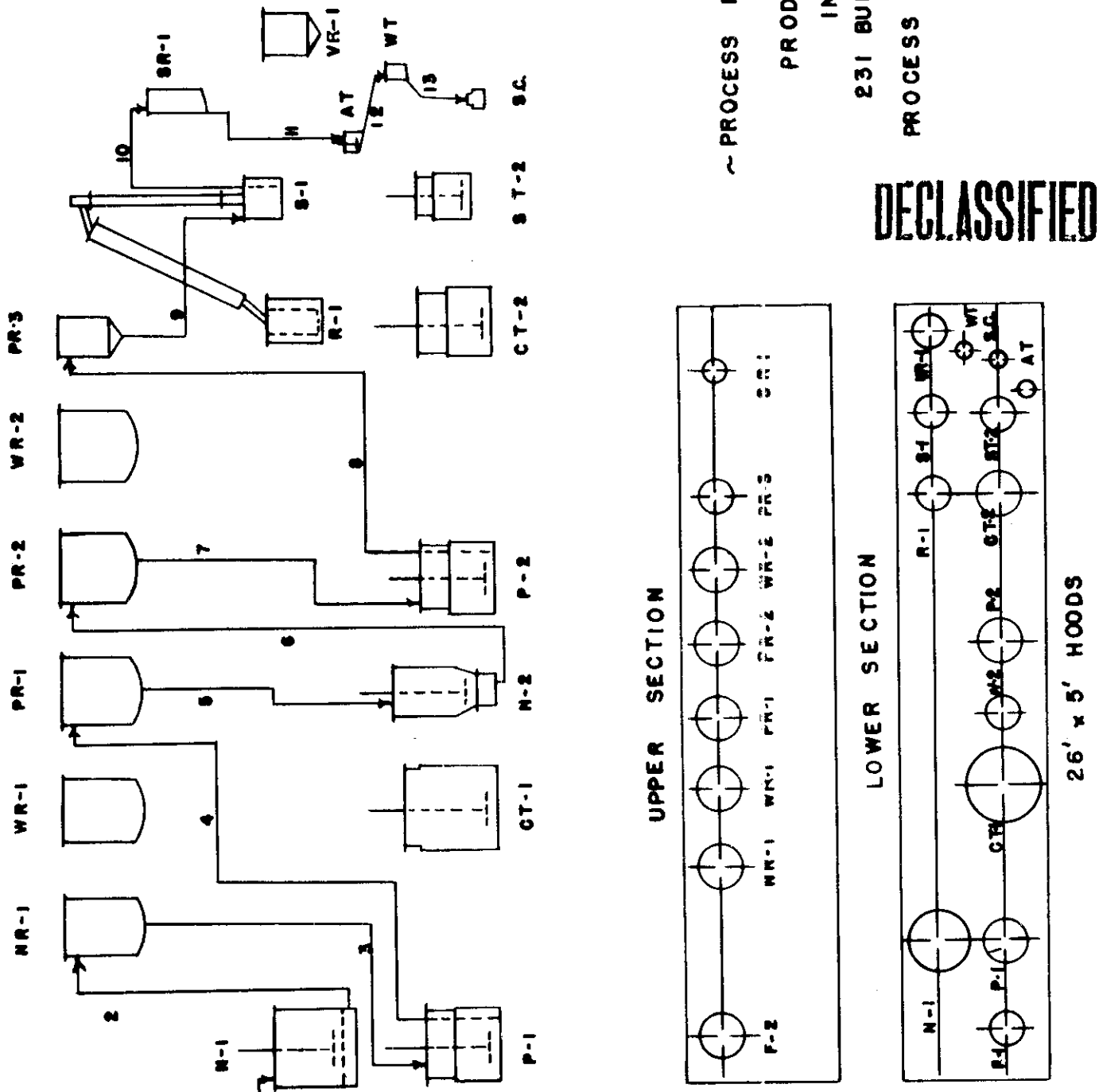
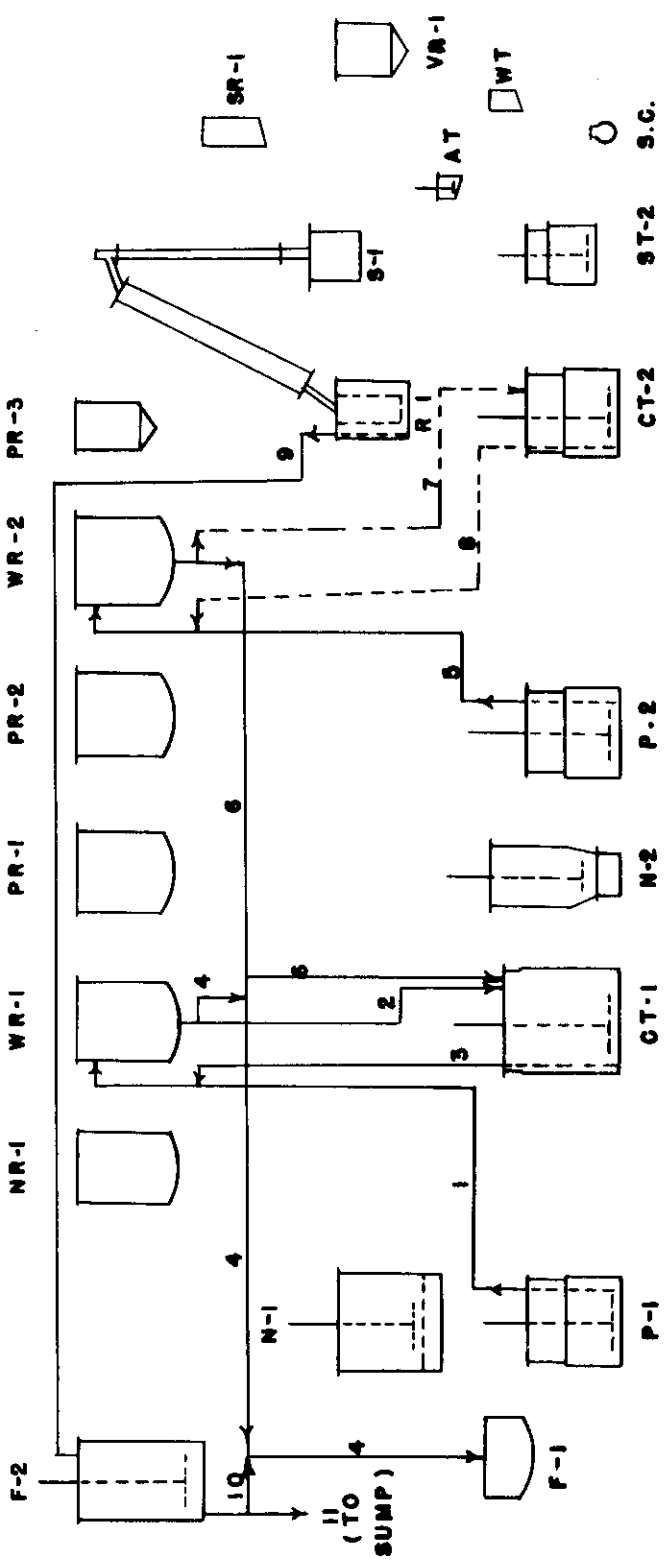

~ PROCESS FLOWSHEET
PRODUCT
IN
231 BUILDING
PROCESS CELLS ~
DECLASSIFIED
SR-1
VR-1
WT
AT
S.C.
S-1
S T-2
R-1
CT-2
PR-3
WR-2
PR-2
P-2
PR-1
N-2
WR-1
GT-1
NR-1
N-1
P-1
F-2
F-1
UPPER SECTION
LOWER SECTION
26' x 5' HOODS

FIGURE 2



PROCESS FLOWSHEET

RECYCLE IN 231 BUILDING PROCESS CELLS

--- LINES FOR THIRD CYCLE OR ALTERNATE USE

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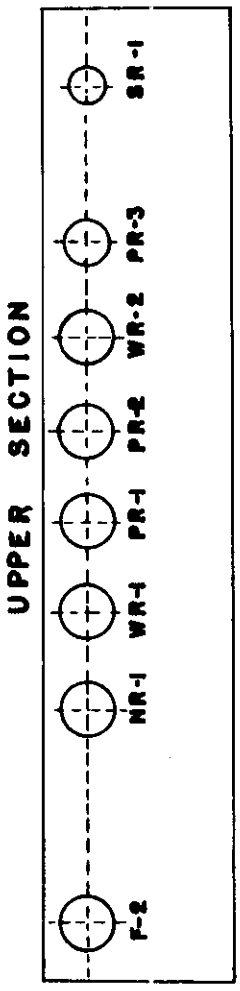
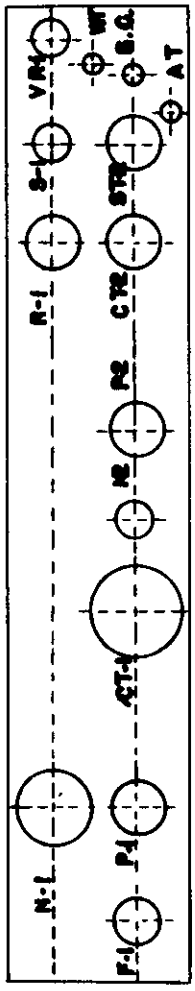


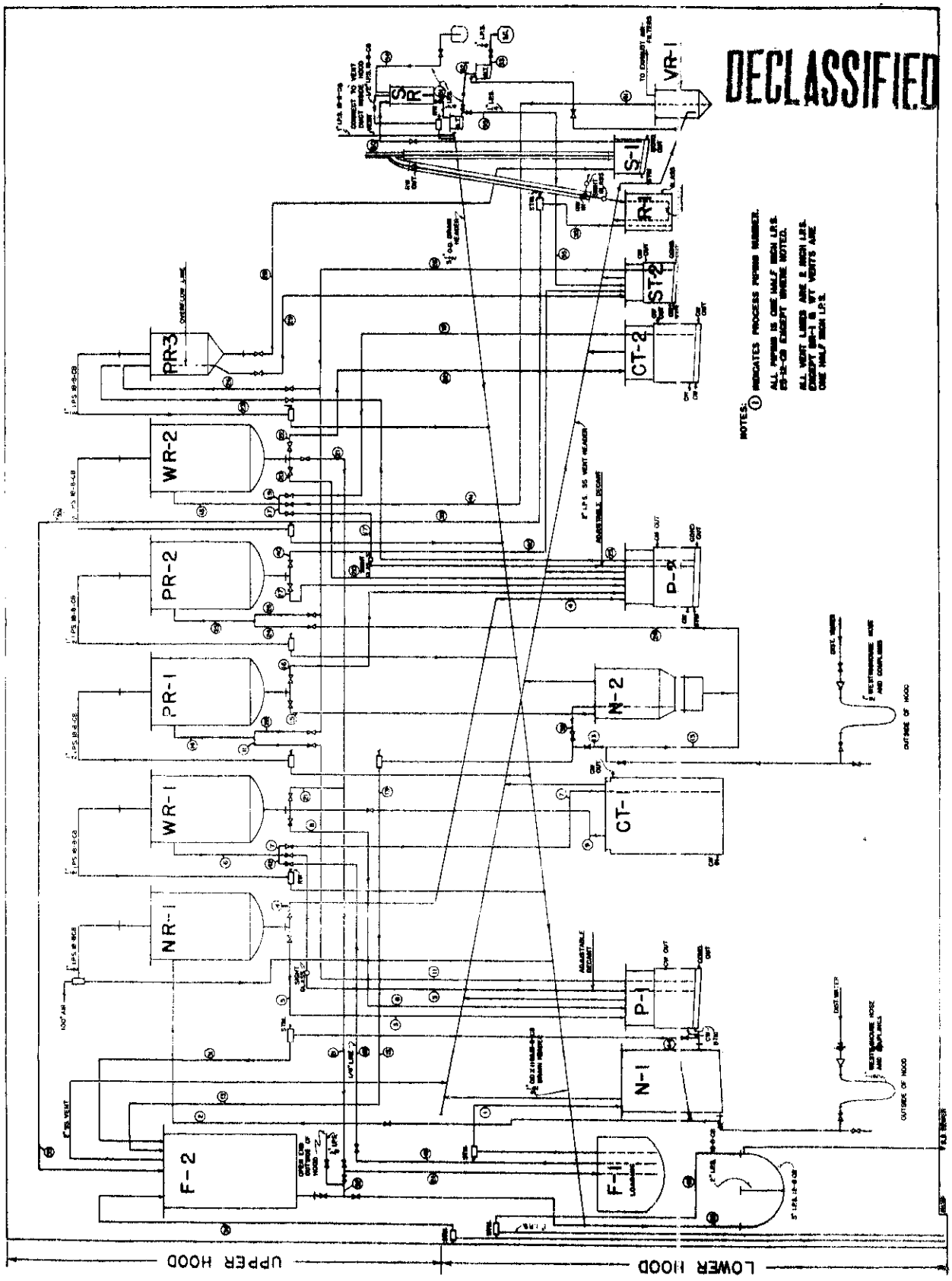


26' x 5' HOODS

DECLASSIFIED

FIGURE 3

PROCESS CELLS IN 231 BUILDING



evolution occurs due to the decomposition of H_2O_2, thus presenting a health hazard. Furthermore, reoxidation of Pu(IV) to Pu(VI) may occur during the heating, since the H_2O_2 is rapidly destroyed, particularly if the concentration of Fe is high.

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First Precipitation and Wash

The precipitation conditions chosen were those which produce a plutonium peroxide precipitate of low solubility and fast settling characteristics suitable for decantation, while minimizing the interference of ferric ion. The conditions ultimately selected involve precipitation from 2N HNO_3 - 0.25M $(NH_4)_2SO_4$ solution. The solution is made 0.20M in $(NH_4)_2SO_4$ prior to prereduction. Following prereduction the P-1 solution is adjusted to 2N HNO_3. A sufficient quantity of 30% H_2O_2 is added to P-1 over a 20-30 minute period to make the solution 10% H_2O_2 by weight. During H_2O_2 addition the temperature is maintained at 20 °C. Digestion of the plutonium peroxide precipitate formed is continued at 20 °C for an additional hour. A two hour settling period at 5-7 °C is allowed before decantation. The supernatant solution is decanted leaving a heel of approximately 5 liters. Three precipitate washes are made, each one consisting of 5 liters of 0.4N H_2SO_4. The precipitate is agitated with the wash solution for 5 minutes and allowed to settle for 30 minutes at 5-7 °C. The wash solutions are also decanted and combined with the original supernatant, which is held in a catch tank, CT-1, for subsequent treatment prior to recycling.

Dissolution of First Precipitate

For dissolving 1.7 liters of 60% HNO_3 are added to the peroxide precipitate in P-1 and the mixture is heated to 60-65 °C until solution is complete. A minimum period of 1 hour is allowed.

Second Filtration

The HNO_3 solution of plutonium is passed through a second filter, in N-2, to remove small quantities of insoluble materials. An E-grade Filtros filter block is used and this filter is also pre-coated with filter aid. The filtered solution is then transferred to the second precipitator, P-2.

Second Cycle

Second Precipitation and Wash

Following the first precipitation of plutonium peroxide, a product purity of approximately 80% is found. A second peroxide precipitation is carried out in order to obtain higher purity. The conditions for the reprecipitation are somewhat different from those used for the initial precipitation. The solution is diluted with water to give 15 liters of 1.5N HNO_3. Concentrated H_2SO_4 is added to make its concentration 0.5N. The H_2O_2 concentration is then made 10% by weight. Ammonium sulfate is avoided in the second cycle since an insoluble ammonium salt of plutonium (probably $(NH_4)_2Pu(NO_3)_6$) has been observed to

precipitate in the adjusting (AT) tank after concentration in the still (S-1) A heel of approximately 5 liters is again left after decantation of the supernatant solution. Three washes with 5-liter portions of 1.0N HNO_3 are made. The washes are decanted, combined with the supernatant and transferred to the catch tank (CT-1) where they are combined with the first cycle supernatant.

Dissolution of Second Precipitate

The second precipitate is dissolved in the same manner as the first precipitate but 2 liters of 60% HNO_3 are used instead of 1.7 liters. This HNO_3 solution is transferred to the receiver (PR-3). The purpose of the receiving vessel (PR-3) is to insure the delivery of a constant volume of solution to the still (S-1) for concentration. Accordingly, PR-3 is filled by the addition of 1N HNO_3 until overflow into ST-2, a small catch tank, is noticed. The whole volume of PR-3, about 9 liters, is delivered to S-1.

Concentration

Water and HNO_3 are distilled from the solution in S-1 until about 8.5 liters of distillate have been collected in R-1, the distillate receiver. approximately 1000 ml. or about 1700 grams of plutonium solution remains as a heel in the still (S-1). The remaining S-1 solution is transferred to the still product receiver, SR-1, from which it is dropped into the adjustment tank, AT, where the volume is measured and the solution sampled for concentration, density and purity determinations.

Transfer to Sample Can

The product solution in the adjustment tank (AT) is delivered in approximately 160-gram portions of plutonium to the weigh tank (WT) at which time the weight of solution delivered to the sample can (SC) may be measured. The solution delivered to the sample can is also checked by weighing it before and after filling.

Final Evaporation

The contents of the sample can, which amount to approximately 1100 grams of 30-40% $Pu(NO_3)_4$ solution, are further evaporated by heating the sample can in a glycerine bath maintained at 125 °C. Air is passed over the solution at a rate of 8 cu.ft./hr. The air passes through a water-cooled condenser to collect the distillate. The evaporation is continued until the weight of distillate collected indicates that about 400 grams of material remain in the sample can. On cooling to room temperature the material in the sample can appears to be a glassy, plastic mass which exhibits no apparent movement when the sample can is tilted. In this state the plutonium is considered safe for shipment.

Recycle of H_2O_2 Supernatants

Appreciable quantities of plutonium (amounting to several grams) are

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present in the combined H_2O_2 supernatants in the catch tank (CT-1). For recovery of this plutonium it is necessary to return the H_2O_2 supernatants to the Concentration Building. The transportation of a solution which is effervescing due to decomposition of H_2O_2 would present a possible health hazard. A further need for decomposing the H_2O_2 lies in the requirement that the H_2O_2 recycle to the Concentration Building process is added at a point just prior to the LaF_3 by-product (oxidized) precipitation. Hence a very large quantity of reducing agent, about 200 equivalents of H_2O_2, must be destroyed. For these reasons it is considered desirable to decompose the H_2O_2 in the Isolation (231) Building before return of the solutions to the Concentration (224) Buildings. The lanthanum contained in the recycled supernatant is then used in the precipitation of the LaF_3 by-product in the concentration process.

The reagent selected to decompose this large quantity of H_2O_2 must satisfy certain requirements. First, from the standpoint of safety with respect to gas evolution, the reduction of H_2O_2 to water is preferable to the oxidation of H_2O_2 to oxygen. Second, the reagent employed should introduce no new ions which might interfere with subsequent processing. Third, the volume of the recycle solution should be kept to a minimum to facilitate transporting it. Very few reagents are capable of satisfying all three of the above requirements. The first requirement appears to have eliminated consideration of agents such as $KMnO_4$, $K_2Cr_2O_7$, etc. Further, most reagents contribute large amounts of new cations to the solution. Of the reagents capable of reducing H_2O_2 to water only nitrite and sulfite ions have been given serious consideration. Neither nitrite nor sulfite contribute undesirable end products since they are oxidized to nitrate and sulfate, respectively. Of these two choices nitrite has been selected since its reaction with H_2O_2 may be carried out with the smallest increase in recycle volume, approximately a doubling in volume. Sulfite, as NH_4HSO_3 or $(NH_4)_2SO_3$ may be used, but, unless the volume increase is at least 3-fold, $La_2(SO_4)_3$ or double salts of $(NH_4)_2SO_4$ and $La_2(SO_4)_3$ precipitate. Since a saturated solution of H_2SO_3 is only 1M, the volume increase using this reagent is 3-fold or more. However, sulfite has the advantage that negligible gas evolution occurs during its use, whereas considerable gas evolution persists throughout the whole procedure when nitrite is used. Furthermore, some decomposition of nitrite to NO_2 occurs as a result of the decomposition of nitrite ion in strong acid.

In actual practice a 40% solution of $NaNO_2$ is used. Since KNO_3 is much less soluble than $NaNO_3$, KNO_2 is not used. Over 46 kilocalories of heat are liberated per mol of H_2O_2 decomposed by $NaNO_2$. Because of the large evolution of heat, a slow rate of addition of $NaNO_2$ to the H_2O_2 solution is used and cooling water is circulated in the jacket of the Tank CT-1 in which the reaction takes place.

The stoichiometry of the process is not that expected from the reaction:

$$NaNO_2 + H_2O_2 \longrightarrow NaNO_3 + H_2O$$

Only 80-85% of the theoretical quantity of $NaNO_2$ is actually required to complete the decomposition of H_2O_2. It appears that simultaneous decomposition

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of H_2O_2 to water and oxygen occurs during the reaction, particularly in the local area of heating where the $NaNO_2$ drops into the H_2O_2 solution. This divergence from exact stoichiometry has made the detection of the end point of the reaction difficult. The addition of excess $NaNO_2$ is considered as undesirable as the addition of too little $NaNO_2$. In order to destroy excess $NaNO_2$ or excess H_2O_2 after decomposition of the peroxide, a 4% solution of $KMnO_4$ is added until the solution shows a permanent coloration due to MnO_4^{-1}. However, if large quantities of $KMnO_4$ are required as a result of failure to locate the end point closely, the Mn(II) formed from the reaction of $KMnO_4$ with H_2O_2 or $NaNO_2$ reacts with additional $KMnO_4$ to form MnO_2. Thus, if the end point is not determined accurately, excessively large quantities of $KMnO_4$ will be used to react with excess reagent ($NaNO_2$ or H_2O_2). The presence of large quantities of MnO_2 is also considered undesirable since it may clog the jets in transferring the recycle solution in the Concentration Building.

The most satisfactory method that has been developed for the determination of the nitrite - peroxide end point involves an electrometric titration utilizing a bimetallic electrode system. A pair of electrodes, one of platinum, the other 90% platinum - 10% rhodium, dip into the H_2O_2 solution. The small difference of potential between these electrodes is recorded continuously by passing this voltage (generally -10 to -20 millivolts with respect to the platinum electrode) through a resistance of several hundred thousand ohms and thence into a continuously recording, photoelectric-type microammeter (General Electric, Model 8CE4CL21). During the addition of the bulk of the $NaNO_2$ very little change in potential is indicated. As the end point is approached the potential becomes more positive and at the end point a sudden increase of potential of +10 to +20 millivolts occurs. With this device the end point can be located to within less than 0.5% of the total $NaNO_2$ requirement (and even as low as 0.1%) and the amount of $KMnO_4$ added is consequently quite small.

In actual operation a minimum quantity of 40% $NaNO_2$ is first added. This quantity is based on the assumption that the solution is 2.25M in H_2O_2 and plant experience has shown that 80% of the stoichiometric quantity of $NaNO_2$ is required. The minimum quantity of $NaNO_2$ is computed in kilograms by multiplying the weight (kg.) of solution in the Catch Tank CT-1 by the factor, 0.17. The minimum nitrite is added at such a rate that the temperature is maintained at 22 - 3 °C by the circulation of cooling water through the jacket of the catch tank. After the minimum quantity of $NaNO_2$ has been added, the electrodes are pre-polarized by discharging 1.5 volts D.C., for 2 minutes through the electrodes in the H_2O_2 solution, with the platinum electrode being made positive. This pre-treatment of the electrodes is necessary in order that a good end point deflection be obtained. The electrode potential is recorded continuously and additional $NaNO_2$ is added until a scale deflection indicating the end point is observed. Potassium permanganate solution (4%) is then added until a permanent coloration appears. The recycle solution is transferred to the recycle cans (F-1) and transported to the Concentration Building. The average weight of recycle solution per plant run amounts to approximately 130 kg. (114 liters). This solution is divided equally among three recycle (F-1) cans.

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Product Purity

The Hanford Plant is committed to deliver plutonium nitrate with a minimum purity of 95% based on the plutonium content. The assay method employs solvent extraction of the plutonium from a sample of the solution in the adjustment tank (AT). The plutonium is extracted with hexone (isobutyl methyl ketone) in the presence of 10 N HNO_3. The aqueous phase is evaporated, then ignited, and the impurities weighed.

The product purity obtained at the end of the first cycle is about 80%. At the end of the second cycle a purity of 95% or greater is obtained.

Disposal of Active Wastes

The waste solutions from the process cells and laboratories containing possible traces of product are collected in a settling tank (illustrated in Figure 85, Chapter II) and after sampling and neutralization with sodium hydroxide are jetted into a reverse flow well or buried sump.

PROCESS CELL EQUIPMENT

The process equipment requirements of the Isolation (231) Building are relatively more simple than those of the Canyon and Concentration Buildings. Since the radiation level is very low (normally 20-25 mr/hr at the outside surface of the process hood), direct visual control of the process is employed. Because of the extremely low physiological tolerance for the product, the primary hazard in the Isolation Building is the spreading of product.

To avoid the spreading of product all operations are carried out in closed vessels installed within a well ventilated hood. A separate room, known as a process cell, is provided for each hood. Five such cells (Cells Number 1 to 5, inclusive) are provided in the building. An additional smaller cell (6-A) is used for the final evaporation of the plutonium nitrate solution to its safe shipping condition.

The process hoods, each 26 feet long, 5 feet wide and 8 feet high, are fabricated from 18-8 stainless steel and glass. All structural members are made of stainless steel. To these members removable wire-glass panels are attached. The equipment within the hoods is suspended from the stainless steel hood frame, with a seamless stainless steel pan provided with a sump constituting the bottom of the frame. This arrangement is convenient both for the inspection and washing of all surfaces. (For a view of this equipment see Figure 81, Chapter II) For details regarding the 26-foot processing hoods see the following blueprints:

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Subject	Number
Cell 1 Arrangement	H-2-173; H-2-174
Cell 1 - Process Piping	H-2-169; H-2-170
Cell 1 - Service Piping	H-2-171; H-2-172
Schematic Arrangement	H-2-165
Cells 2, 3, 4 - Arrangement	H-2-218; H-2-219
Cells 2, 3, 4 - Process Piping	H-2-220; H-2-221
Cells 2, 3, 4 - Service Piping	H-2-222; H-2-223
Cells 2, 3, 4 - Schematic Arrangement	H-2-224
Cell 1 - Head End and Scale Tank	H-2-350
Cells 2, 3, 4 - Head End and Scale Tank	H-2-225

Process Vessels

The process vessels are fabricated from 25-12 Cb-stabilized stainless steel. All pipe lines carrying the product are 1/2-inch standard pipe size and are also made of 25-12 stainless steel. Stainless steel (18-8) is used for the service piping. The use of 25-12 stainless steel is directed toward minimizing the contamination of the plutonium solutions due to corrosion products. The design of the individual vessels has been based on providing the greatest possible simplicity constistent with operational procedures and avoiding the spread of product. Whenever possible, the vessels have top inlet and discharge connections to avoid leakage. The fixed connections to the vessels are made by welded, screwed, or gasketed flange fittings. Movable connections are made through water seals.

Nearly all the vessels are provided with neutron acceptors because of limiting mass considerations. These acceptors consist of pipes and hollow agitator shafts and discs loaded with 10% cadmium - 90% lead shot. The process vessels are also equipped with vents so that all vapors and sprays may first be collected as much as possible in a trap (VR-1) before being exhausted through the filter box in the ventilating system.

The following enumerates and briefly describes the various vessels. The arrangement of these vessels is shown in Figure 3. Details drawings of several of the vessels are illustrated in Figures 4 and 5.

Vessel Number F-1

This vessel is used to transport the F-10-P solutions from the Concentration Buildings to the Isolation Building and to return recycle solutions to

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FIGURE 4

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PRECIPITATION TANK

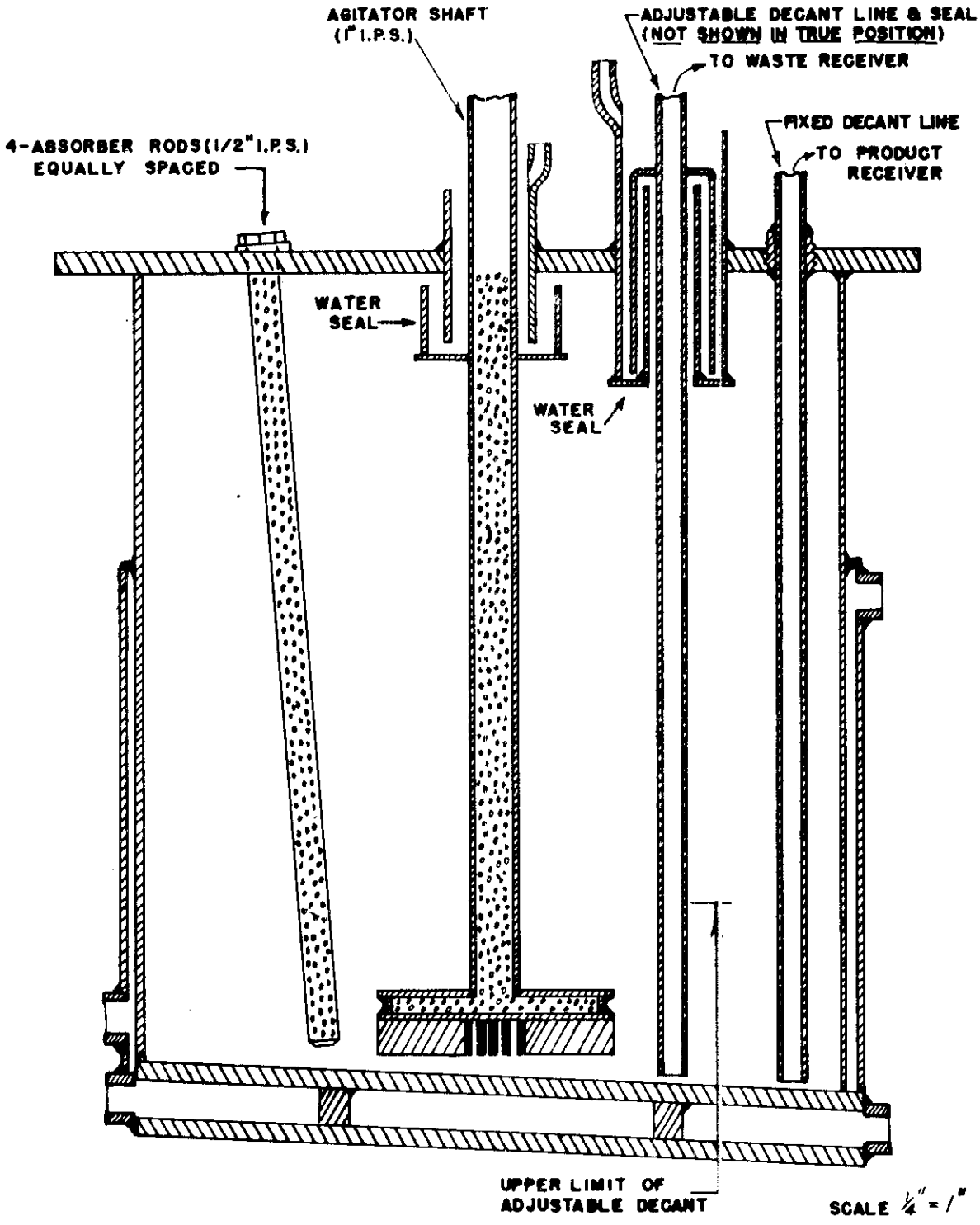

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FIGURE 5

VACUUM RECEIVER

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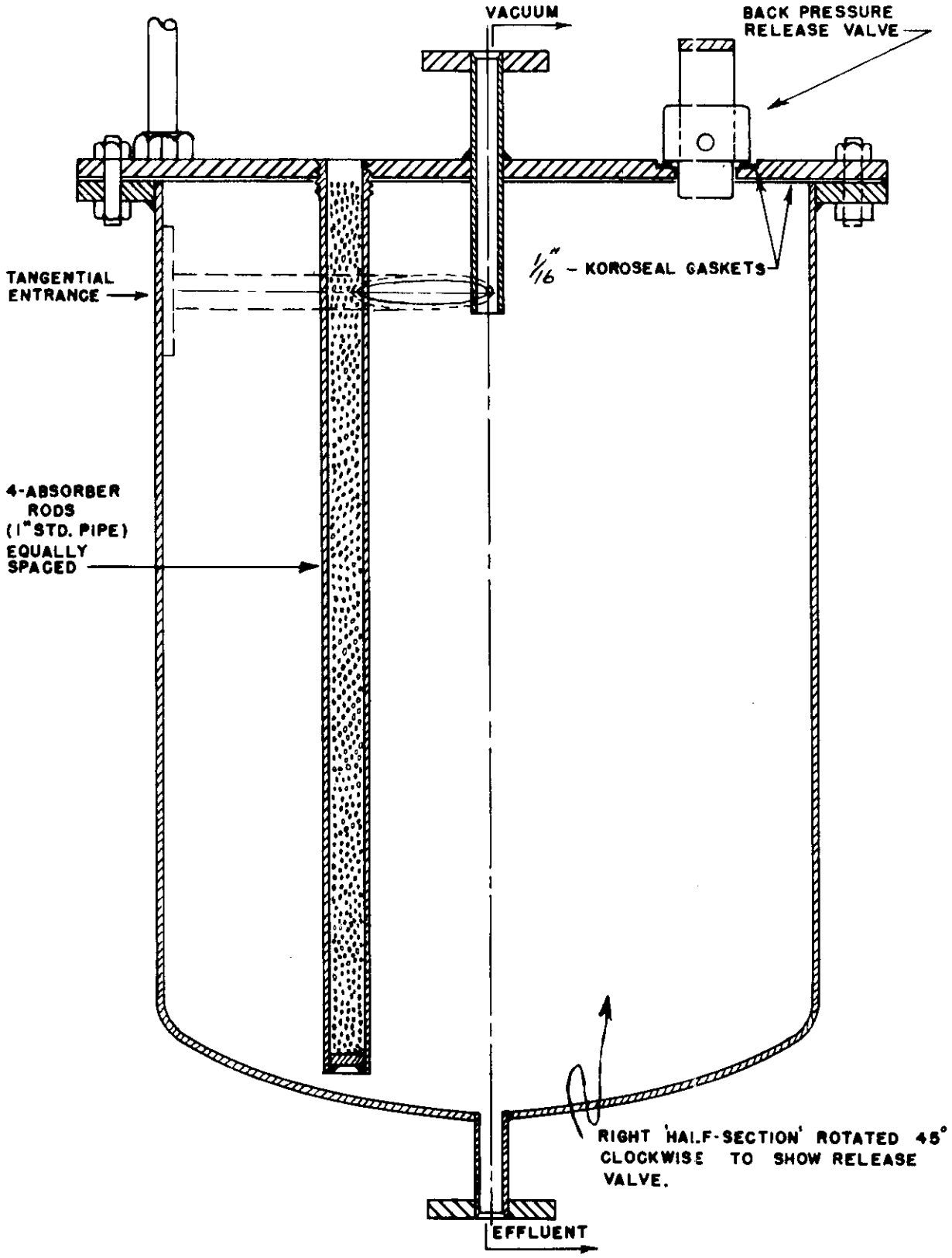

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the Concentration Building. For this reason the vessel is also known as the "recycle can". The F-1 Vessel is placed within an outer "carrier" vessel during transportation. The carrier vessel has a completely removable head which is held in place with hinged clamps. The carrier has attached stainless steel skids in order that it may be lifted and carried with a standard manually operated lift truck.

The inner vessel has one opening in the top, the inner edge of which is accurately beveled. This opening is closed with a clamp-operated, spherically ground plug, thus providing a liquid-tight, metal-to-metal seat. The opening and the area over which the plug travels is enclosed within a well on the top of the vessel. This limits the area over which any leakage may spread and further allows any leakage to be flushed into the vessel. Both the inner and outer vessels are equipped with slots or eyes for engaging hoist hooks. (See Figure 68, Chapter II for an illustration of this vessel).

In operation, the combined vessels are carried into the process cell. The inner vessel is lifted from the outer vessel by a travelling chain hoist. A hinged panel is opened at the end of the process hood and the inner vessel is carried within the hood by the travelling hoist. The vessel is then raised in place under the intake line to N-1 (or the descent line from WR-1). (For details regarding this vessel see Blueprint No. D-64263).

Vessel Number N-1

This nutsche filter is used for the filtration of the F-10-P solution. It has a total capacity of 39 gallons or 148 liters with working capacity of 60 liters. The vessels consist of a simple nutsche-type filter fabricated from 25-12 stainless steel in which a fused, porous alumina plate (Filtros Type E - Fine Porosity) is seated on a Koroseal gasket against an integral stainless steel plate. Filter aid (Hyflo-Super-Cel) is added to the vessel as a water slurry through a feed funnel. Under normal operating conditions a 30-liter charge filters in about 15 minutes. The filtrate passes to the Vacuum Receiver NR-1.

When the filtration rate slows down to an unsatisfactory level, the filter cake can be backwashed with distilled water, a connection being provided to the lower section of the filter. Lines are provided to allow the removal by jetting of backwash solutions from the upper or lower sections to F-2.

The vessel is provided with a disc-type agitator, the shaft and disc of which are filled with cadmium-lead shot. The cover plate of the vessel contains a glass illumination port as well as a sight glass. The agitator shaft enters through a water seal in the cover. (For details regarding this vessel see Blueprints No. D-64458, H-2-194, H-2-141 and H-2-185).

Vessels Number NR-1, WR-1, PR-1, WR-2, PR-2

These identical vessels are vacuum receivers (Fig. 5) used to transport product and waste solutions from tank to tank. They have a total capacity of 20 gallons or 76 liters with a working capacity of 45 liters. The movement of solutions from N-1, P-1, and P-2 depends upon lifting them by vacuum to a

receiving vessel. From the receiving vessel the solution is dropped by gravity to the next appropriate vessel.

The inlet to the vacuum receivers is made through a tangential nozzle which prevents splashing and possible entrainment of product in the exhaust to the ventilating system. The bottom of the vessel is elliptical to allow good drainage from the vessel. Vacuum is produced by air and water jets. NR-1 and PR-2 are operated by air jets because of the necessity of obtaining higher vacuum in conjunction with the N-1 and N-2 Filters. WR-1, WR-2 and PR-1 employ water jets. A weight-loaded relief valve is provided in the cover to release the back pressure in the event that a jet discharge line should become plugged. A set of four neutron absorber rods consisting of 1/2-inch pipes filled with cadmium-lead shot is attached to the cover of each vessel. (For details regarding these vessels see Blueprint No. D-65223).

Vessels Number P-1, P-2

These two identical vessels are precipitation tanks (Fig. 4). They have a total capacity of 22 gallons or 83.5 liters. Working capacity is 60 liters. The tanks have both side and bottom jackets. The side jacket is served with chilled water for cooling during precipitation and settling of the peroxide precipitate. During dissolution of the peroxide precipitate steam is passed into the bottom jacket to bring the temperature to 55 - 65 °C. Following dissolution the solution is cooled by passing chilled water into the side jacket.

The cover provided for these vessels is equipped with numerous intake and discharge lines as well as agitator and movable decant line openings. Illumination and sight ports made of glass and solution samplers are also provided. The cover is bolted to the tank and sealed with a Koroseal gasket. The product solution enters through a pipe line which is sealed into the cover by a threaded bushing. The angle of discharge is such that the liquid strikes the wall tangentially and flows down the side wall. Chemical additions are made through a funnel-opening in a valved line. The discharge of this line is directed toward the agitator shaft. This assures good distribution of reagents and flushing of the precipitate from the upper surface of the agitator shaft and disc. The supernatant and wash decant line enters the vessel through a water seal. Such an arrangement is required to allow vertical movement of the decant line. Movement of the decant line is controlled by a screw lift. A "bulls-eye" sight glass is located in the decant line so that any turbidity in the decant solution may be observed before significant quantities of precipitate are withdrawn. The disc-type agitator (filled with cadmium-lead shot) also enters through a water seal. The purpose of these water seals is to prevent the escape of mist or spray from the vessels. In addition to the neutron absorber in the agitator four filled rods attached to the cell cover project into the solution.

With the exception of a bulk-sampler connection on P-1, both vessels have the same accessory equipment. This consists of a liquid weight indicator, recording thermometer and combination sampler and liquid level indicating device. (For details regarding these vessels see Blueprints D-64468, H-2-130A, H-2-130-B, H-2-188 and H-2-185)

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Vessel Number N-2

This vessel is a smaller nutsche type filter used for the filtration of the dissolved peroxide precipitate from P-1. It has a total capacity of 8 gallons or 30 liters. Working capacity is 20 liters. There are only four pipe connections to the vessel; product inlet, top backwash draw off line, chemical feed, and combination product and bottom backwash discharge. The disc-type agitator enters the cover through a water seal. The agitator shaft and disc filled with cadmium-lead shot are the only neutron absorbers in this vessel. The cover of the vessel also has illumination and sight ports. A recording thermometer is also connected to the vessel.

The construction of this vessel differs from all others in that the body of the vessel is split into two parts. The filter plate support ring is held between the flanges of the upper and lower sections. To remove the filter plate it is necessary to drop the lower section of the vessel and remove the filter plate support ring. A Filtros Grade E, fine porosity fused alumina filter is used. (For details regarding this vessel see Blueprints D-64900, H-2-195, H-2-144 and H-2-185.

Vessel Number PR-3

This vacuum receiver vessel is used to transfer the plutonium nitrate solution from P-2 to the still, S-1. The receiver also functions as a constant volume tank for charging S-1. It has a total capacity of 9.03 liters in Cell 4, and 9.345 liters in Cell 3. An overflow line discharges any excess solution into the solution tank (ST-2). Two fixed inlet lines enter through the cover so that solution can be drawn from either P-2 or ST-2. Four absorber rods are provided in the vessel. (For details regarding this vessel see Blueprint H-2-204).

Still S-1

The still, S-1, is used for the concentration of the plutonium nitrate solution obtained from the dissolution of the second peroxide precipitate. It has a total capacity of 5.5 gallons or 20.8 liters. Working capacity is 12 liters. Steam heat is supplied through a bottom jacket operated at 50 lbs./sq.in. pressure. Since the operation is an evaporation rather than a distillation, an open column consisting of a 3-foot length of 3-inch 25-12 stainless steel pipe is adequate for the separation of entrained materials. A simple, parallel-downflow condenser, consisting of a 2-inch, 25-12 stainless steel pipe is used. The entire system including the condensate receiver, R-1, is closed. Non-condensable gases are drawn off through the ventilation system. The volume of solution remaining in the still is determined by subtracting the condensate volume in R-1 from the known charging volume and applying an empirical correction. The condensate in R-1 is jetted to F-2 from which it may be sent to waste.

The still pot of S-1 contains a neutron absorber in the form of a "spider". This consists of a triangle made of stainless-steel pipe resting on short legs. The pipe is filled with cadmium-lead shot. A recording thermometer is also connected to the still pot of S-1. (For details regarding this vessel see Blueprint D-64497).

[redacted]

Vessel Number SR-1

This vacuum receiver vessel is used to transfer the concentrated plutonium nitrate solution from S-1 to the adjustment tank (AT). It has a total capacity of 1.8 gallons or 6.8 liters and a working capacity of 2 liters. SR-1 is a cylindrical vessel with a sloped bottom. Solution from S-1 enters through the top through a tangential nozzle. Another line entering through the top of the vessel allows the transfer of product solution from a sample can into SR-1. Vacuum is supplied by a water jet. The solution in SR-1 is drained into AT tank or may be transferred into the solution tank (ST-2). No neutron absorbers are provided. (For details regarding this vessel see Blueprint D-64861).

Vessel AT

The adjustment tank (AT) is a small vessel of 0.72-gallon or 2.73-liter total capacity with a working capacity of approximately 2 liters. Its original function (and hence the name) was to allow the adjustment of the plutonium nitrate solution to a fixed concentration. At present it is used for sampling purposes. The cover of the vessel is provided with a chemical feed line (not used at present), a sight port, a vent line, a hand operated agitator and a gate valve for sampling (the valve is opened and a pipette inserted into the tank). Inside the vessel a calibrated scale for volume measurements is also provided. The tank is filled and drained through pipes which enter through the bottom. The bottom has a slight slope to improve drainage. The solution in the AT tank may be transferred to the weigh tank (WT) or the solution tank (ST-2). No neutron absorbers are present in this vessel. (For details regarding this tank see Blueprints H-2-148-A,B,C, and H-2-215).

Vessel WT

The weigh tank (WT) is a freely floating vessel suspended from a balance. It has a total capacity of 0.9 gallons or 3.4 liters and a working capacity of 2 liters. The inlet and vent lines are rigidly supported and enter it through water seals which allow the movement of the tank. The tank drains through an outlet pipe located at the lowest edge of the steeply sloped bottom. In draining, the sample can is raised into position under the outlet pipe. (For details regarding this vessel see Blueprints Number H-2-131, H-2-199).

Sample Can

The sample can is the final shipping container for the plutonium. This container (made of 18-8) is a small cylindrical vessel with a spherical bottom. The total capacity is about 1.2 liters and has a maximum working capacity of about 1 liter. A threaded plug containing a small vent fits into the neck of the vessel. Above this plug a special cap containing a filter medium (Johns Manville, Chemical Warfare Service Specifications 197-54-249) is fitted. The filter medium is held in place by wire screens. A small nozzle extends from the top of the cap. The purpose of the vents and of the filter medium is to allow the release of gas pressure which is generated within the vessel. A pair of trunion pins extend from near the top of the container. These pins are used for supporting the sample can within the hood.

[redacted]

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The sample can is placed in an outer cylindrical container for transportation. The outer container has a large flat base. It is also provided with a lid held in place with thumb screws, and a handle.

The sample can in its shipping container is illustrated in Figure 84, Chapter II. (For details regarding this sample can see Blueprint No. D-64837).

Vessel CT-1

This is a catch tank (CT) and is used for the decompcsition of H_2O_2 in the peroxide supernatants. It has a total capacity of 53.5 gallons or 203 liters and a working capacity of 180 liters. The vessel is jacketed completely on the sides. Cold water is circulated only. The tank is equipped with sight and illumination ports, agitator, chemical feed line, sampler, and the bimetallic electrode system (previously described) enclosed in a standpipe. (For details regarding this vessel see Blueprints No. H-2-202, H-2-130 and H-2-185).

Vessel CT-2

This vessel is a spare catch tank, smaller than CT-1 and of the same size and general appearance as P-1 and P-2. It has a total capacity of 22 gallons or 83.5 liters and a working capacity of 60 liters. It has no movable decant line. CT-2 is used occasionally for H_2O_2 decomposition. Since no electrode system is provided, the end point of the reaction is determined by noting the behavior of small additions of $KMnO_4$ to the solution. If effervescence is observed, indicating excess H_2O_2, more $NaNO_2$ is added, until further additions of $KMnO_4$ indicate that all H_2O_2 is destroyed. (For details regarding this vessel see Blueprints D-64468, H-2-130, H-2-188, and H-2-185).

Vessel ST-2

This is a small vessel having the same general features found in the other vessels, that is, agitator, sight and illumination ports and a sampler. It has a total capacity of 7 gallons or 26.5 liters and a working capacity of 18 liters. ST-2 serves the general utility purposes of a holding tank and of returning various solutions from the final part of the process to earlier parts. Thus, ST-2 collects the overflow from PR-3, and SR-1 and AT may be drained into it. From ST-2 solutions may be returned to either PR-1, PR-2 or PR-3. (For details regarding this equipment see Blueprints D-64471, H-2-193, H-2-140, H-2-185)

Vessel R-1

The condensate receiver (R-1) is a displacement type vessel used to collect and measure the volume of distillate from S-1. It has a total capacity of 10 liters which also is its working capacity. The vessel consists of an outer glass vessel in which a large stainless steel cylinder rests. Hence, although the overall size of R-1 is quite large, the distillate (about 9 liters) fills only a small ring between the steel cylinder and the glass wall and the volume may be read from a calibrated scale with good accuracy. No sampler is incorporated in R-1. The distillate may be discharged to F-2 for sampling and disposal. (For details regarding this vessel see Blueprints Number H-2-198, H-2-145).

[redacted]

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Vessel F-2

This is a large general utility vessel used to collect the back washes of filters N-1 and N-2, and leachings from filter N-1. It has a total capacity of 100 liters. The distillate from R-1 is collected in F-2. Washes of the hood may be drawn up from the floor of the hood to be sampled. F-2 may be discharged directly into the sump tank or into an F-1 container. F-2 is equipped with an agitator, sampler, and sight and illumination ports. (For details regarding this vessel see Blueprints No. D-64467, H-2-197, and H-2-185).

Vessel VR-1

This is a small vent receiving vessel. Its total capacity is 5.14 gallons or 19.5 liters and the working capacity is 15 liters. All sprays and condensed vapors are collected in VR-1 before exhausting to the outside. The condensate collected in VR-1 may be jetted into WR-2. (For details regarding this vessel see Blueprint No. H-2-216).

Plans are under consideration for the installation of a filter (N-3) between the still receiver (SR-1), and the adjustment tank (AT), for the purpose of filtering the final product solution prior to final evaporation.

The equipment for the evaporation of the final product solution is illustrated diagrammatically in Figure 82, Chapter II. (For details regarding the drying equipment see the following Blueprints H-2-247, H-2-323, H-2-309, H-2-265, H-2-271).

Accessory Equipment

Recording Thermometers

A resistance-type thermometer connected to a recording Leeds and Northrup "Thermohm" Micromax is employed. Four-point micromaxes are used so that two of these instruments are sufficient for each process cell. The thermometer well, in which the resistance element is located, is attached to the vessel cover with a threaded bushing. The lower end of the well extends nearly to the bottom of the vessel so that it is possible to determine the temperature of all process solutions. The upper end of the thermometer well extends through the hood panel.

Manometers

The weight of solution in the various vessels is determined by a manometer system. A 1/8 inch pipe extends to the bottom of the vessel and a small flow of air is passed through the pipe. The liquid head is read directly on the manometer and the solution weight is found by reference to a calibration chart.

Samplers

Sampling of the process vessels is accomplished by means of a vertically travelling sample cup keyed to a spiral-cut cam. The cam is mounted within a closed tube which projects above the tank to a point flush with the top of

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the hood. A sliding plate covers the opening to the tube except when a sample is being removed. The capacity of the sample cup is about 1 ml. A revolution counter is connected to the handle of the sampler. Liquid level may be determined by means of this counter after prior calibration. (For details regarding this sampler see Blueprints No. H-2-210 and H-2-135).

A bulk sampler is also provided for P-1. A dip pipe (1/4-inch) extends nearly to the bottom of the vessel. The upper end of the dip pipe is expanded to contain a valve and a tight fitting ground seat into which the actual sampler is placed. The sampler consists of a small cylindrical vessel of 100-ml. capacity. Valves are located above and below the vessel. A short section of pipe, tapered to fit the seat provided in the dip pipe, extends below the lower valve. The sampler is first evacuated by connecting it to a vacuum line and opening the upper valve and closing the lower valve. When evacuated, the upper valve is closed, the sampler connected to the dip pipe, and the lower valve opened to draw solution into the sampler. The lower valve is then closed and the sampler removed. A cap is provided to fit the end of the sampler. (For details regarding this sampler see Blueprints No. H-2-214 and H-2-150).

Agitators

All the agitators in the various vessels are powered by slow speed, air driven motors. The operating speed range of these units is 30 to 120 rev./min.

Chemical Handling

All chemical solutions are transported from the solution makeup room in stainless steel containers. These containers have a wide neck (with a lid) for filling. The solutions are drained through a valve located at the bottom of the container. The containers are brought into position and supported during draining on travelling chain hoists. The solutions drain into the vessel through a feed line equipped with a funnel and valve.

Cooling Water

The process cell is provided with cooling water of two temperatures, 20 and 5 °C. The 5° water is supplied from a refrigerating system. The 20° raw water is used in the operation of the still.

Valve Packing

All valve stems in the processing cells are packed using blue African asbestos lubricated with a non-saponifiable grease (Merco-Nordstrom 147 Special Lubricant). This packing has been found to hold up well in practice.

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REFERENCES

1. Report CN-261
2. Report CN-328
3. Report CN-343
4. Report CN-319
5. Report CN-914
6. Report CN-1041
7. Report MUC-GTS-285
8. Report CL-697
9. Report CS-2438
10. Letter CL-P-376
11. Letter CL-P-354
12. Report CN-2158
13. Report CK-1511
14. Letter CL-P-395
15. Letter CL-P-387
16. Report CN-1946
17. Letter CI-P-361
18. Report CS-2614
19. Report CN-1370
20. Report CN-1585
21. Letter MUC-GTS-998
22. Letter CI-P-196
23. Letter CI-P-209
24. Report CN-1700
25. Report CN-2085
26. Report SE-PC-35
27. Report CN-1510
28. Letter CL-P-274
29. Letter CL-P-368
30. Letter CL-P-327
31. Report CN-1762
32. Report SE-PC-20

[redacted]

APPENDIX

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FLOW SHEET

Table III on the following pages gives the flow sheet of the isolation process as practiced about September 1, 1945.

LOG SHEETS

Following the flow sheet is given a complete set of operating log sheets for the isolation process. These log sheets in themselves constitute the detailed operation instructions for the process.

[redacted]

[redacted]

APPENDIX

TABLE III

ISOLATION FLOW SHEET

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Building 231

A. Filtration of F-10-P Solution

1) Receive 34 kilograms (30.3 liters; 8 gal.) of F-10-P solution in PR container from Concentration (224) Building.

2) Jet solution from PR container to filter N-1 containing 1.5 kilogram precoat of elutriated Hyflo-Super-Cel.

3) Add a slurry containing 30 grams of elutriated Hyflo-Super-Cel in 200 ml. of water.

4) Filter by suction, drawing filtrate to vacuum receiver NR-1.

5) Rinse lines from PR to NR-1 with 500 grams (485 ml.) of 6% HNO_3.

6) Drop solution from NR-1 to precipitator P-1.

7) Take P-1 sample for production assay and acidity.

B. Pre-Reduction

1) Add 1.85 kg. (1.5-l.) of 42% $(NH_4)_2SO_4$.

2) Adjust temperature to 25 °C.

3) Add 600 grams (510 ml.) of 30% $(NH_4)_2SO_3$.

4) Agitate for 30 minutes.

5) Adjust acidity to a value between 1.8 and 2.2N with 60% HNO_3 or 25% KOH.

Solution Composition (in P-1)

	Kilograms	%
Product	0.375	1.0
$La(NO_3)_3$	2.2	5.75
KNO_3 (estimated)	0.5	1.3
$(NH_4)_2SO_4$	0.78	2.0
$(NH_4)_2SO_3$	0.18	0.45
HNO_3	3.87	10.1
H_2O	30.4	79.4
Total	38.3	(34 l.)

[redacted]

[redacted]

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C. First Precipitation, Decantation, and Washing

1) Adjust P-1 solution to 20 °C.

2) Add 15.75 kg. (14.0 l.) of 30% H_2O_2 over a 20 to 30 minute period.

3) Agitate for 1 hr. at 20 °C.

4) Cool P-1 solution to 6 °C.

5) Allow slurry to settle for 2 hours at 6 °C.

6) Draw supernate from P-1 to vacuum receiver WR-1, lowering decant line slowly through maximum distance without disturbing settled solids. (Heel about 20-25 ml./g. product).

7) Drop supernate to catch tank CT-1 and hold at 7 °C.

8) Add 6 kg. (6-l.) of 2% H_2SO_4 to P-1.

9) Agitate for 5 minutes.

10) Allow solids to settle for 1/2 hour.

11) Decant to WR-1.

12) Repeat item 8.

13) Repeat item 9.

14) Repeat item 10.

15) Repeat item 11.

16) Repeat item 8.

17) Repeat item 9.

18) Repeat item 10.

19) Repeat item 11.

20) Drop washes from WR-1 to CT-1.

[redacted]

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Solution Composition (in CT-1)

	Kilograms	%
Product	0.005	0.1
$La(NO_3)_3$	2.14	3.35
KNO_3	0.49	0.77
$(NH_4)_2SO_4$	0.75	1.17
$(NH_4)_2SO_3$	0.17	0.27
HNO_3	3.77	5.91
H_2SO_4	0.36	0.56
H_2O_2	4.2	6.58
H_2O	51.8	81.2
Total	63.8	(58.5-1.)

D. Dissolution and Filtration

1) Add 2.3 kg. (1.72-1.) of 60% HNO_3 to P-1.

2) Heat slowly (over 30 minutes) to 60-65 °C.

3) Hold at 60-65 °C for 1 hour.

4) Cool to 25 °C.

5) Transfer solution from P-1 to vacuum receiver PR-1.

6) Add 5 kg. of 1.2% HNO_3 to P-1 and agitate for 5 minutes.

7) Draw wash from P-1 to PR-1.

8) Drop to filter N-2.

9) Add a slurry containing 20 grams of elutriated Hyflo-Super-Cel in 100 ml. of water.

10) Filter by suction, drawing filtrate to vacuum receiver PR-2.

11) Drop solution from PR-2 to precipitator P-2.

Solution Composition (in P-2)

	Kilograms	%
Product	0.370	2.37
$La(NO_3)_3$	0.06	0.39
$(NH_4)_2SO_4$	0.025	0.16
KNO_3	0.01	0.06
HNO_3	1.14	7.31
H_2O	13.99	89.71
Total	15.6	(14.3-1.)

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E. Second Precipitation, Decanting, and Washing

1) Add water to bring P-2 solution weight to 16 kg.

2) Add 400 grams (220 ml.) of 96% H_2SO_4 to P-2.

3) Cool P-2 solution to 20 °C.

4) Add 8.0 kg. (7.2-l.) of 30% H_2O_2 to P-2 over a 20 to 30 minute period.

5) Agitate for 1 hour at 20 °C.

6) Cool to 6 °C.

7) Allow slurry to settle for 2 hours at 6 °C.

8) Draw supernate from P-2 to vacuum receiver WR-2, lowering decant line slowly through maximum distance without disturbing settled solids. (Heel about 20-25 ml./g. product).

9) Drop supernate to CT-1 and hold at 7 °C.

10) Add 6 kg. (5.85-l.) of 6% HNO_3 to P-2.

11) Agitate for 5 minutes.

12) Allow solids to settle for 1/2 hour.

13) Decant to WR-2.

14) Repeat item 10.

15) Repeat item 11.

16) Repeat item 12.

17) Repeat item 13.

18) Repeat item 10.

19) Repeat item 11.

20) Allow solids to settle for 1 hour.

21) Decant to WR-2.

22) Drop washes from WR-2 to CT-1.

[redacted]

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Solution Composition (in CT-1)

	Kilograms	%
Product	0.001	0.003
$La(NO_3)_3$	0.057	0.17
KNO_3	0.01	0.03
$(NH_4)_2SO_4$	0.024	0.07
H_2SO_4	0.364	1.16
HNO_3	1.96	5.7
H_2O_2	2.4	6.97
H_2O	29.6	85.9
Total	34.4	(32.5-1.)

F. Dissolution and Transfer to Still

1) Add 2.73 kg. (2.0-1.) of 60% HNO_3 to P-2.

2) Heat slowly (over 30 minutes) to 55-60 °C.

3) Hold at 55-60 °C until precipitate dissolves.

4) Cool to 25 °C.

5) Draw solution from P-2 to vacuum receiver PR-3.

6) Add water to PR-3 to bring volume to 9 liters.

7) Drop solution from PR-3 to evaporator S-1.

Solution Composition (in S-1)

	Kilograms	%
Product	0.369	3.45
HNO_3	1.44	13.5
H_2SO_4	0.02	0.19
$(NH_4)_2SO_4$	0.001	0.01
$La(NO_3)_3$	0.003	0.03
H_2O	8.87	82.9
Total	10.7	(9-1.)

G. Concentration and Sample Can Loading

1) Evaporate to 1.67 liters in S-1 as indicated by volume of condensate received in R-1.

2) Cool solution in S-1 to 50 °C.

[redacted]

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3) Draw solution from S-1 to vacuum receiver SR-1.

4) Drop from SR-1 to "adjustment tank" AT.

5) Sample solution in AT.

6) Drop calculated weight of solution from AT to weigh tank WT.

7) Drop solution from WT to sample can SC.

8) Transfer condensate from R-1 to receiver F-2 by steam jet.

9) Sample and dispose of solution in F-2.

Solution Composition (in SC)

	Kilograms	%
Product nitrate	0.326	27.7
HNO_3	0.58	49.3
H_2SO_4	0.009	0.76
$(NH_4)_2SO_4$	0.0004	0.03
$La(NO_3)_3$	0.0013	0.11
H_2O	0.26	22.1
Total	1.18	(0.71-1.)

H. Final Evaporation in Sample Can

1) Connect SC to final evaporation equipment.

2) Bring SC to 125 °C by glycerine bath.

3) Collect a volume of condensate calculated to leave 390 grams of semi-solid in SC.

4) Cool SC to room temperatures, pack, and ship.

Composition of Material in SC

	Kilograms	%
Product Nitrate	0.326	83.6
HNO_3	0.053	13.6
H_2SO_4	0.009	2.3
$(NH_4)_2SO_4$	0.0004	0.1
$La(NO_3)_3$	0.0013	0.3
Total	0.390	(0.19-1.)

[redacted]

APPENDIX

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I. Treatment of Combined Supernatant Solutions in CT-1

1) Add 44.8 kilograms (39.4 liters) of 20% $NaNO_2$ at a rate to keep temperature below 25 °C.

2) Polarize bimetallic electrode system with platinum positive.

3) Add additional 20% $NaNO_2$ (assume 12.5 kg. or 11-l.) slowly until recording photoelectric microammeter shows sharp deflections indicating all H_2O_2 destroyed.

4) Add 4% $KMnO_4$ (assume 4 kg. or 4-l.) until a permanent pink color indicates excess nitrite destroyed.

5) Sample solution in CT-1 to assay recycle.

6) Transfer solution to 224 Bldg. via WR-1 and RC containers.

Solution Composition (in CT-1)

	Kilograms	%
Product	0.006	0.0036
$La(NO_3)_3$	2.2	1.3
KNO_3	0.6	0.36
$(NH_4)_2SO_4$	0.94	0.56
H_2SO_4	0.72	0.43
HNO_3	5.6	3.35
$NaNO_3$	14.1	8.45
$Mn(NO_3)_2$	0.18	0.11
H_2O	142.7	85.4
Total	167	(149-l.)

[redacted]

[redacted]

Date _____
Run No. _____

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INCOMING MATERIAL

Date Received _____ Time _____

Source _____ Code _____

Container No. _____ Beckman Survey _____

231 BUILDING PROCESSING

Run No. _____ Cell No. _____

Date Started _____ Time _____

Date AT assay received _____ Time _____

REMARKS:

[redacted]

Date _____
Run NO. _____

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I - FIRST CYCLE - RECEIPT OF SOLUTION

1. Receive product solution from Vault "A"
   1. Date _____
      Time _____
      Loaded Wt. _____
      Unloaded Wt. _____
      Net Wt. _____

2. Obtain Beckman and Sandy readings on PR container.
   2. Mr/hr _____
      Sandy _____
      Time _____

II - FIRST CYCLE - FILTER CAKE IN N-1

Leave the filter cake undisturbed. We will continue to use this filter cake for subsequent runs until need for a change is indicated. When this occurs, use regular log for "Backwashing and Precoating N-1".

III - FIRST CYCLE - FILTRATION

1. Check state of system. Simulate transfer of solution from N-1 to NR-1 and dropping solution to P-1. If a significant amount of solution (1 Liter or more) is carried to P-1 do not proceed until Senior Supervisor has been advised.
   1. System checked _____

2. Sol'n Transfer — Transfer solution from F-1 to N-1. Start transfer with steam jet, then, allow the solution to siphon from F-1 to N-1
   AGITATOR AT REST - NO VACUUM ON NR-1.
   2. Time start _____
      Time end _____

3. Filter Aid Add'n — Prepare filter aid by slurrying 30 gms of elutriated filter aid in 200 cc of H_2O. Add to N-1 via funnel. Rinse funnel with about 50 ml of water.
   3. Wt. of Slurry added: _____

4. Filtration — Filter from N-1 to NR-1 using full vacuum.
   4. Time start _____
      Time end _____

5. Add 500 gm of 6% HNO_3 to F-1 via funnel. Rinse funnel with about 50 ml of water.
   5. Wt. 6% HNO_3 added _____ gm

6. Transfer wash solution from F-1 to N-1 with steam jet.
   6. Time end _____

Date _____
Run No. _____

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III - FIRST CYCLE - FILTRATION (CONT'D.)

7.	Draw wash from N-1 to NR-1	7. Time end_____
8.	Protect delivery line with drip pan.	8. Done by_____
9.	Add water to liquid seals on P-1.	9. Done by_____
10.	Drop filtered solution and washes from NR-1 to P-1. Start agitator and run at 30 RPM.	10. Time end_____ Sampler Turns_____ Sol'n Volume_____L Man. Rdg._____ Sol'n Wt. in P-1_____Kg
11.	Agitate for 5 minutes at 30 RPM. Take Beckman reading on P-1.	11. Beckman_____
12.	Take a bulk sample consisting of at least 10 grams of solution.	12.
P-1 Sample		
	a. Check that sampler is clean and dry.	a. Checked by_____
	b. Insert sampler into dip pipe connection. Attach rubber bulb blower and force air through sampler and dip pipe.	
	c. Take tare weight of sampler.	c. Tare Wt._____gm
	d. Evacuate sampler. Insert into dip pipe connection and withdraw sampler.	
	e. Take gross weight of sampler.	e. Gross Wt._____gm Net Wt._____gm
	f. Fill out a sample label tag.	f. Time done_____ Serial No._____

[redacted]

Date _____
Run No. _____

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IV - FIRST CYCLE - PRE-REDUCTION

1.	With agitation at about 80 RPM add 1.85 Kg of 42% $(NH_4)_2SO_4$. (Ammonium sulfate)	1. $(NH_4)_2SO_4$ added _____ Kg Time end. _____
2.	Adjust temperature to 25°C (±2°C).	2. Time start _____ Time end. _____
3.	Add 600 gm of 30% pre-reduction chemical. Rinse funnel with about 50 ml of water.	3. Amt. Added _____ gm
4.	Continue agitation for thirty minutes.	4. Time end. _____
5.	Cool to 20° C (± 2° C).	5. Time end. _____
6.	HNO_3 assay reported by laboratory on sample P-1.	6. _____ N
	If the assay reported is between 1.8N and 2.2N, proceed to Section V.	Sampler Rdg. _____ Solution vol. _____ L
Adjust Acidity	If the assay is less than 1.8N, add 60% HNO_3. Quantity of HNO_3 to be added as determined by reference to chart	60% HNO_3 added _____ Kg
	If the assay is greater than 2.2N, add 50% KOH. Quantity of KOH to be added as determined by reference to chart.	50% KOH added _____ Kg

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

V - FIRST CYCLE - PRECIPITATION

1. Determine initial solution weight and volume in P - 1.

   Lower decant line to maximum Strike depth then raise decant line and sampler to upper position.

   Agitate at 30 RPM and maintain temperature at 20°C ± 2°C during strike.

   If the solution weight is between 35 and 40 Kg, add 15.75 Kg (14.0 L) of 30% H_2O_2. If the solution weight is greater than 40 Kg, add the 15.75 Kg of 30% H_2O_2 plus an additional .50 Kg for each Kg of solution weight in excess of 40 kg. Add the H_2O_2 over a period of 20 to 30 minutes at a uniform rate. Rise funnel with about 50 ml of water.

1. Sampler turns* _____
   Initial volume _____
   Man. Rdg. _____

   Initial weight _____ Kg
   Wt. H_2O_2 added _____ Kg
   Time start _____
   Time end _____

2. Agitate at 30 RPM for 1 hour at 20°C (± 2°C).

2. Time end _____

3. Cool to 6°C (± 2°C) with agitation at about 30 RPM.

3. Time end _____

4. Stop agitator. Determine final solution weight. SHUT OFF LIQUID LEVEL BUBBLER. Allow to settle Settle 2 hours at 6°C (± 2°C).

4. Man. Rdg. _____
   Soln. Wt. _____
   Time end _____

[redacted]

[redacted]

Date_____
Run No._____

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VI - FIRST CYCLE - DECANTATION AND WASH

Decant Supernatant	1. Lower decant line slowly. Draw supernatant from P-1 to WR-1.	1. Time start_____ Time end_____
	2. Add H_2O to liquid seal on CT-1. Start cooling CT-1 and drop supernatant from WR-1 to CT-1. Cool to and maintain temperature at 7 °C (± 3 °C).	2. Water seal checked by: _____ Man. Rdg. _____ Soln. Wt. _____ Turns on sampler _____ Volume by sampler _____
	3. Take Beckman reading on CT-1	3. Beckman_____
1st Wash	4. Add 6 Kg of 2% H_2SO_4 to P-1 with agitator running at 30 RPM during addition. Agitate for five minutes after addition.	4. Wt. H_2SO_4 added_____ Kg Time end _____
	5. Shut off agitator. Settle until supernatant is clear or a minimum time of ½ hour.	5. Time end _____
	6. Lower decant line slowly and draw supernatant from P-1 to WR-1. Decant as far as possible without disturbing precipitate. Raise decant line.	6. Time end_____
2nd Wash	7. Add 6 Kg of 2% H_2SO_4 to P-1 with agitator running at 30 RPM during addition. Agitate for 5 minutes after addition.	7. Wt. H_2SO_4 added_____ Kg Time end_____
	8. Shut off agitator. Settle until supernatant is clear or a minimum of ½ hour.	8. Time end_____
	9. Lower decant line slowly and draw supernatant up to WR-1 from P-1. Decant as far as possible without disturbing precipitate. Raise decant line.	9. Time end _____
3rd Wash	10. Add 6 Kg of 2% H_2SO_4 to P-1 with agitator running at 30 RPM during addition. Agitate for 5 minutes after addition.	10. Wt. H_2SO_4 added_____ Kg Time end _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VI - FIRST CYCLE - DECANTATION AND WASH (Cont'd.)

11.	Shut off agitator. Settle until supernatant is clear or a minimum of ½ hour.	11. Time end _____
12.	Lower decant line slowly and draw supernatant up to WR-1 from P-1. Decant as far as possible without disturbing precipitate. Raise decant line.	12. Time end _____
13.	Drop washes from WR-1 to CT-1. Hold temperature at 7° C (± 3° C).	13. Time end _____

VII - FIRST CYCLE - DISSOLUTION

1.	Check water seals on P-1. Run agitator at about 30 RPM.	1. Done by _____
2.	Via chemical feed funnel add slowly 2.00 Kg (1.5 liters) of 60% HNO_3 to P-1.	2. HNO_3 added _____ Kg
3.	Via bulk sampler, add 300 gm. (220 ml.) of 60% HNO_3 to P-1.	3. HNO_3 added _____ gm
4. Dissolve	Raise temperature slowly over a 30 minute interval to 60-65° C. Hold at 60-65 C until precipitate is dissolved. (Minimum of 1 hour.	4. Time start _____ Time @ 60° C _____ Time end _____
5. Cool	With agitator at 30 RPM reduce temperature to 25° C (± 5°C). Stop agitator and measure volume and weight of solution.	5. Man. Rig. _____ Wt. of Sol'n. _____ Sampler turns _____ Solution Vol. _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

VIII - FIRST CYCLE - FILTRATION OF SOLUTION OF FIRST PRECIPITATE

NOTE: The existing bed of filter aid on the N-2 plate is not to be removed until the rate of filtration becomes unsatisfactory. When necessary use authorized leg, "Backwashing and Precoating Of N-2".

1.	Transfer solution from P-1 to PR-1.	1. Time start _____
2.	Add 5 Kg. of 1.2% HNO_3 to P-1.	2. Wt. added _____
3.	Agitate P-1 for 5 minutes at 30 RPM.	3. Time end _____
4.	Transfer wash from P-1 to PR-1	4. Time end _____
5.	Check valves to prevent filtered solution from passing to F-2 during subsequent operations.	5. Done by _____
6.	Fill water seals on N-2 and P-2. Lower decant line on P-2 maximum distance, then raise to upper position.	6. N-2 by _____ P-2 by _____
7.	With both agitation and suction off, slowly drop solution from PR-1 to N-2.	
8.	Add 20.0 gms of elutriated filter aid slurried in 100 ml of H_2O to N-2 via funnel. Rinse funnel with about 25 ml of water.	8. Amt. filter aid _____ Time added _____
9.	Apply full suction to N-2 via PR-2; start N-2 agitator at 30 RPM.	9. Time start _____ Time end _____
10.	Drop solution from PR-2 to P-2.	10. Time start _____
11.	Stop N-2 agitator.	11. Time off _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

IX - SECOND CYCLE - PRECIPITATION

Special irregular operation - to be carried out only when authorized by Senior Supervisor.	Authorized by _____
Transfer ___ Kg. of solution from ST-2 to P-2 via PR-2.	Kg. transferred _____ Time end _____

1.	Dilute the solution in P-2 with distilled water to final weight of 16 Kg. (_____ Kg. when above special operation performed.)	1. Time start _____ Water added _____ Man.Reading _____ Final soln. Wt. _____
2.	Add 400 gm. of 96% H_2SO_4. In addition, add 25 gm of 96% H_2SO_4 for each Kg of solution in excess of 17 Kg.	2. 95% H_2SO_4 _____ gm
3.	Cool to 20° C (± 2° C).	3. Time @ 20° C _____
4.	Determine solution weight in P-2.	4. Man.Rdg. _____
	Agitate at 30 RPM. Maintain temperature at 20° C (± 2° C). Raise decant line and sampler to upper position.	Soln. Wt. _____
	If the solution weight is between 15 and 18 Kg. add 8.0 Kg (7.2 L) of 30 H_2O_2. If solution weight	H_2O_2 added _____ Kg
Strike	is greater than 18 Kg. add the 8.0 Kg. plus 0.5 Kg. for each Kg. of solution in excess of 18 Kg. Add over a period of 20 to 30 minutes with uniform rate.	Time start _____ Time end _____

[redacted]

Date_____
Run No._____

DECLASSIFIED

IX - SECOND CYCLE - PRECIPITATION (Cont'd)

Di-ges-tion	5. Agitate at 30 RPM for 1 hour at 20°C (± 2°C).	5. Time end _____
	6. Cool to 6°C (± 2°C) with agitation at about 30 RPM.	6. Time end _____
Sett-ling	7. Stop agitator. Make final reading of solution weight. TURN OFF LIQUID LEVEL BUBBLER. Settle for two hours. Maintain temperature at 6°C (± 2°C).	7. Time start _____ Man. Rdg. _____ Soln. Wt. _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

X - SECOND CYCLE - DECANTATION AND WASH

	1. Lower decant line slowly and draw supernatant from P-2 to WR-2. Decant as far as possible without disturbing precipitate. Raise decant line.	1.	Time end _____
	2. Fill water seals on CT-1 tank.	2.	Done by _____
	3. Drop supernatant from WR-2 to CT-1. Hold temperature at 7°C (± 3°C).	3.	Time end _____
1st Wash	4. Add 6 kg of 6% HNO_3 to P-2. Agitate for 5 minutes at 30 RPM.	4.	Wt. of HNO_3 _____ Time end _____
	5. Turn off agitator and settle until supernatant is clear - not less than ½ hour minimum time.	5.	Time start _____ Time end _____
	6. Lower decant line slowly and draw supernatant from P-2 to WR-2. Decant as far as possible without disturbing precipitate. Raise decant line.	6.	Time end _____
2nd Wash	7. Add 6 Kg of 6% HNO_3 wash to P-2. Agitate for 5 minutes at 30 RPM.	7.	Wt. HNO_3 added _____ Time end _____
	8. Turn off agitator and settle until supernatant is clear - not less than ½ hour minimum time.	8.	Time start _____ Time end _____
	9. Lower decant line slowly and draw supernatant from P-2 to WR-2. Decant as far as possible without disturbing precipitate. Raise decant line.	9.	Time end _____
3rd Wash	10. Add 6 Kg of 6% HNO_3 wash to P-2. Agitate for 5 minutes at 30 RPM.	10.	Wt. HNO_3 added _____
	11. Turn off agitator and settle until supernatant is clear - not less than one hour minimum time.	11.	Time start _____ Time end _____
	12. Lower decant line slowly and draw supernatant from P-2 to WR-2. Decant as far as possible without disturbing the precipitate. Raise decant line.	12.	Time end _____
	13. Drop combined wash solutions from WR-2 to CT-1. Hold temperature at 7°C (± 3°C).	13.	Time end _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

XI - SECOND CYCLE - DISSOLUTION

1. Read volume in P-2.	1. Sampler Rdg. _____ P-2 Volume _____
2. Secure approval of Supervisor to proceed.	2. Approved by _____
3. Add H_2O to seals on P-2. Bring agitator to about 30 RPM.	3. H_2O checked _____ Time start _____
4. Via funnel, add 2.73 Kg (2.00 liters) of 60% HNO_3 slowly to P-2.	4. 60% HNO_3 added _____
5. Raise temperature uniformly over a 30 minute interval to 60-65°C. Hold at 60-65°C until precipitate is dissolved. (Minimum of 1 hour)	5. Time start _____ Time at 60°C _____ Time dissolved _____
6. Cool P-2 to 25°C (± 2°C). Stop agitation.	6. Time at 25°C _____ Man. Rdg. _____ Soln. Wt. _____

XII - SECOND CYCLE - TRANSFER TO STILL

1. Measure solution volume in P-2.	1. Sampler Rdg. _____ P-2 Vol. _____
2. Open valve in overflow line from PR-3 to ST-2. If no flow occurs or when flow stops, close valve.	2. Checked by _____
3. Transfer solution from P-2 to PR-3.	3. Time end _____
4. Open valve in PR-3 to ST-2 overflow line.	
5. Add 6% HNO_3 to PR-3 from a weighed reagent can until discharge is first observed on the overflow line into ST-2. Determine weight of 6% HNO_3 added.	5. Initial can Wt. _____ Final can Wt. _____ WT. HNO_3 added _____ Overflow checked by _____
6. Close valve in PR-3 to ST-2 overflow line.	
7. Drop solution from PR-3 to S-1. Allow solution to drain for 15 minutes.	7. Time start _____ Time end _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

XIII - CONCENTRATION

1.	After product solution is in S-1, CLOSE ALL VALVES.	1. Checked by _____
	a) Start condenser water.	a) Time start _____
	b) Check contents of R-1.	b) R-1 Mar. Rdg. _____ Soln. Wt. _____ R-1 Scale Rdg. _____ Soln. Vol. _____
	c) Turn steam on S-1 jacket, purge trap, and adjust steam pressure to 50 p.s.i.g.	c) Time steam on _____ Steam pressure _____
2.	Concentration of solution in S-1.	
Evaporation	a) First drop of condensate over.	a) Time over _____ Max. S-1 Temp. _____ R-1 Mar. Rdg. _____ Soln. Wt. _____ R-1 Scale Rdg. _____ Soln. Vol. _____
	b) Net condensate volume to be collected in R-1 = _____ L This figure will be furnished by the Senior Supervisor.	
2.	Cool S-1 to 50°C (± 5°C)	
	a) Turn off steam to S-1 jacket.	a) Time off _____
	b) Open by-pass valve around S-1 steam trap.	b) Done by _____
	c) Turn on water to S-1 jacket. Regulate water pressure to 20 p.s.i.g.	c) H_2O on _____ H_2O pressure _____
	d) Turn off water to S-1 jacket.	d) Temperature _____ H_2O off _____
4.	Turn off water to condenser. Close by-pass valve around S-1 steam trap	4. H_2O off _____ By-pass closed _____

[redacted]

[redacted]

Date _____
Run No. _____

XIII - CONCENTRATION (Cont'd)

DECLASSIFIED

5. Transfer product solution from S-1 to SR-1 by performing the following operations in order:

   a) Open S-1 to SR-1 valve wide.

   b) Start SR-1 jet. — b) Time start _____

   c) After 30 minutes, stop SR-1 jet. — c) Time stop _____

   d) Close S-1 to SR-1 valve.

6. Transfer product solution from SR-1 to AT. Drain 30 minutes. Close valve between SR-1 and AT. — 6. Time start _____ Time end _____

7. Take Beckman reading on AT. — 7. Beckman _____

8. After agitating AT, sample in quadruplicate. Label: AT, Run No., Serial No., Date, Time. Deliver 3 samples to Laboratory. Store 1 sample in Vault "B". — 8. Sampled by _____ Serial No. _____ AT Scale Rdg. _____ Vol. in AT _____

9. Record time AT Assay received. — 9. Time received _____

10. Load into tared sample cans as approved by supervisor, following separate log for Sample Can Loading and Final Evaporation. Record numbers of sample cans loaded. — 10. Can No. _____ Can No. _____ Can No. _____ Time complete. _____

11. Transfer distillate in R-1 to F-2. Check that F-2 will hold the charge. — 11. F-2 checked by _____

12. Sample F-2 only when directed by the Senior Supervisor. Label: R-1, Date, Time, Run No., Serial No. — 12. Sampled by _____ Serial No. _____ F-2 Man. Rdg. _____ Wt. of Soln. in F-2 _____

13. If total product in R-1 solution is below specified limit, drop solution to tank sump. — 13. Senior Supervisor Approval _____ Time start _____

14. If total product in R-1 solution is above specified limit, drop solution to R.C. vessel. — 14. Time start _____ R. C. Can No. _____

Initial H.I. Survey _____
Tare Weight _____
Gross Weight _____
Net Weight _____
Final H.I. Survey _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

FIRST AND SECOND CYCLE - WASTE HANDLING

1. Determination of MINIMUM amount of 20% $NaNO_2$ to be added.

   Solution Wt. x .35 = Minimum Weight of 20% $NaNO_2$

   _____ x .35 = _____ Kg of 20% $NaNO_2$

   In addition to the reagent can containing the minimum addition of $NaNO_2$, another can containing about 15 Kg of 20% $NaNO_2$ is required for the final $NaNO_2$ addition.

   1. Man. Rdg. _____
      Soln. Wt. _____

2. With the agitator at 80 RPM and cooling water to jacket at full flow, add the minimum quantity of 20% $NaNO_2$ at a rate such that the temperature is maintained at 22°C (± 3°C).

   2. $NaNO_2$ added _____ Kg
      Time start _____
      Time end _____

3. After the minimum quantity of $NaNO_2$ has been added, turn rotary switch to "PT+" (position 4) and leave in this position for 2 minutes. Then turn to "OFF" (position 3).

   3. Time start _____
      Time end _____

4. Plug instrument in, turn rotary switch to "sensitivity 2" (position 2) and chart speed switch to "FAST". Mark recording chart with Run No. Add final 20% $NaNO_2$ with agitator at 80 RPM. Stop $NaNO_2$ addition when chart pen makes an obvious deflection (3 to 4 small chart divisions). Add successive small portions of $NaNO_2$ until a large deflection indicates end-point. Mark the recorder chart at points where $NaNO_2$ addition was started and stopped. Turn switch to "OFF" position, unplug connection, and stop cooling water.

   4. Time start _____
      Initial Wt. of $NaNO_2$ can _____ Kg
      Final Wt. _____ Kg
      $NaNO_2$ _____ Kg
      Time end _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

XIV - FIRST AND SECOND CYCLE - TREATMENT OF SUPERNATANTS (Cont'd.)

$KMnO_4$ Add'n	5. With agitator at 80 RPM, add 4% $KMnO_4$ until a permanent pink color persists.	5. $KMnO_4$ _____ ml Time end _____
Sample CT-1-R-1.2	6. Sample CT-1 in duplicate. Label: CT-1-R-1.2 and complete sample tag.	6. Sampled by _____ Serial No. _____ Time sampled _____ Soln. Wt. _____ Sampler Rdg. _____ Soln. Vol. _____
Transfer	7. Survey four transfer cans. Record tare weights.	7. Can Nos ____ ____ ____ Tare Wts. ____ ____ ____ H.I. Survey ____ ____ ____
	8. Drop any solution in WR-1 to CT-1.	8. WR-1 Checked _____
	9. Transfer 45 Kg of solution from CT to WR-1. Drop solution into surveyed transfer can. Drop any remaining heel in WR-1 to CT-1.	9. Final Man. Rdg. _____ Final Soln. Wt. _____ Can No. _____ Gross Wt. _____
	10. Transfer 45 Kg of solution in CT-1 to WR-1. Drop solution into surveyed transfer can. Drop any remaining heel in WR-1 to CT-1.	10. Final Man. Rdg. _____ Final Soln. Wt. _____ Can No. _____ Gross Wt. _____
	11. Transfer 45 Kg of solution in CT-1 to WR-1. Drop solution into a surveyed transfer can. Drop any remaining heel in WR-1 to CT-1.	11. Final Man. Rdg. _____ Final Soln. Wt. _____ Can No. _____ Gross Wt. _____

[redacted]

Date _____
Run No. _____

DECLASSIFIED

XIV - FIRST AND SECOND CYCLE TREATMENT OF SUPERNATANTS (Cont'd.)

12. Transfer balance of solution in CT-1 to WR-1 (Not to exceed 50 Kg). Drop into surveyed transfer can. Drop any remaining heel in WR-1 to CT-1.

12. Final Man. Rdg. _____
Final Soln. Wt. _____
Can No. _____
Gross Wt. _____

13. Total weight of solution loaded:

Can No.	_____	_____	_____	_____
Final H.I. Survey	_____	_____	_____	_____
Gross Wt.	_____	_____	_____	_____
Tare Wt.	_____	_____	_____	_____
Net Wt.	_____	_____	_____	_____

Total of Net Wts. _____

[redacted]

[redacted]

Date _____
Run No. _____

DECLASSIFIED

INSPECTION RECORD - MECHANICAL

Status - New _____ Returned _____

Date approved by H. I. for reuse _____ Approved by _____

All OK's should be indicated by date and initials of inspector.

Inspection of Parts:

Can	Number	Clean & No Burrs	Lubricated	Freeness of Assembly & Disassembly
1. Body	_____			
a. Lower threads		_____		
b. Upper threads		_____		
2. Adapter Plug	_____			_____
a. Outer threads		_____	_____	
b. Inner threads		_____	_____	
c. Gasket				
3. Breather plug	_____			_____
a. Threads		_____	_____	
4. Filter Cap	_____			_____
a. Outer threads		_____	_____	
b. Inner threads		_____	_____	
5. Retainer ring	_____	_____	_____	_____
6. Filter assembly				
a. Two SS screens)				
) Assembled by _____				
b. Two pads of Media)				
Carrier	_____			_____
1. Lid	_____			
2. Lugs			_____	
3. Thumb Screws			_____	
4. Drilled base plate				
5. Paint condition				

Complete can OK for taring _____ Date _____
Supervisor

[redacted]

Run No._____ Date_____

PLE CAN LOADING AND FINAL EVAPORATION - DETERMINATION OF TARE WEIGHTS

DECLASSIFIED

Weigh the following items after checking that serial numbers match and filter cap assembly contains filter medium:

	WEIGHT	Checked for HEW	Checked for USA
1. Sample Can			
2. Carrier & Filter Assy. (Outer and inner lids, 3 Thumb Screws, 2 Eye Bolts, 2 Lock Washers, 2 Hex Nuts, Filter cap Assy. without large gasket)			
3. Adapter Plug Assembly with Upper and lower gaskets, 2 GX gaskets but without temporary gasket.			
4. Temporary gasket (for adapter plug)			
5. Two filter cap gaskets plus 3 lead seals.			
6. 1 Filter cap gasket with 2 lead seals			
B. Weigh the following assemblies:			
1. Part load tare (items in A_1 and A_2)			
2. Evaporation tare (items in A_1, A_3, and A_4)			
3. Shipping tare (items in A_1 A_3, and A_5)			

C. Make the following pre-loading checks:

1. Check sum of items A_1 and A_2 against B_1. Sum____Checked by____
2. Check sum of items A_1, A_3 & A_4 " B_2. Sum____Checked by____
3. Check sum of items A_1, A_3, & A_5 " B_3. Sum____Checked by____
4. Notify U. S. Army representative that tare weights are ready to be checked. Notified by_____ Time_____
5. After Army check, place tared temporary gasket in adapter plug assembly. Done by_____
6. Secure supervisor's approval to load can. Approved by_____

Date _____
Run No. _____

DECLASSIFIED

II - SAMPLE CAN LOADING AND FINAL EVAPORATION - FIRST LOADING

A. Solution transfers

1. Fill the weigh tank seal with water. 1. Done by _____

2. Place spare empty sample can in load position. Open WT discharge valve and allow to drain for 10 minutes. Close WT discharge valve. Lower the can. Cover the open end of the discharge line with cup provided. Place spare sample can in support stand. 2. Done by _____

3. With the hook, place the can to be loaded in the loading position.

4. Check freedom of weight tank. Check that weigh tank discharge valve is closed. Zero the weigh tank scale by adding weights. 4. Done by _____

NOTE: The following tables in Item II May contain figures from both Item II (First Loading) and Item III. (Supplementary Loading). The final net weight of solution charged is indicated separately in Items II and III.

5. Record initial WT scale reading at item "7b' below. Without agitation drop calculate weight of solution from AT to WT. Any amount remaining in AT should be dropped to another weighted can as soon as possible.

6. Record Volume Dropped from AT

	ITEM II	ITEM III
(a) Initial At reading	_____	_____
(b) Initial AT volume	_____	_____
(c) Final AT reading	_____	_____
(d) Final AT volume	_____	_____
(e) Total volume dropped from AT	_____	_____

DECLASSIFIED

Date _____
Run No. _____

II - SAMPLE CAN LOADING AND FINAL EVAPORATION - FIRST LOADING (Cont'd.)

A. Solution transfers (cont'd.)

7. Record Weight Received into WT

	Item II	Item III
Weights added to Pan (1)		
(2)		
(3)		
(4)		
(5)		
(6)		
(7)		
(8)		
(9)		
(10)		
(a) Total		

	Item II	Item III
(b) Initial Scale Reading		
(c) Final Scale Reading		
(d) Difference in Scale Readings		
(e) Total weight in WT (a ± d)*		

*Add (d), if Final (c) is larger than Initial (b); subtract (d) if Final (c) is smaller than the Initial (b).

Date _____
Run No. _____

DECLASSIFIED

II - SAMPLE CAN LOADING AND FINAL EVAPORATION - FIRST LOADING (Cont'd.)

A. Solution Transfers (Cont'd.)

8. Drop solution from WT to sample can. Allow system to drain for 30 minutes before recording weight left in WT (use following table).

		Item II	Item III
Weights Remaining on Pan	(1)		
	(2)		
	(3)		
	(4)		
	(5)		
	(6)		
	(7)		
(a) Total			
(b) Initial scale reading (Copy from 7b)			
(c) Final scale reading (after drop)			
(d) Difference in scale readings			
(e) Total weight remaining in WT (a ± d)*			

* Add (d) if Final (c) is larger than Initial (b); subtract (d) if Final is smaller than Initial.

[redacted]

DECLASSIFIED

Date _____
Run No. _____

II - SAMPLE CAN LOADING AND FINAL EVAPORATION - FIRST LOADING (CONT'D.)

A. Solution transfers (cont'd.)

9. Record Weight Loaded to Can

	Item II	Item III
(a) Total weight received in WT (copy from 7e)		
(b) Total weight remaining in WT (copy from 8e)		
(c) Weight loaded to can by diff. (a - b)		
(d) Total weight loaded to can: Item II & III		

10. Lower sample can. — 10. By _____

11. Cover open end of weight tank discharge line with cup provided. Place sample can in holder. — 11. By _____

12. If another loading must be made before evaporation, open hood door and insert temporary plug in sample can. — 12. Temporary plug inserted by _____

13. If can is ready for evaporation, use wrench to install adapter plug assembly in sample can. — 13. Adapter plug installed by _____

14. Survey the sample can. — 14. Remarks _____

15. With supervisor's approval, install filter cap assembly (without gaskets) on can loosely. — 15. Approved by _____

16. With hook, remove can and place into carrier. Bolt carrier lid in place. — 16. By _____

DECLASSIFIED

Date _____
Run No. _____

AMPLE CAN LOADING AND FINAL EVAPORATION - FIRST LOADING (Cont'd.)

B. Determination of Weight Loaded

1. Record initial scale reading on line (b) below. Then, with caution to avoid splashing of contents, place assembly on bench scale and weigh. (If another loading is required before evaporation, assembly consists of can, filter cap assembly without large gasket, temporary plug, carrier, cover, and thumb screws. If can is ready for evaporation, assembly consists of can, filter cap assembly without large gasket, adapter plug assembly with temporary gasket, carrier, cover, and thumb screws.)

Weighing checked by _____

	Wts. on Pan	
	First Weighing	Weighing Prior to loading in Item III
1.	_____	_____
2.	_____	_____
3.	_____	_____
4.	_____	_____
5.	_____	_____
6.	_____	_____
7.	_____	_____
8.	_____	_____
9.	_____	_____
10.	_____	_____

(a) Total Wts. on Pan	_____	(aa) _____
(b) Initial scale reading	_____	(bb) _____
(c) Final scale reading	_____	(cc) _____
(d) Difference in scale reading.	_____	(dd) _____
(e) Total Wt. of Can (a ± d)*	_____	(ee) _____

2. Copy tare weight from item IB1 if temporary plug is in can or from item IB2 is adaptor plug assembly is in can _____
3. Determine net weight of solution charge (IIB1a-IIB2) _____
4. (a) If sample can is ready for final evaporation, transport to Room 6A on rubber-tired dolly, without splashing. Omit Item III.
   (b) If sample can is only partially loaded, place can in Vault B carefully and follow III subsequently.
   (c) If intermediate evaporation is required, obtain pages 231-I-I thru 231-U-I.

*If Final (c) is larger than Initial (b), add (d) otherwise subtract (d).

DECLASSIFIED

Date _____
Run No. _____

III - SAMPLE CAN LOADING AND FINAL EVAPORATION - SUPPLEMENTARY LOADING

A. Weighing before Loading

1. Record Run No. of supplementary material	1. Run No. _____
2. Move the partially filled sample can from Vault "B" to cell with care to avoid splashing.	2. Time _____
3. Reweigh sample can. Record on page "F" Item 1.	3. Done by _____

B. Solution transfers

1. Fill the weigh tank seal with water.	1. Done by _____
2. Place spare empty sample can in load position. Open WT discharge valve and allow to drain for 10 mins. Close WT discharge valve. Lower the can. Cover the open end of the discharge line with cup provided. Place spare sample can in support stand.	2. Done by _____
3. With the hook, place can to be loaded in the loading position. Remove filter cap assembly and discard temporary plug.	
4. Check freedom of weigh tank. Check that weigh tank discharge valve is closed. Zero the weigh tank scale by adding weights.	4. Done by _____
5. Record Initial WT scale reading on page "C" and AT gauge reading and volume on page "B". Without agitation, drop the calculated amount of solution from AT to weigh tank. Any solution remaining in AT should be loaded into another weighed can as soon as possible. Record weight received into WT and final WT scale reading on page "C" and AT gauge reading and volume on page "B".	5. Done by _____
6. Drop the solution from weigh tank to sample can. Allow system to drain 30 minutes before recording weight left in WT (use table on page "D"). Record weight loaded to can on page "E".	
7. Lower sample can and place in can holder.	7. Done by _____
8. Cover open end of weigh tank discharge line with cup provided.	8. Done by _____
9. Use wrench to install adapter plug.	9. Done by _____

Date_____
Run No._____

DECLASSIFIED

MPLE CAN LOADING AND FINAL EVAPORATION - SUPPLEMENTARY LOADING

Solution Transfers (Cont'd)

10. Survey the sample can. — 10. Remarks_____

11. With supervisor's approval, install filter cap assembly (without gasket) on can loosely. — 11. Approved by_____

12. With hook, remove can and place into carrier. Bolt inner and outer lids in place. — 12. Done by_____

C. Determination of Net Load

1. Record initial scale dial reading on line (b) below. With caution to avoid any splashing of the contents of the vessel, place assembly on scale and weigh.

Checked by_____

Weights on Pan	
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	
11.	
12.	

(a) Total	
(b) Initial Scale Reading	
(c) Final Scale Reading	
(d) Difference in scale Reading	
(e) Total wt. of Can (a ± d)*	

2. Copy tare weight from item TB_2 _____

3. Determine net total load in can (III C1o - III C2) _____

4. Determine net supplementary load (III C3 - II B3) _____

5. Transport to Rm. 6A on rubber-tired dolly. Move slowly to avoid splashing. If intermediate evaporation required, obtain pages 231-I-I thru 231-U-I — 5. Time_____

*If Final (c) is larger than initial (b) add (d); otherwise subtract (d)

Date _____
Run No. _____

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VI - FINAL EVAPORATION

A. Preliminary Operations

1.	Open hood, raise cover of G-1 and check that G-1 has been drained.	1. Checked by _____
2.	Check that CR-1 is empty.	2. Checked by _____
3.	Raise connector plug of H-1 to stop.	
4.	After 5 minutes, unlock rack and rotate to load position.	4. Time done _____
5.	Place W-1 on top of C-1.	
6.	With hand yoke, place the loaded sample can in rack, using care to center the can.	
7.	Remove hand yoke and close hood, leaving lid of G-1 raised.	7. Time done _____
8.	Remove filter cap from sample can with large wrench and place in holder.	
9.	Remove final closure plug with small wrench and place in holder. If "Koroseal" gasket sticks to plug, pry it loose with pointed tool before moving plug aside.	
10.	Remove "Koroseal" gasket from plug with pointed tool and place in disposal vessel.	
11.	Swing rack into working position and lock in place with alignment rod.	
12.	Carefully screw down the connector plug until a tight joint is made. (Do not use enough force to shear pin)	12. Time done _____

Date_____
Run No._____

DECLASSIFIED

VI - FINAL EVAPORATION

A. Preliminary Operations (Cont'd)

13. If no intermediate evaporation has been made, make the following calculations to determine the volume of condensate to be collected.

(a) Total solution in can (III C3 or II B3, whichever is larger) _____ g

(b) To remain in can 390 g

(c) Weight to be collected (a-b) _____ g

(d) Volume to be collected

Item (c) (_____) ÷ Sp. Gr.* (_____) = _____ ml

14. If intermediate evaporation has been made, make the following calculations to determine the volume of condensate to be collected:

(a) Total solution in can (V C 4) _____ g

(b) To remain in can 390 g

(c) Weight to be collected (a - b) _____ g

(d) Volume to be collected _____

Item (c) (_____) ÷ Sp. Gr.* (_____) = _____ ml

* Specific gravity measured after collecting 75% of the volume calculated on a basis of 1.38 Sp. Gr.

Date _____
Run No. _____

DECLASSIFIED

VI. FINAL EVAPORATION (Cont'd)

B. Evaporation Operations

1. With G-1 drain valve closed, fill G-1 with water to top of rack.	1. Time filled _____
2. Drain G-1 to sewer and close drain valve.	2. Time drained _____
3. Check that valve CR 1 to CR-2 is closed.	3. Checked by _____
4. Open valve AF-1 to H-1.	
5. Open valve CR-1 to VB-1.	
6. Turn on cooling water of C-2 and C-3.	
7. Turn on steam to jets #2 and #3.	
8. Adjust air flow through sample can to 8 ($\pm$ 0.5) C.F.H. by varying water level in VB-1. (Do not change rate in adjacent units from 8 ($\pm$ 0.5) C.F.H.)	8. Air Flow _____ C.F.H. Adjusted by _____
9. Turn on water to C-1.	
10. With G-1 drain valve closed, add hot glycerine from G-3 to a level about 3/4 inch below top of rack. (Do not overflow G-1.)	
11. Close G-1 lid.	
12. Open valve admitting 25 p.s.i.g. steam to G-1 jacket.	
13.. Bring G-1 to 125°C.	13. Time 125°C _____

Date _____
Run No. _____

DECLASSIFIED

VI - FINAL EVAPORATION (Cont'd.)

B. Evaporation Operations (Cont'd.)

14. Continue evaporation at 8.0 C.F.H. and 125° bath temperature, Record specific gravity, bath temperature, volume of condensate collected, and air flow rate every 15 minutes. (Spargo CR-1 for one minute before reading Sp. Gr.)

	Time	Temp.	Sp.Gr.	C.F.H.	Graduate Reading	Cond. Vol. (ml)	Recorded By
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							
14.							
15.							
16.							
17.							
18.							
19.							
20.							

TOTAL _____

Date_____
Run No._____

DECLASSIFIED

VI - FINAL EVAPORATION (Cont'd)

B. Evaporation Operations (Cont'd)

15. When volume in CR-1 is 25 ml. less than that calculated in step A-13 or 14, close G-1 jacket steam valve. — 15. Steam off_____

16. Open valve G-1 to G-2. — 16. Valve open _____

17. Turn on jet #5 and open G-1 lid. — 17. Jet on_____

18. When G-1 is empty, turn off jet #5, close valve in line from G-1 to G-2, and drop glycerine to G-3. — 18. Jet off_____
Valve closed_____
Glycerine dropped_____

19. Fill G-1 with water and leave sufficient water running to overflow a small stream into sewer. — 19. Time water on_____

20. After cooling can for 1 hour, shut off water and drain G-1 to sewer. — 20. Time water off_____
G-1 Temp._____

21. Close valve AF-1 to H-1.

22 Sparge CR-1 for one minute and record final volume and Sp.Gr. on page 231-L.

23. Turn off water to C-1.

24. Close valve CR-1 to VB-1.

25. If other drying units are not in use, 25. Done by_____
(a) Turn off steam to jets #2 and #3
(b) Turn off cooling water of C-2 and C-3.

26. If other units are in use, readjust air flow through them to 8 (± 0.5) C.F.H. by altering water level in VB-1. — 26. Air Flow_____C.F.H.
Adjusted by_____

Date_____
Run No._____

DECLASSIFIED

NAL EVAPORATION (Cont'd)

Supplementary Operations

1. Raise connector plug to stop.	1. Time done_____
2. After 5 minutes, unlock rack and rotate to load position.	2. Time done_____
3. Remove final closure plug from holder with small wrench and screw plug tightly into adapter plug in top of sample can. Tighten adapter plug.	
4. Survey the sample can.	4. Remarks_____
5. Insert two filter cap gaskets.	
6. Remove filter cap from holder with large wrench and mount it loosely on top of sample can.	
7. Dry and survey the sample can.	7. Remarks_____
8. With supervisor's approval move sample can to carrier and tighten thumb screws and hex nuts.	8. approved by_____
9. Return can to cell 5 for weighing.	
10. Open valve CR-1 to CR-2.	
11. Turn on water to jet #1 and draw condensate from CR-1 to CR-2 Turn off water when CR-1 is empty.	
12. If this unit is to be used again immediately, proceed to item 16. If not, proceed to item 13.	
13. Place W-1 in sample can rack.	
14. Swing rack into working position. and lock in place with alignment rod.	
15. Carefully screw down the connector plug to make a good joint with W-1 (Do not use enough force to shear pin)	15. Time done_____

[redacted]

Date_____
Run No._____

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VAL EVAPORATION (Cont'd)

Supplementary Operations (Cont'd)

16. If volume in CR-2 is less than 7 liters, proceed to item VI D.

17. If volume in CR-2 is 7 liters or more, take composite sample:

17. CR-2 volume_____
Sampled by_____
Time sampled_____
Serial No._____

(a) Check that all valves leading to CR-2 are closed.

(a) Valves checked_____

(b) Turn on water to jet #1.

(c) Open CR-2 sparger valve.

(c) Time open_____

(d) After 5 minutes, open air lift valve.

(d) Time open_____

(e) After 5 minutes longer, turn off water to jet #1.

(e) Time off_____

(f) Close CR-2 sparger valve.

(g) Close air lift valve.

(h) Take sample from cup by pipette. (Close valve after sampling.)

18. If assay of CR-2 composite is below specified limit, discharge to sewer.

18. Authorized by_____
Time done_____

19. If assay of CR-2 composite is above specified limit:

(a) Set CR-3, with gate valve open, in place below CR-2.

(a) Done by_____

(b) Open drain valves CR-2 to CR-3.

(b) Time CR-2 empty_____

(c) Twenty minutes after CR-2 is empty, close valves CR-2 to CR-3.

(c) Time closed_____

(d) Lower CR-3 and mount drip cup on CR-2 drain line.

(e) Close CR-3 gate valve.

(e) Time closed_____

(f) Recycle solution from CR-3 as directed.

[redacted]

LEGAL NOTICE
FTW-F0475-C

Date_____
Run No._____

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INAL EVAPORATION (Cont'd)

D. Determination of Weight Shipped

1. After recording initial scale reading on line (b) below, weigh complete assembly of vessel and carrier including seals:

Weights on Pan	1)_____
	2)_____
	3)_____
	4)_____
	5)_____
	6)_____
	7)_____
	8)_____
	9)_____
	10)_____
a)Total	_____
b)Initial scale	_____
c)Final scale	_____
d)Difference in scale	_____ Weighed by_____
e)Total can wt. (a ± d)*	_____ Checked for FEW by_____ Checked for USA by_____

2. Copy tare weight from item III3 _____
3. Determine net weight of material shipped (VI D1-VI D2) _____ **
4. Determine Weight of Solution Evaporated
   a) Gross Wt. Soln. charged (III3 or IIIC3 or VC4, whichever was obtained last) _____
   b) Net Wt. of matl. to be shipped(VI-D-3)_____
   c) Wt. of liquid evaporated (a-b) _____

*If Final (c) is larger than Initial (b), add (d); otherwise subtract (d).

**If net weight is 420 gm or less, can is satisfactory for shipment. If weight is greater than 420 gm return to final evaporation equipment and remove condensate to reduce net weight to 390 gm.

Date_____
Run No._____

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INAL EVAPORATION (Cont'd)

Preparation for Shipment

1. Open hood door. With hook place sample can in holding jig. Close hood door.

2. Remove filter cap assembly.

3. Obtain H.I. approval for inside of sample can.

3. Remarks_____
Max. Smear_____
Location_____

4. Insert filter cap assembly and tighten up.

5. Obtain H.I. approval for carrier and outside of sample can.

5. Remarks_____
Max. Smear_____
Location_____

6. Obtain mr. reading.

6. mr/hr at surface_____
Date obtained_____

7. Open hood door and with hook, place complete sample can assembly in carrier. Tighten thumb screws and hex nuts on carrier cover and permanently secure lead seals in place with U. S. sealer.

8. Transport to Vault "B"

9. Record date shipped_____

[redacted]

Date_____
Run No._____

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OF WEIGHTS

I - Mtl. Dropped from AT

(1) Total volume from AT (II A 6 e)_____ x Sp. G. = _____ (Item II)

(2) " " " " (II A 6 e)_____ x _____ = _____ (Item III)

(3) Total weight from AT

II - Mtl. to WT

(1) Total weight into WT (IIA 9 a)_____ (Item II)

(2) Total weight into WT (IIA 9 a)_____ (Item III)

(3) Total weight received into WT

III - Mtl. to Can (From WT Scale)

(1) Total weight loaded to can (IIA 9 c)_____ (Item II)

(2) Total weight loaded to can (IIA 9 c)_____ (Item III)

(3) Total weight into can (IIA 9 d)

IV - Overall weight into Can (From Bench Scale)

Total weight into can (IIB3 or IIIC3, whichever is larger

V - Weight of Mtl. Shipped

Net weight of Mtl. shipped (VI D 3)

VI - Weight of Mtl Evaporated (by difference)

Total weight of Soln. evaporated (VI D4 c)

VII - Weight of Liq. Evap. (by measure)

Total volume of soln. evap. (VI B 14)_____ x Sp. G. _____

[redacted]

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SECTION C - SEPARATIONS

CHAPTER IX - WASTE DISPOSAL

Process wastes from the 200 Area present a disposal problem because of their acidity and radioactivity. All wastes are neutralized before disposal. The dangerously active wastes are stored in buried tanks, while the less active material is released underground through reverse flow wells.

The disposal of specific process wastes is discussed in this chapter.

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CHAPTER IX - WASTE DISPOSAL

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GENERAL CONSIDERATIONS

The actual disposal of a given Separations plant waste depends on its intrinsic value, acidity, associated fission activity, and product content. The most radioactive wastes must be stored in underground tanks to protect personnel from radiation and to avoid contamination of the river or underground water supply. Because of the extreme toxicity of product, it is necessary that product-containing wastes not be discarded above ground. This avoids the possible formation of product-containing dust which might result if such wastes were discharged into open ditches after monitoring.

The chief types of wastes and their disposal are given in the paragraphs following.

Canyon (221) Building

Four types of active process waste are formed in the 221 Building:

1) The alkaline coating removal waste, containing small but undetermined amounts of activity

2) The metal waste from extraction, containing all of the uranium, about 90% of the original fission activity, and 1/2 to 1% of the product

3) The by-product cake solution and waste solution from the first decontamination cycle, containing about 10% of the original fission activity and 2% of the product

4) Second cycle waste, containing less than 0.1% of the fission activity, and about 1% of the product

The Canyon Building process wastes are stored in a series of buried tanks known as 241 Building. Cell sewage can be sent to the buried tanks, to settling tanks and dry well, or buried sump as advisable.

Concentration (224) Building

The Concentration Building process wastes comprise solutions of the bismuth phosphate and lanthanum fluoride by-product cakes, the product precipitation waste, and metathesis wastes. The total fission activity is less than 0.001% of that present in the starting metal. About 4% of the product is contained in these wastes. It is considered safe to send these wastes into the ground. The waste flows through a settling tank where the bulk of the fission and product activity settles out, and thence to a reverse flow or dry well, or a buried sump. Cell drainage goes to the settling tank.

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Isolation (231) Building

Active wastes from laboratory sinks, washings from the process cells, and water from water aspirators in the Isolation Building contain very small amounts of product.

The wastes are collected in a 3900-gallon stainless steel sump tank (231-W-151), and after neutralization with sodium hydroxide are jetted into a reverse flow well or buried sump.

Miscellaneous Wastes

Active liquid collects at the base of the 291 Building stack. It is formed from condensate from the dissolver off-gas vent jets. This drainage flows to buried tanks.

The Control Laboratory (222) discards a small amount of active waste from analytical samples and equipment decontamination. This waste is run down a special stainless steel sink connected to a dry well or buried sump.

Cooling water from the jackets of process vessels in Canyon and Concentration Buildings is collected in a retention basin. After monitoring to determine that no activity is present, the basin is emptied into an open ditch. Drainage from uncontaminated areas in 271, 221, 222 and 224 Buildings goes directly to this ditch. Cooling water from the Metal Storage (212) Buildings has an activity so low as to be innocuous and goes directly to an open ditch.

Sanitary wastes are sent to a septic tank and tile field.

241 BUILDING

The waste disposal area is designated as 241 Building. The layout of these areas and their connections to the process buildings is shown in Figure 1 for T and U areas and Figure 2 for B area. Each of the process areas, T, U, and B has its own waste disposal facilities. In addition, 241 C, some 4,500 feet east of 221-B, is a complete group of tanks and diversion boxes like those shown on the drawings of the other areas, but lacking the retention basin, settling tank and dry well. This can be connected up for 200-B Area waste disposal through Diversion Box 241-B-154 in such manner as may appear desirable in the future. At present no waste lines are provided from this diversion box to the 241 C Area.

Geology

Wastes from 224, 222 and 231 Buildings are disposed of through dry wells. Furthermore, it may prove desirable at some future time to stretch out the

[redacted]

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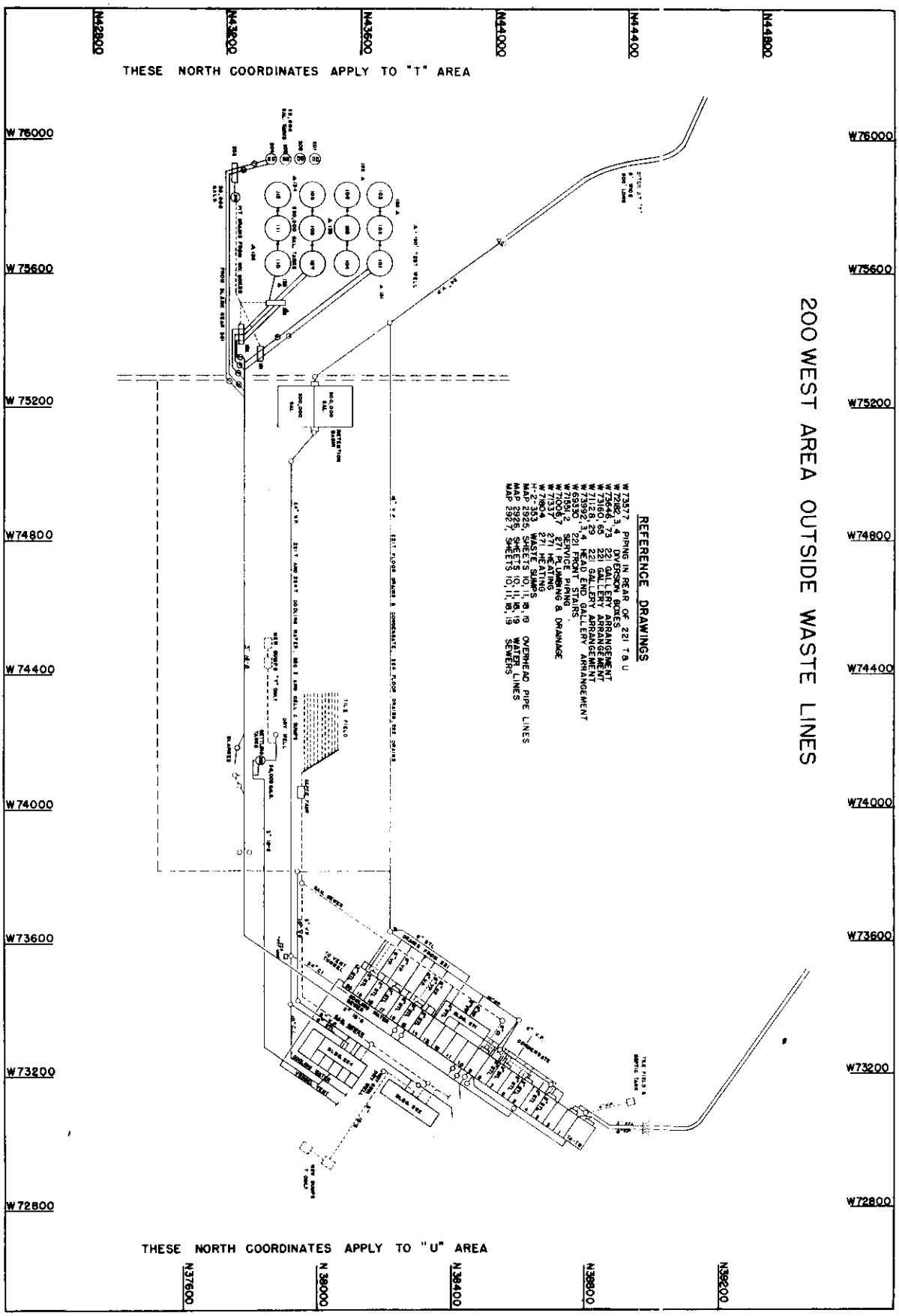
FIGURE 1



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HW-1047-C

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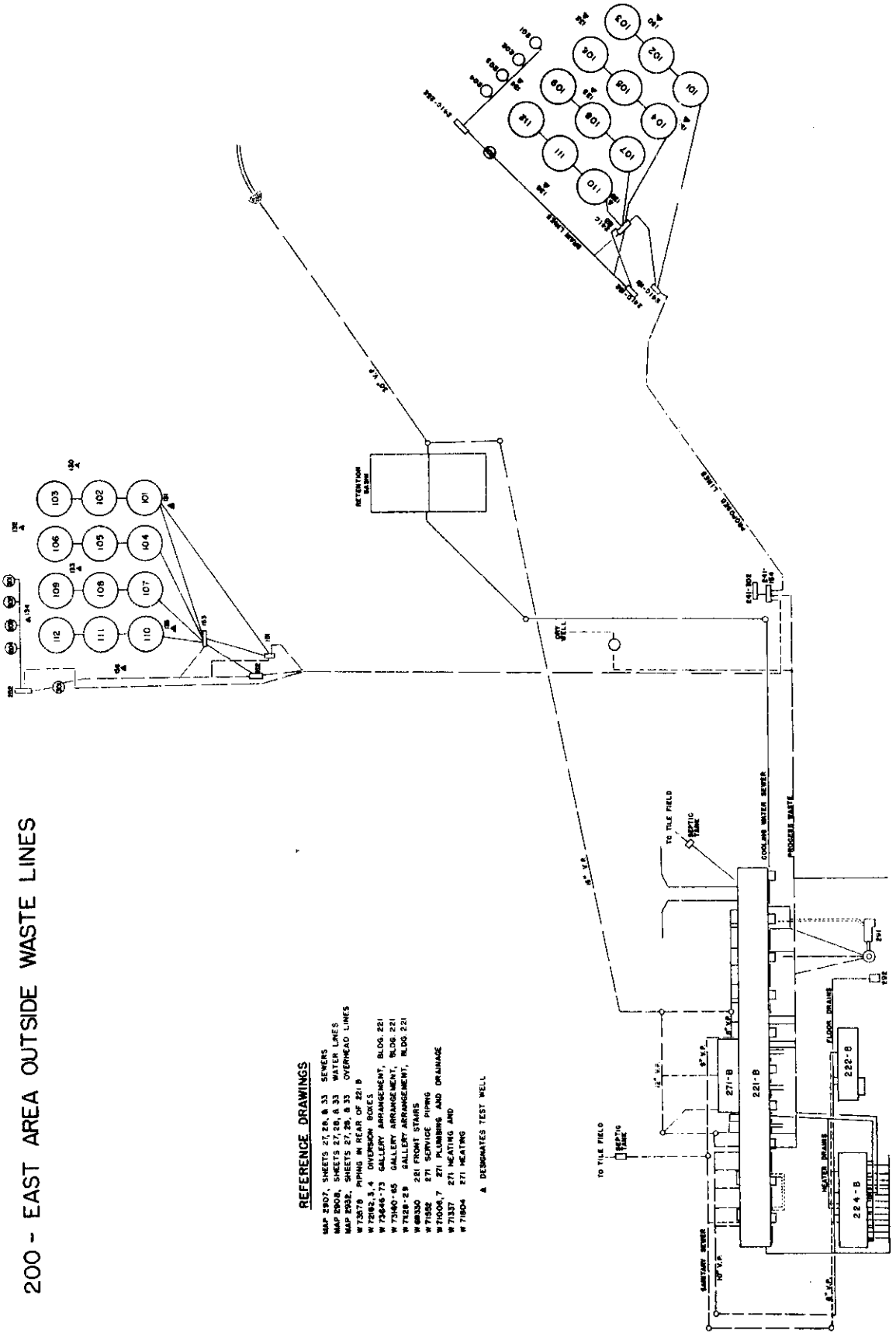
FIGURE 2

200 - EAST AREA OUTSIDE WASTE LINES



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capacity of the waste storage tank system by emptying some of the less active wastes into additional dry wells, or buried sumps. The safety of this procedure is dependent on the geology of the area in which the well is drilled or the sump buried.

Most of the H.E.W. area consists of basalt on which has been deposited a loosely cemented sandy clay known as the Ringold formation. The basalt projects through the Ringold at some points, such as Gable Mountain and Gable Buttes. Both these formations are, for practical purposes, impermeable to water and unsuited for waste disposal.

In times past the Columbia River has cut deep channels in the Ringold formation. These were subsequently filled with coarse glacial gravels and are well suited for underground waste disposal. Their water-bearing capacity is high and enormous volumes of dilution water are periodically introduced from flash floods and during periods of high river level. Waste introduced into this system would be greatly diluted, decontaminated by percolating through the soil, and would eventually get down to the Yakima River, near Richland.

Most of the 200 Area is believed to be above such a channel and is therefore suited for drilling of the reverse flow wells, or construction of the buried sumps.

However, the Ringold formation is essentially at the surface a short distance west and south of the 200-W Area. Some place east of Cold Creek Valley, there is believed to be a Ringold bluff, the bank of one of the old extinct river channels. Where this formation occurs above the water table, the course of the wastes is unpredictable. They may collect in pockets on the Ringold surface, be forced to the surface farther south where the land is lower, or picked up by a single flash flood and carried down to the Yakima-Richland area immediately.

75-Foot Diameter Storage Tanks

The Canyon Building process wastes are stored in twelve tanks numbered X-101 to X-112, inclusive. These are 75 feet in diameter, and are of reinforced concrete construction with a steel liner on the bottom and sides. The tanks rest on waterproof pads and are waterproofed on the sides and top with tar and gunnite. Capacity to the overflow outlet is 530,000 gallons, with a liquid depth of 16 feet.

These tanks are arranged in groups of three in cascade, with fresh waste always going to the first tank of a series (X-101, X-104, X-107, X-110).

Tanks X-101, 102 and 103 are each equipped with an air cooled reflux condenser. Tanks X-104, 105 and 106 are provided with a condenser foundation and duct blanked off at grade. The other six tanks have no provision for a condenser.

In addition to the overflow piping, each tank has four 3-inch stainless

steel inlet pipes. At the first tank in each cascade series of three, two or three of these inlets are connected to a diversion box by stainless steel lines. All others are blanked off close to the respective tanks.

Each tank has eight risers up to grade, in sets of four on the opposite sides of the tank. These are 4-inch and 12-inch steel pipes terminating a few inches inside the roof of the tank. One of the 4-inch pipes carries an indicating thermometer showing the temperature of the tank contents. Two dip tubes in one of the 12-inch sleeves are used for liquid level and gravity measurements. The higher tube ends 5 feet above the low tube. The other risers are unused, but are available for additional instrumentation, sampling, or pumping out.

Shielding from radiation is provided by approximately 9 feet of fill over the tanks. The condenser ducts are formed in two right-angled bends to retard escape of radiation through these openings. The risers are capped with a flange carrying an 18-inch long steel or steel and concrete shielding plug. To allow venting of the tanks, one of these plugs has been removed from each tank except the three having condensers, and a T-shaped vent pipe attached.

These 75-foot diameter tanks are now connected to receive waste as follows:

X-101 receives metal waste from Tank 9-1. Tanks X-102 and 103 receive from Tank X-101 by cascade. Alternate connections from Tank 10-1 are available for emergency use.

X-107 receives from Section 15-R. Tanks X-108 and 109 receive from Tank X-107 by cascade. Under current operating practice, Section 15-R handles first cycle and coating wastes. Tank 5-9 is also connected to Tank X-107. This tank also receives the 291 Stack drainage.

X-110 receives from Section 15-L. Tanks X-111 and 112 receive from Tank X-110 by cascade. Under current operating practice, Section 15-L handles second cycle wastes.

X-104, X-105, and X-106 is a spare series of tanks, and is connected to Diversion Box 241-153. Jumpers are available permitting hook-up to Tanks 9-1, 10-1, or Section 15.

The temperature and weight factor readings are recorded daily on all tanks containing any waste. Weight factors are measured with portable equipment.

Currently, the volume of active waste delivered to the tanks per run (1.5 metric tons) is 5700 gallons for metal waste, 4700 gallons for coating and first cycle and 3600 gallons for second cycle wastes. Each group of three tanks, therefore, is filling at roughly the same rate. This will eventually lead to a situation such that the area can no longer be conveniently used although tanks S-104, 105, and 106 will still be empty. At this stage, it seems probable that the spares could be used for metal waste and

that additional decontamination waste capacity can be made available by emptying some of the less active wastes into the ground.

It is not known whether any great amount of self-heating of the metal waste tanks will occur. Long-term stability data are also lacking. If appreciable self-heating takes place, then the bulk of the uranium and fission activity will be quickly precipitated, as the carbonate complex is stable for only a day or two at 75°C. It should be possible to determine the activity distribution in the metal waste tanks by lowering a specially shielded ion chamber into a water tight metal sleeve, extending to the bottom of the tank, and noting the vertical distribution of activity. If precipitation occurs, the bulk of the activity will be concentrated on the precipitate. Precipitation in the first metal waste tank may lead to the accumulation of excessive activity in one tank, with consequent overheating. If this is found to occur, it may be desirable to install additional piping whereby metal waste tanks could be filled individually, rather than by cascade.

No equipment is available for sampling or otherwise investigating conditions in the tanks, except liquid level and pH. At monthly intervals, a pH determination is made, using an electrode which is lowered into the tank and discarded after readings have been made. Since the lines of the waste tanks are steel, it is desirable that the pH should be not less than 7 for all wastes.

20-Foot Diameter Storage Tanks

In each 241 Area there is a row of four smaller buried tanks, X-201 to X-204, inclusive, of the same type of construction as the large tanks. These are 20 feet in diameter with a capacity of 55,000 gallons at a liquid depth of 24 feet.

The 20-foot tanks are not arranged in cascade and have no overflow outlets. Each tank is connected individually by two stainless steel lines to Diversion Box 241-252 and is further provided with two spare inlets. There are two condensers on each tank, duplicates of those on Tanks X-101, 102 and 103. The number and arrangement of risers, instrumentation and shielding of the small tanks are identical with the 75-foot tanks.

Originally, serious consideration was given to decontaminating the metal waste before disposal, to facilitate ultimate recovery of the uranium. One or two scavenger precipitates were to be made and separated out along with alkali-insoluble material. These highly active by-product cakes were to be slurried out of Centrifuges 9-2 or 10-2 and sent to the 20-foot storage tanks via Tanks 9-4 or 10-4. These tanks have never been used for the purpose for which they were designed.

Tank 10-4 is connected to Diversion Box 241-252 and can be connected to the inlet lines by available jumpers. A line from Tank 10-4 to Tank X-201

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is completely installed but is not used. The original line from Tank 9-4 has been broken near 241-361 Settling Tank. It is planned to use the lower portion of this line to slurry the contents of 361 Tank down to Tank X-201 when the former shows signs of filling up with solids.

Diversion Boxes

At each 241 Building are four diversion boxes through which the seven process waste lines into the tank area can be connected up to the tanks in various ways. Active connections through these boxes are indicated in Figures 1 and 2. A more detailed diagram indicating also the spare connections available is shown in Figure 3. A short distance east of 221-B Building is an extra box, 241-B-154, which permits waste to be sent either to 241-B or C. All process waste lines from 221-B go through this box.

The boxes are of reinforced concrete. Outlets are arranged in a row near the bottom. Inlets are in a second row above outlets, on the same side of the box. The pipes into the box terminate in flange and kick plate assemblies like those used for canyon and trench piping. Connections from inlets to outlets are made with prefabricated jumpers fitted with automatic connectors. A variety of jumpers is provided, so that connections can be changed or replaced remotely after the boxes become contaminated. All piping is stainless steel. The boxes are shielded by fill along the sides and by concrete slab covers.

20-Foot Diameter Catch Tanks, (241-301)

At each 241 Building there is a buried 36,000 gallon unlined reinforced concrete tank piped up to receive drainage from the four diversion boxes. These tanks are buried about 6 feet below grade. Risers and instrumentation are similar to those on the larger buried tanks. An extra 17,000 gallon catch tank (241-B-302) identical in construction with the above, is provided for collection of the drainage from 241-B-154 Diversion Box.

Temperature and weight factors on these tanks are read daily. An abrupt rise in weight factor reading would indicate a leak in one of the diversion box connections.

Retention Basin and Ditch

The retention basin consists of two square concrete pools holding 500,000 gallons each. Inlet and outlet connections permit one half of the basin to be filled while the other is draining. At full operation, one-half of the basin fills per shift. Flow is then diverted to the other half and a sample is taken for gross activity analysis. When released for disposal, the full half is then drained to the ditch.

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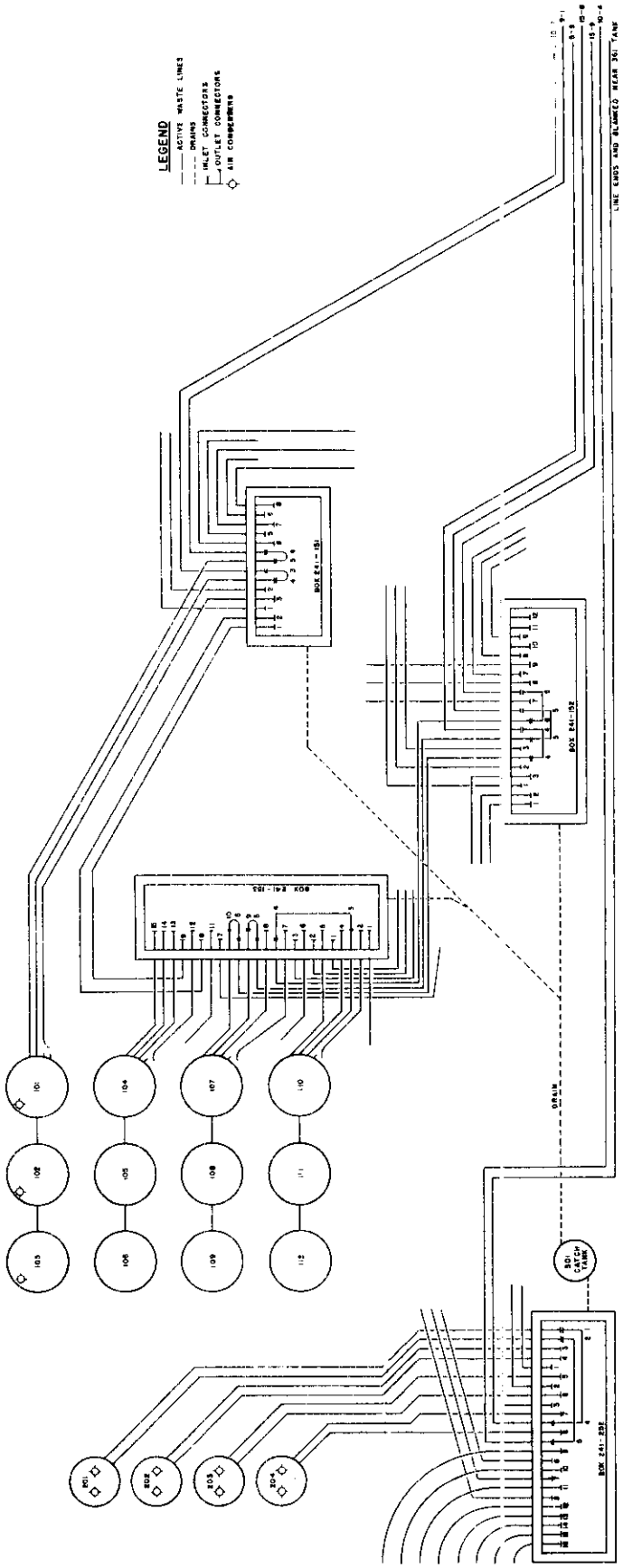
FIGURE 3

DIAGRAM OF UNDERGROUND LINES TO DIVERSION BOXES, BUILDING 241, AREAS T, U, B, & C.



The basin receives process cooling water through a 24-inch sewer running along the side of 221 Building with connection to 224 Building. The sewage collected in the sumps of Section 5, 221 Building and Cell C, 224 Building can also be jetted into this main if desired.

Outlet from the basin is through a 24-inch sewer. This connects with an 18-inch sewer carrying floor drains and other innocuous wastes, except sanitary waste, from 271, 221, 224, and 292 Buildings. The combined sewage flows to an open ditch.

The ditches, which are 800 or more feet long, terminate in the vicinity of the low point of their respective areas. The ground at these points does not absorb water very rapidly and it appears likely that swamps will eventually form near the end of each ditch.

Settling Tank, Dry Wells and Buried Sumps (241-361)

Wastes from 224 Building flow to a buried 20-foot diameter settling tank. This is an unlined concrete tank with instrumentation similar to the other 241 Building tanks. The tanks are vented through a 6-inch riser from the manhole on top of the tank. Capacity at overflow is 36,000 gallons with a liquid depth of 15 feet.

Inlets from 224 Building and from Section 5 of 221 Building are provided. Contents of the tank overflow to a dry well. The inlet is 4 inches below the outlet to reduce turbulence when waste is being jetted to the tank. A baffle across the tank near the outlet also helps to produce a clear effluent. The inlet is 3-inch and the outlet 2-inch diameter stainless steel pipe.

The overflow from each 361 Tank goes to a 6-inch diameter dry well near by. In T Area water was struck at 287 feet and a second well, ("A"), was therefore drilled to 206 feet. This is the one in use. The U and B wells are 302 feet in depth. The lower portion of the casing is perforated to facilitate discharge of waste into the soil.

Each well has a pressure gauge with line running to 50 feet from the bottom. Air from a portable source of supply can be used to measure hydrostatic head in the well. At present, the wells empty freely and the water level does not rise above the bottom of the dip tube after pumping water into it at the rate of 100 gal./min. for 20 minutes.

Recently it has been observed that more of the dry or reverse flow wells have become partially filled with sand which has apparently flowed in through perforations in the lower end of the casings. For this reason buried sumps have been constructed in all locations where reverse flow wells were provided. These buried sumps consist of 12 foot by 12 foot by 4 foot high reinforced wooden cages with open bottom buried in gravel 18 to 19 feet below the grade surface. These sumps are connected to the 241-361 Settling Tanks by 3-inch stainless steel pipe. Two such sumps are connected in series with a 1-foot high inverted trap being provided in the line connecting the two sumps.

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Liquid level measurements are made on the settling tank before jetting as a check against leakage or plugging of the overflow, dry well, or buried sumps.

Effluent to the well is sampled occasionally, inspected for clarity and analyzed for product and by-product activity. So long as the tank effectively retains solids, little activity should escape. At present, the effluent is decontaminated by factors of 250 to 1,000 and is almost perfectly clear. When solids and activity begin to leak through the contents of the tank will be flushed to X-204. On the basis of experiments indicating that Concentration Building wastes precipitate 3% by volume of solids, the 361 Tank is expected to fill up in about 250 runs.

The overflow rate of 361 Tank is limited by the 2-inch outlet, rather than by the capacity of the well. Liquid level in the well does not appear to reach the dip tube, 50 feet from the bottom of the well, at any time. There is, so far, no indication that the well will plug up with continued use. Liquid level in the tank drops back to the outlet level within about one-half hour after jetting a normal 2500 gallon portion of waste.

The risers from 361 Tank have their lower ends only 1 foot above the overflow level. Under these conditions, 2400 gallons of additional liquid in the tank will seal the vents. To avoid putting undue pressure on the manhole cover, it is advisable to jet no more than 3,000 gallons to the tank at one time and to wait 1 hour between jettings. (In order to correct this situation an additional vent has been added to this tank in a pipe attached to the lid).

Dry Wells and Buried Sumps, 222-110

Near the right front corner of 222 Building is a 75-foot dry well constructed of 3-inch stainless steel piping. The bottom 25 feet of the casing is perforated. The well is connected to the stainless steel "hot" sink in the laboratory. Two buried sumps in series are also provided which may be used in place of the dry wells.

Test Dry Wells

Seven 6-inch wells are placed at selected locations among the waste storage tanks. All are 150 feet deep. These wells receive no waste. Instruments are lowered into them to detect activity arising from leaks in the storage tanks.

Dry Well and Buried Sumps, 231-W-150

Near the 231 Building buried waste tank is a 6-inch diameter dry well into which the contents of the tank can be jetted. The well is 150 feet deep, with perforations in the lower 32 feet. Two buried sumps in series are also provided,

due to the fact that this dry well has partially filled with sand. These sumps are used at present in place of the dry well.

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CANYON (221) BUILDING WASTE DISPOSAL EQUIPMENT

Process waste lines in the Canyon Building are diagrammed in Figure 4. The several types of waste are collected and treated in Section 5, 9, 10, and 15.

Sewers

Two 24-inch sewers serve the canyon proper. The cell sewer runs underneath the building near the pipe trench. This is constructed in two sections each pitched to drain into the deep sump in Section 5-R. At the rear of the building is the cooling water sewer with 8-inch connections into the left side of each cell wall. This receives cooling water from all process vessels.

Section 5, Cell Sewage

The bottom Section 5-R is 47 feet 9 inches below deck level as compared to 28 feet in the standard section. This permits the two sections of the cell sewer to come into the sides of Section 5-R and drain into Tank 5-7, a 17,000 gallon rectangular open stainless steel tank illustrated in Figure 5. Drains through the back wall of Section 5-R from the pipe trench and ventilating tunnel also empty into Tank 5-7. (See Figure 6 for a diagram of the cell piping and gauge board for Section 5).

Sewage in Section 5-R itself collects in a trough and sump tank (5-8) on the floor from which a jet connection into Tank 5-7 is provided.

Section 5-L contains two 8-foot diameter by 14-foot high tanks, each with a capacity of 5000 gallons. These are unagitated jacketed tanks. There are no steam or water connections to the Tanks 5-6 and 5-9 jackets. These tanks can be used for sampling and treatment of cell sewage jetted up from Tank 5-7.

Scale tanks 5-6E and 5-9E of 75 gallons capacity, can be used for caustic additions to either of the tanks in 5-L. The 400 gallon scale tank, 5-7A, has no chemical header connections.

The Section 5 gauge board has a liquid level and density recorder for each tank, an activity recorder connected to each tank and a temperature recorder connected to everything but the sump, 5-8.

Air and steam spargers are provided for Tanks 5-6, 5-7 and 5-9. These three tanks, all have sampler connections to the deck. Tank 5-8 has no sampler connection.

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FIGURE 4

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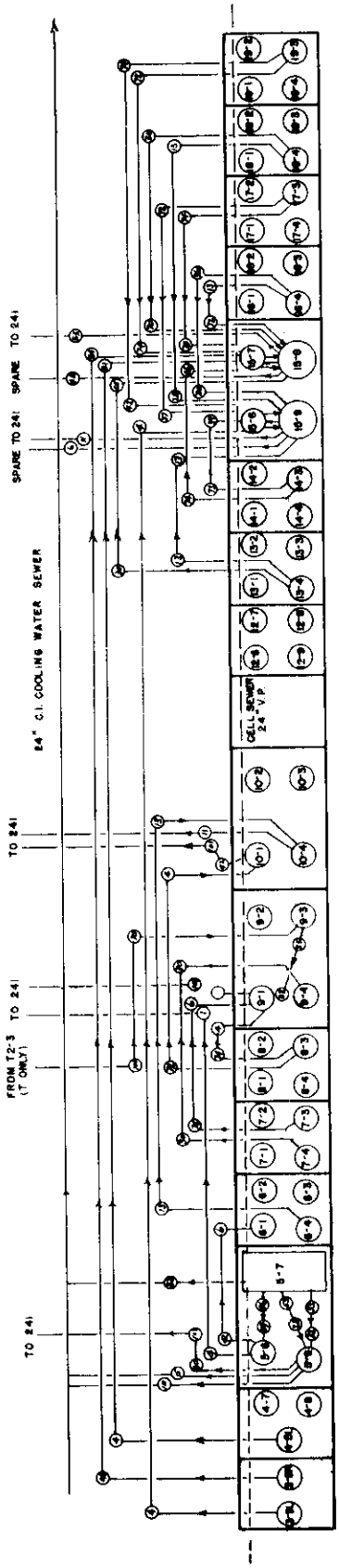
CANYON (221) BUILDING WASTE LINES. CELL AND TRENCH CONNECTIONS.



WASTE TANK CAPACITIES

5-6, 5-9 5,000 GAL.
5-7 17,000 GAL.
9-1, 10-1 5,000 GAL.
8-4, 10-4 775 GAL.
8-3, 10-3 4,000 GAL.
15-6, 15-7 3380 GAL.
[illegible]

REFERENCE DRAWINGS

W 73338-41 TRENCH PIPING
W 74066 ALTERNATE TRENCH PIPING
W 73901-11 CELL ARRANGEMENT
W 74867 ALTERNATE CELL ARRANGEMENT
W 70101-4 PIPING THROUGH CONCRETE
W [illegible] PIPING THROUGH CONCRETE
W 70144-5 PIPING THROUGH CONCRETE
W 73086-93 PIPING THROUGH CONCRETE, HEAD END
W 74174-6 CELL ARRANGEMENT, HEAD END

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FIGURE 5

SECTION 5 - DEEP CELL OF CANYON BUILDING

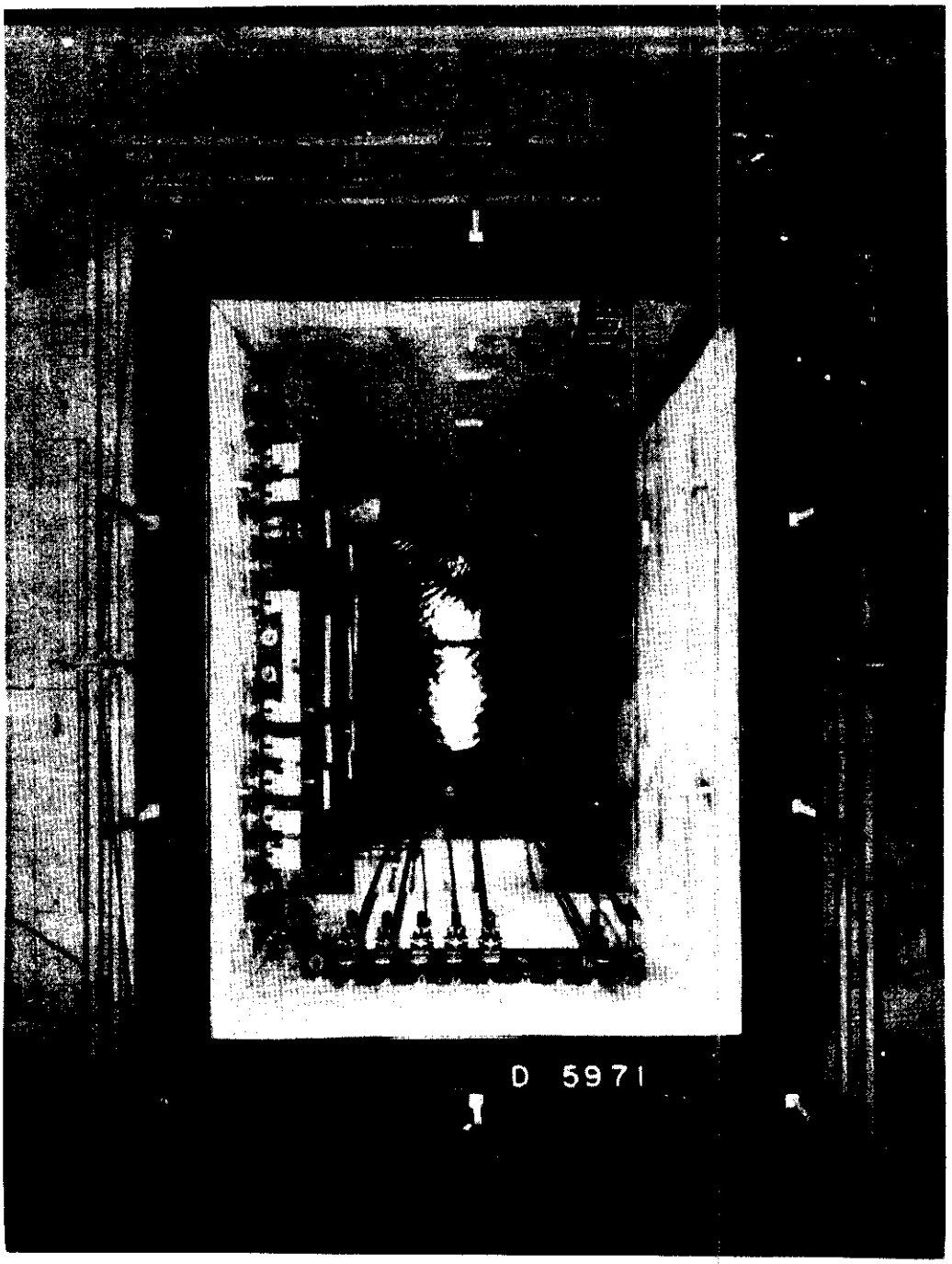

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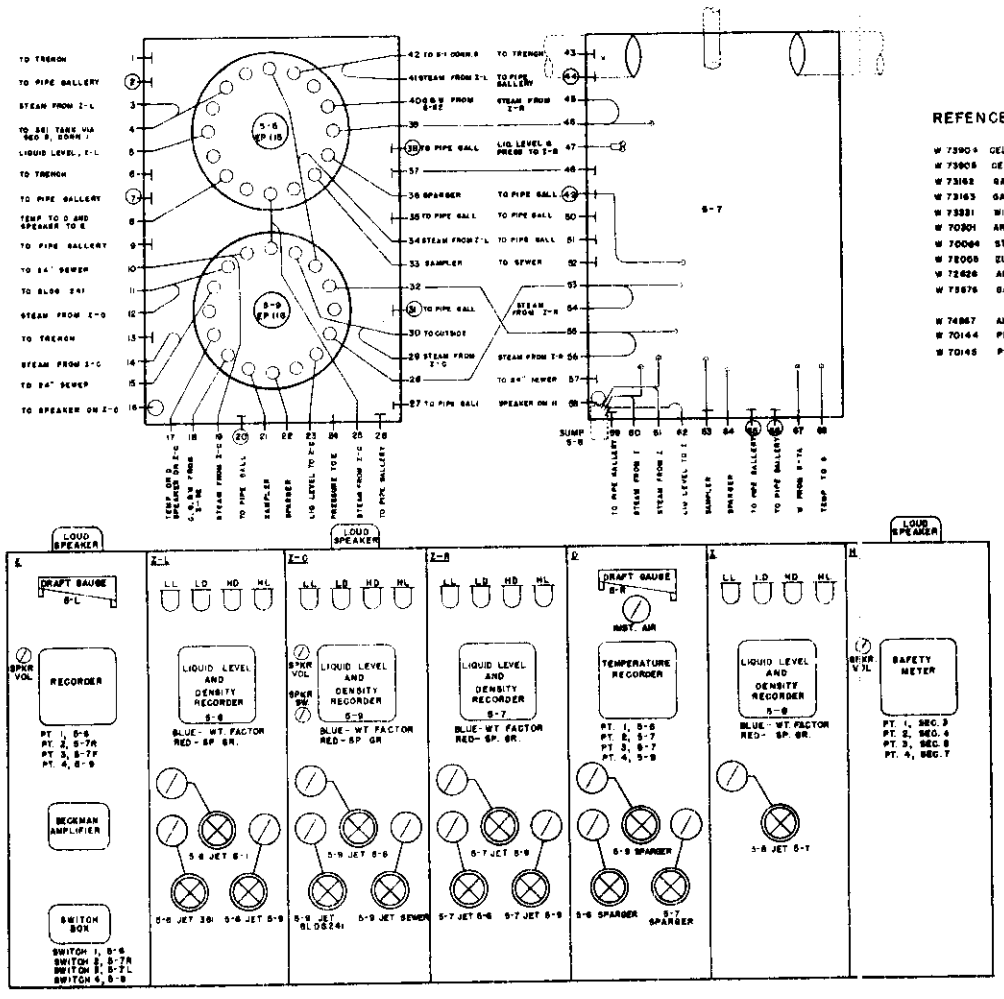
FIGURE 6

CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 5



REFENCE DRAWINGS

W 73904	CELL PIPING
W 73905	CELL PIPING
W 73162	GALLERY ARRG'T AND PIPING
W 73163	GALLERY ARRG'T AND PIPING
W 73331	WIRING DIAGRAM
W 70301	ARRANGEMENT KEY
W 70084	STRUCTURAL KEY
W 72068	ELECTRICAL KEY
W 72626	ARRANGEMENT, S-6 & S-9
W 73876	GAUGE BOARD AND SERVICE PIPING HEADERS
W 74867	ALTERNATE CELL PIPING
W 70144	PIPING IN CONCRETE
W 70145	PIPING IN CONCRETE

LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WALL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LD LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM

Jet connections within the section enable material to be jetted from the 5-8 Sump Tank to Tank 5-7, from Tank 5-7 to either of the left-hand tanks, and from Tank 5-6 and Tank 5-9 to each other. Both Tank 5-9 and Tank 5-7 are connected by jet lines to the cooling water sewer; however, the Tank 5-7 steam supply line is blanked in the pipe gallery.

Material from Tank 5-6 can be jetted to the 361 Settling Tank. This line originally went to Tank 9-1, and was intended for the disposal of very hot wastes that might collect in Section 5 due to a boil-over during dissolving or carbonate neutralization. In Section 9, this line has since been tied in with the outlet from Tank 9-4 to Tank X-204. The latter has, in turn, been broken near 361 Tank to provide the connection from Section 5.

Highly active material can be transferred to Tank 5-9 and jetted from there to 241 Building, Tank X-107. This waste line is tied into the stack drainage line. The valve in the stack drain line should be closed before jetting from Tank 5-9 to Tank X-107.

Sections 9 and 10 - Metal Waste Treatment

The instrumentation and equipment of these sections are practically identical with those of the standard sections (Chapter II) except that the Precipitators 9-1 and 10-1 are somewhat larger; 9 1/2 by 10-foot tanks with a capacity of 5000 gallons. (See Figure 7 for a diagram of the cell piping and gauge board for Section 9, and Figure 8 for that of Section 10). Also, the Ring Balance density recorder for Tank 9-4 has been altered to read Tank 9-1 pressure to detect gas evolution during carbonate neutralization. This has been done by placing the instrument across the draft gauge cell pressure line and the liquid level recorder vessel pressure line. Similar changes have been made in Section 10.

Two 400-gallon weigh tanks in position "A" and "B" supply soda ash to the precipitators. The "A" tanks are also connected to the caustic header. Spare chemical headers S and T are connected to 75-gallon "E" tanks in each section, also feeding the precipitators.

The precipitators, or waste neutralizers receive extraction waste from the catch tanks in Sections 7 and 8. The solution tanks, 9-4 and 10-4, also have inlets from the -4 tanks in Sections 6 and 7. The latter lines, which are not used, allow for disposal of the by-product cake from a pre-extraction decontamination step.

Section 15 - Decontamination Wastes

The cell piping and gauge board of Section 15 are shown in Figure 9. It may be noted that it differs from the standard sections in having no cross transfer lines between the two cells. This section is also illustrated further in Figures 10 and 11.

Each half of the section contains a small and large tank and functions

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FIGURE 7

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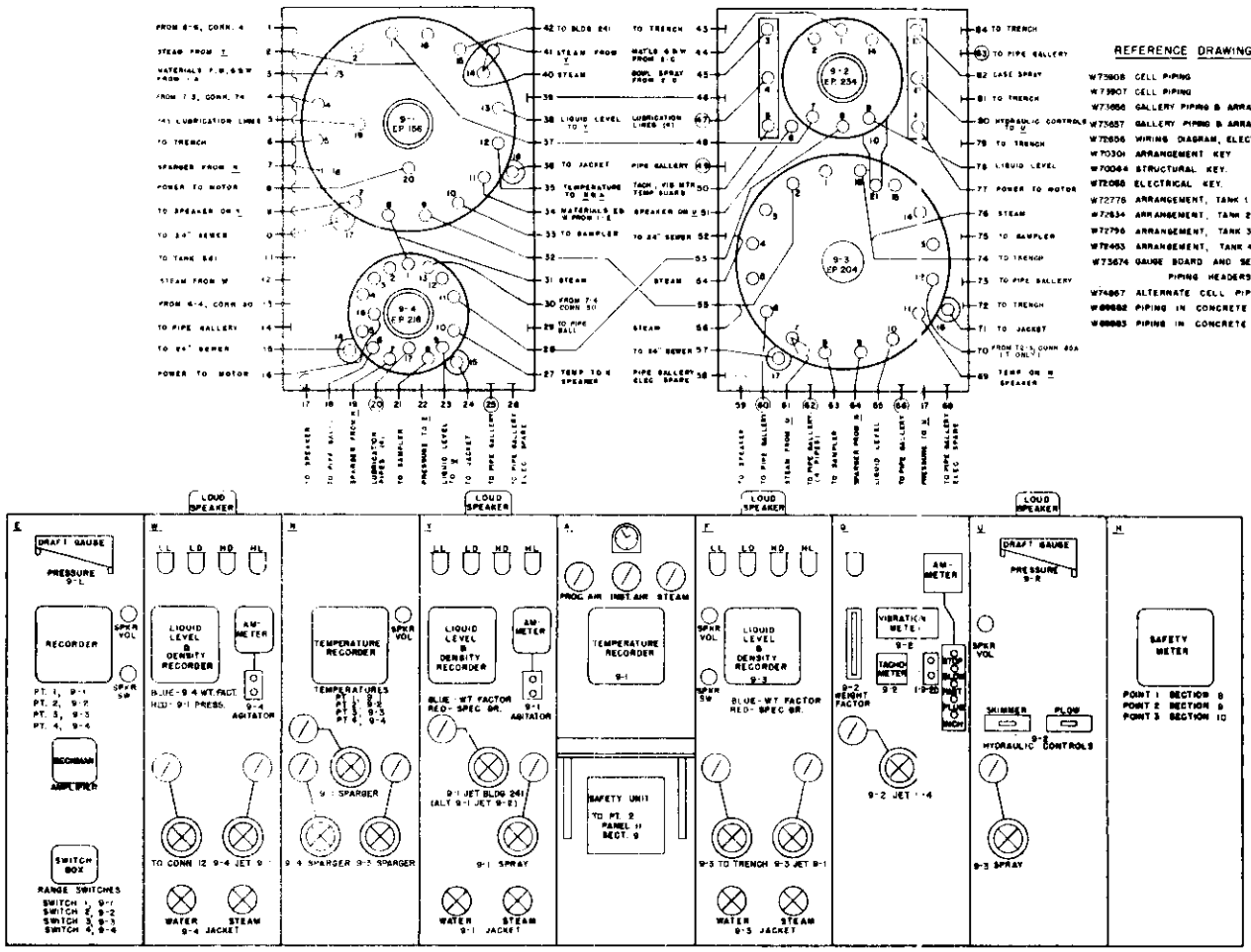
CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 9



LEGEND

- EQUIPMENT WITH MOTOR DRIVE
- EQUIPMENT WITH NO MOTOR DRIVE
- PRESSURE GAUGE
- GANG VALVE
- ORDINARY VALVE
- SPARE CELL WALL CONNECTOR NOZZLE
- LINE BLANKED IN PIPE GALLERY
- CONNECTOR NOZZLE
- BUBBLER, AIR SUPPLY TO MANOMETRIC SYSTEM
- LL LOW PRESSURE LEG, LIQUID LEVEL SYSTEM
- HL HIGH PRESSURE LEG, LIQUID LEVEL SYSTEM
- LO LOW PRESSURE LEG, DENSITY SYSTEM
- HD HIGH PRESSURE LEG, DENSITY SYSTEM

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FIGURE 8

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 10

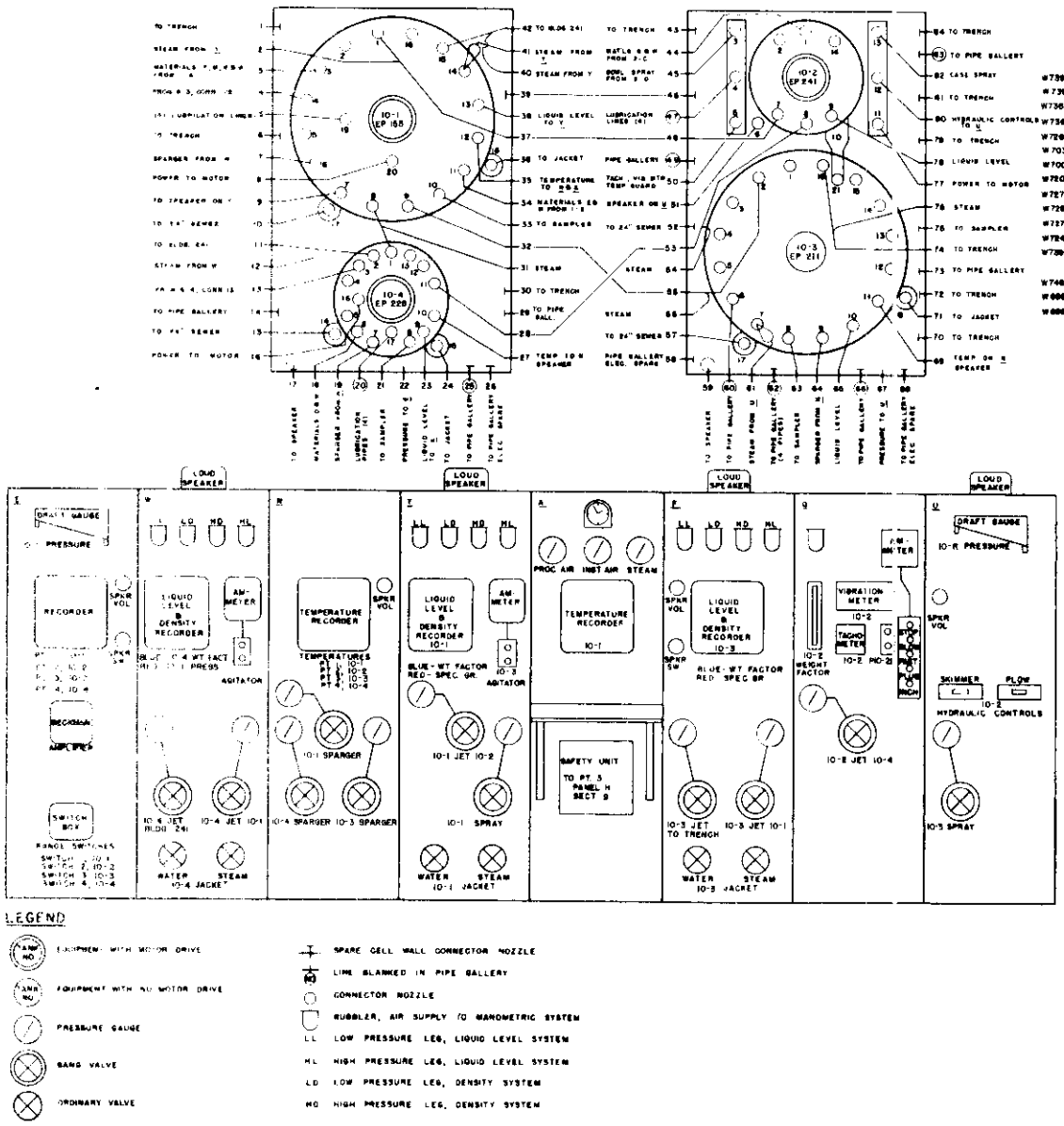

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FIGURE 9

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CELL PIPING AND GAUGE BOARD DIAGRAM, SECTION 15

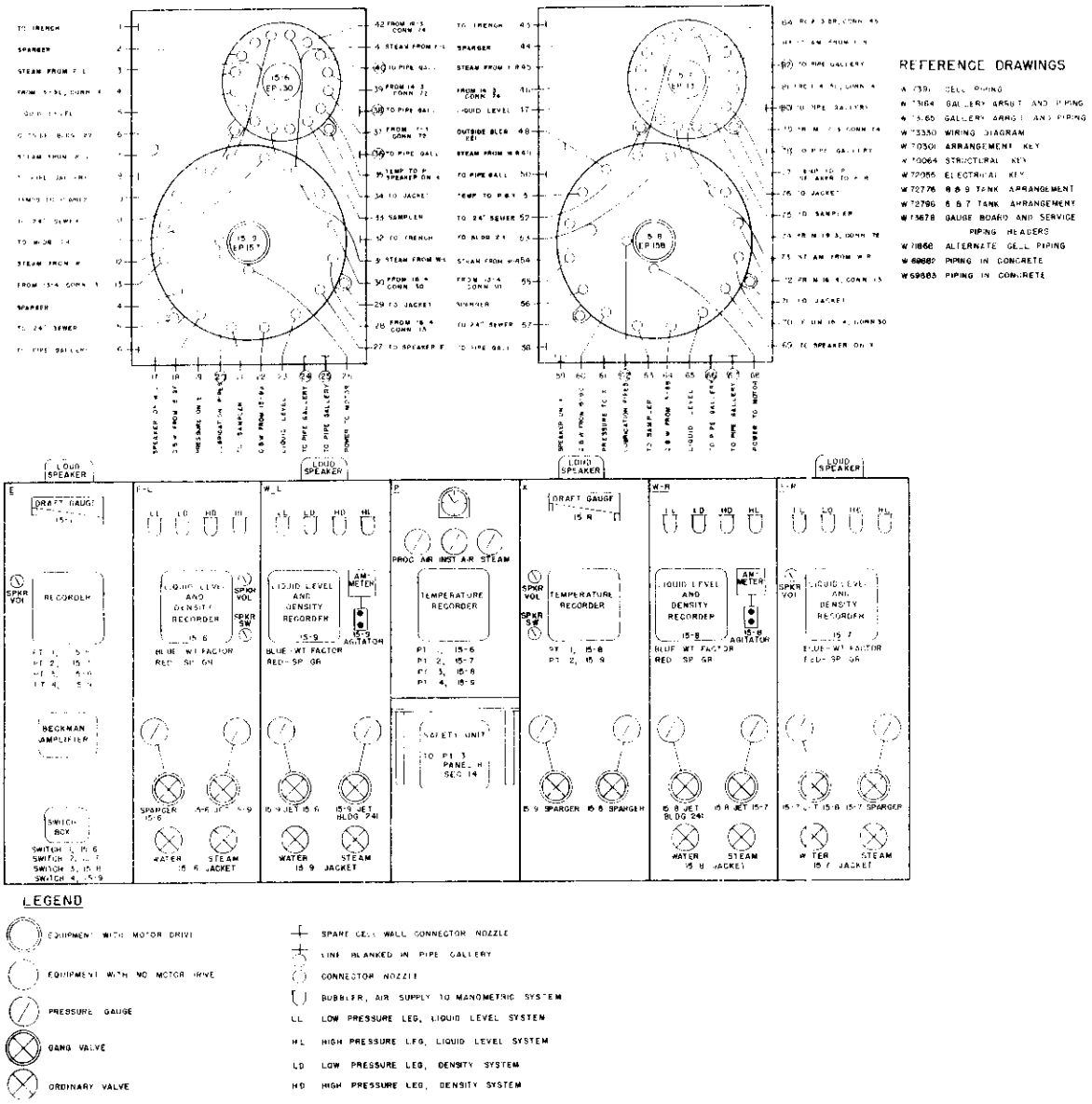

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FIGURE 10

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SECTION 15 - CANYON BUILDING

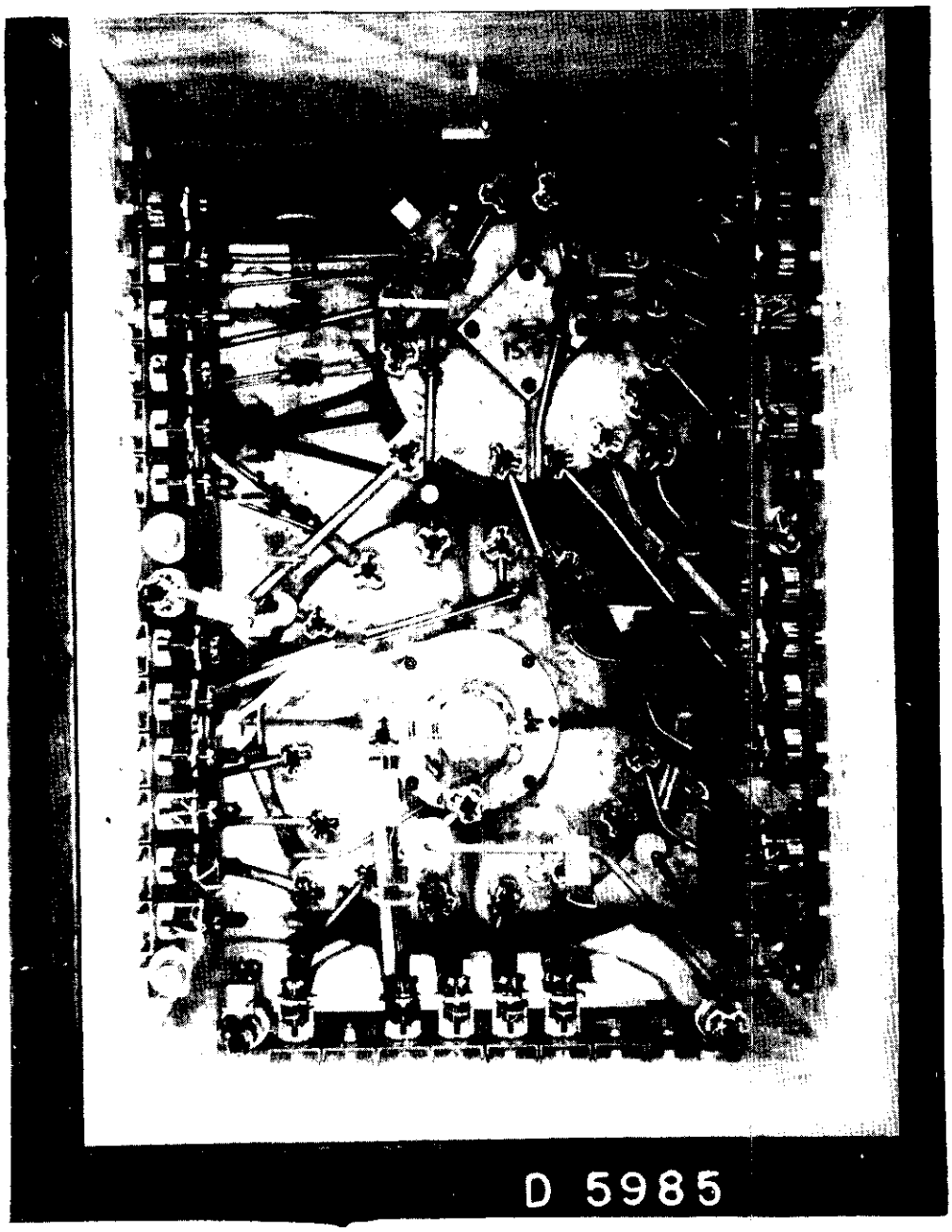

FIGURE 11

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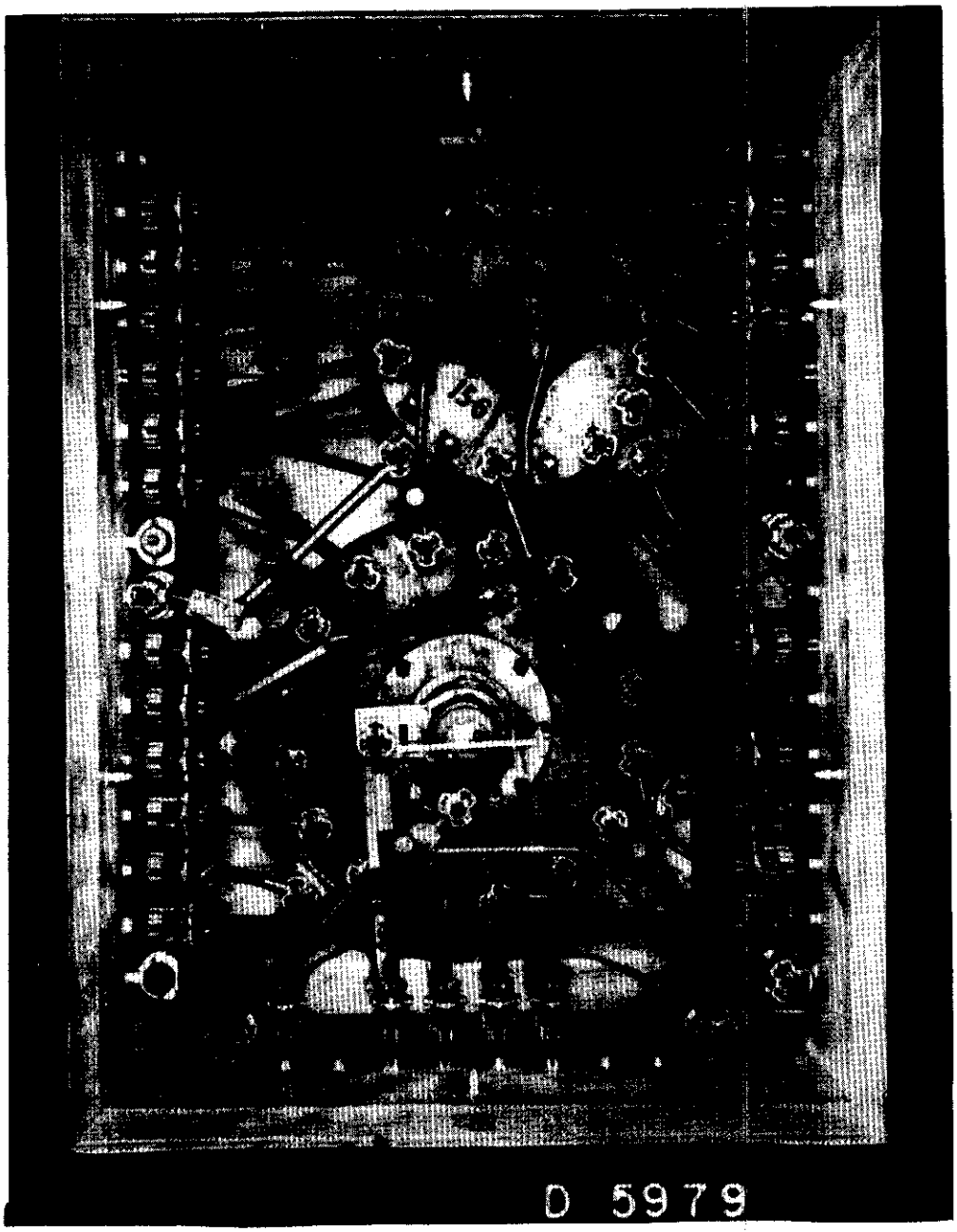
SECTION 15 - CANYON BUILDING



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independently to receive, neutralize, and dispose of waste. The smaller tanks, 15-6 and 15-7, are identical to the 6 1/2-foot diameter by 14-foot high crude product storage tanks, 12-6 and 12-9. They are unagitated jacketed vessels with a capacity of 3,380 gallons (20.2 gal./in.). The large tanks, 15-8 and 15-9, are 5000-gallon vessels duplicating 9-1 and 10-1.

Instruments on the gauge board record the liquid level, density, temperature and activity of all tanks. Each tank is provided with a sparger and Tanks 15-8 and 15-9 also have agitators.

Each of the small tanks is piped to receive by-product cake solution from all of the solution tanks in the by-product sections; 13-4, 16-4 and 18-4. Each of the large tanks can receive waste from all of the catch tanks in the product precipitation sections; 14-3, 17-3 and 19-3. There are two lines to 241 Building; Tank 15-8 to Tank X-107 and Tank 15-9 to Tank X-110. Cell piping, gang valves, etc., for an additional line from each of these tanks to 241 Building are installed, and blanked off in the rear of 221 Building. Coating waste from 3-5R goes to Tank 15-6 and from the other two dissolvers to Tank 15-7. Wastes received in the smaller tanks are jetted to the larger tank in the same cell for neutralizing.

All neutralizing is done in the larger tanks, through four 400-gallon scale tanks, 15-9A, 15-9F, 15-8B and 15-8C, all connected to the caustic header.

WASTE DISPOSAL PROCESS AND OPERATIONS - CANYON (221) BUILDING

Section 5, Cell Drainage

When some 10,000 pounds have accumulated in Tank 5-7 it is jetted over to Tank 5-6 and sampled. Analysis is for activity and acidity. Normally, the waste can be jetted to 361 Tank without treatment. Acidic material should be neutralized since neither the settling tank nor the dry well is of acid-resistant construction.

There is no upper limit established on the activity that can be sent to the settling tank and dry well. If a bad leak of overflow occurred in the canyon, the activity collected in Section 5 might be so high that it should be sent to the tank farm. In April, 1945 the 224 Building was sending to 361 Tank waste with an average gamma count of 25 c/(min.)(ml.). If a Section 5 waste runs more than 100 or possibly 1000 times this activity, it should probably be sent to permanent storage.

Metal Waste Treatment, Section 9 (Section 10 Spare)

The metal waste is brought nearly to the neutral point with caustic and then treated with excess sodium carbonate. This procedure yields an almost completely soluble waste, at minimum total volume.

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The composition of the carbonate complex formed with the uranium is not definitely known. There is some evidence that phosphate is included in the complex. Extraction wastes containing 0.4M or less phosphoric acid (0.6M is standard) give 15-30% undissolved solids, by volume, after standard treatment with caustic and soda ash, whereas at 0.6 to 0.8M phosphoric, the solids are 0 to 4%.

Toward the end of the initial caustic neutralization, at a pH of 2 to 3, the uranium is thrown down as a very bulky precipitate. It is reported that, if the caustic is added rapidly, the precipitate is less gelatinous and dissolves more readily on subsequent carbonate addition. Operating practice, therefore, is to add the caustic as rapidly as possible, not permitting the temperature to rise above 75 °C.

The optimum amounts of caustic and carbonate were originally set up by experiment as those which gave the minimum volume of neutralized waste containing less than 1% by volume of undissolved solids. On this basis, the caustic requirement was found to be 86-98% of that needed to reach a pH of 7, i.e., sufficient to give a pH of 1.2 to 6.

Since the original determination of the amounts of neutralizing chemicals required for treating the metal waste, operating conditions have been changed. Metal waste treatment is now calculated on the following basis:

For each 100 lb. of 73.5% phosphoric acid add 84.0 lb. of 50% caustic
For each 100 lb. of 93.0% sulfuric acid add 136.6 lb. of 50% caustic
For each 2200 lb. of metal add 1368.0 lb. of 50% caustic
For each 100 lb. of 50% caustic required, add 49.2 lb. of 30% soda ash
For each 2200 lb. of metal, add 12,745 lb. of 30% soda ash

The above amounts of caustic are calculated to be 90% of that required to reach a pH of 7.0. Additional carbonate to reach neutrality is calculated on the assumption that bicarbonate is formed, with little or no carbon dioxide evolution. The further amount of carbonate added to complex the metal is based on direct experimental determinations. It offers a margin of safety and is somewhat more than is strictly necessary.

Failure to use the full amount of caustic is not likely to cause any particular difficulty. There will be some gas evolution when the carbonate is added and slightly more carbonate will be necessary. The entire neutralization can in fact be carried out with soda ash. The sole reason for using caustic is the volume saving resulting from its greater solubility. Excess caustic may form precipitates which are difficult to dissolve. Hence, it is advisable to hold the caustic addition definitely short of the neutral point.

The 30% soda ash solution is saturated at 30 °C and freezes to a slush at 27 °C. It must, therefore, be kept in heated storage tanks and transferred in lagged and chased lines.

Experimental data on the amount of self-heating to be expected from the

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metal waste are not available. In May, 1945 Tank 241-T-101, with waste from some 40 tons of metal was no warmer than the other tanks; 76-80°F. However, by June 1st these tanks were 10-15°C warmer than those containing second cycle wastes. Various calculators have estimated that a 75-foot storage tank full of metal waste will self heat to boiling in (1) 200-700 days, and (2) not at all.

The carbonate complex of uranium is stable at room temperature, but most of the metal will precipitate, as a uranium-phosphate-carbonate mixture, if heating occurs. At 75°C, the waste precipitates half to three quarters of the metal in five days. The precipitate carries 90% or more of the fission activity.

Coating Waste, Section 15

The coating solution proper, water wash, and acid wash are successively received in Tanks 15-6 or 15-7. When all three are in Section 15, they are mixed, jetted to the corresponding neutralizer and thence to 241 Building. The operation is extremely simple. No control samples are taken. The mixed waste should be jetted to 241 Building immediately. There is some possibility that the insoluble form of alumina will precipitate after the coating solution is diluted and partially neutralized with the washes.

Decontamination Wastes, Section 15

First cycle wastes are treated in Section 15-R, second cycle in Section 15-L. This makes it possible to segregate the less active second cycle wastes at 241 Building in the X-110 series of tanks, from which it may eventually be safe to discard them into the ground.

As an example of the operating procedure, a by-product cake solution is transferred from Tank 13-4 to Tank 15-7 and half of a product waste from Tank 14-3 is jetted to Tank 15-8. The two are then mixed in Tank 15-8 and neutralized with 50% sodium hydroxide, keeping the temperature below 50°C. The waste is then jetted to 241 Building. To complete the disposal of a first cycle waste, the second half of the waste from Tank 14-3 is brought into Tank 15-8, neutralized and jetted out. The by-product cake solutions are not neutralized by themselves because of the vigorous reaction between the 10N nitric acid solution and strong caustic. No samples are taken.

Caustic requirements for decontamination waste neutralization are calculated on the basis of 10% excess over the theoretical amounts. The quantity of 50% sodium hydroxide required to neutralize each of the process solutions to a pH of 7 has been determined experimentally:

For each 100 lb. of 60% HNO_3, add 79.1 lb. of 50% caustic
For each 100 lb. of 73.5% H_3PO_4/1.5% HNO_3 add 116.3 lb. of 50% caustic
For each 100 lb. of 14% $(NH_4)_2SiF_6$ solution add 29.2 lb. of 50% caustic
For each 100 lb. of 24% $BiONO_3$ solution add 27.4 lb. of 50% caustic
For each 100 lb. of 20% Fe solution add 0.7 lb. of 50% caustic

[redacted]

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The neutralized wastes contain a quantity of suspended solids - hydroxides or phosphates of bismuth, scavenger metals, chromium and iron and probably sodium silicofluoride. These slurries jet without difficulty. The settling characteristics have not been determined. In view of the general high carrying of both product and by-product activities by alkaline precipitates, it is safe to state that at least 90% of each type of activity will be removed from the supernatant by any substantial amount of precipitate.

CONCENTRATION (224) BUILDING WASTE DISPOSAL EQUIPMENT

Process waste lines in 224 Building are diagrammed in Figure 12. The several types of waste are all treated in Cell C.

Process Sewers

Cooling water from 224 Building process vessels flows to a 10-inch sewer in the rear of the building which joins the 221 Building sewer. Two sewers under Cells A and B and Cells D and E respectively deliver cell drainage to the deep right hand portion of Cell C. Drainage in Cell F collects in sumps and is jetted up into Tank F-8.

Cell C

The right-hand portion of Cell C is 19 feet lower than the floors of the other cells. Floor drainage from A, B, C-L, D and E collects in Tank C-7, a 9-foot by 9-foot 4,000 gallon tank. In the left hand side of the cell is the waste neutralizer, a 9-foot by 9 foot, 4,000 gallon jacketed agitated tank.

Inlets to Tank C-8 are provided from all of the process vessels in which wastes are normally formed (see Figure 12). In the present process these comprise bismuth phosphate by-product cake solution from A-4, fluoride by-product slurry from D-4, fluoride waste from E-4, and metathesis wastes from F-7 and F-8. Cell drainage is also received in C-8 from C-7 and from the Cell F sumps via F-8. The only outlet is to the 361 Settling Tank.

Gauge board instrumentation records the liquid level, density, temperature and activity of solutions in tanks C-7 and C-8. Caustic is supplied to tank C-8 from the 400 gallon scale tank, C-8A.

CONCENTRATION (224) BUILDING WASTE DISPOSAL PROCESS AND OPERATIONS

Waste disposal procedures in 224 Building are conducted on a more flexible basis than in 221 Building. This is necessary because five wastes per

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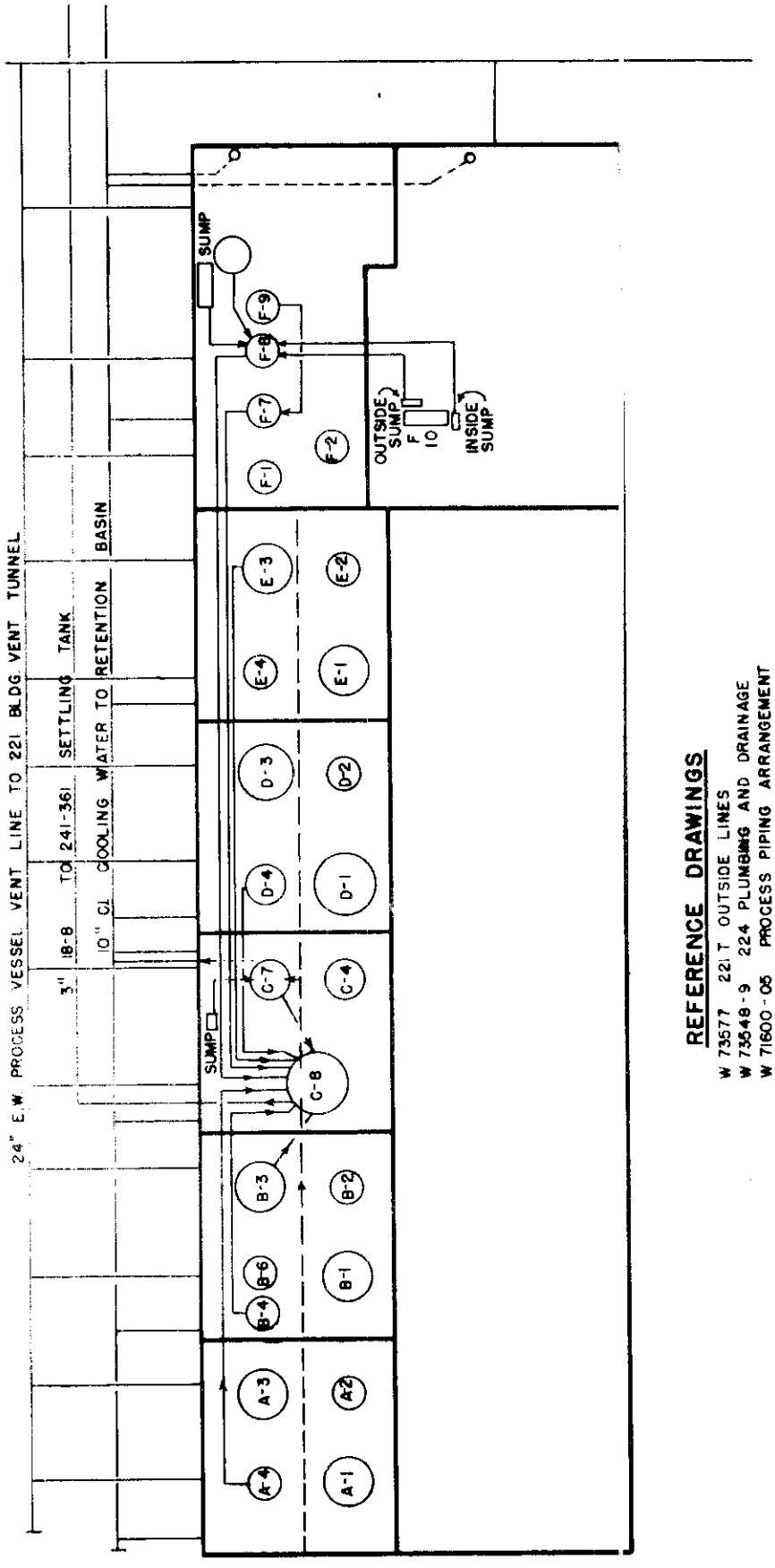
FIGURE 12

PROCESS WASTE LINES, CONCENTRATION (224) BUILDING



REFERENCE DRAWINGS

W 73577 221 T OUTSIDE LINES
W 73548-9 224 PLUMBING AND DRAINAGE
W 71600-05 PROCESS PIPING ARRANGEMENT
W 72937-9 CELL PIPING ARRANGEMENT
W 73527-9 CELL PIPING ARRANGEMENT
W 72360-1 CELL PIPNG ARRANGEMENT
W 76179-80 CELL PIPING ARRANGEMENT
W 74673-77 CELL PIPING ARRANGEMENT

run must be put through Cell C equipment. The wastes may be disposed of individually, or two or three may be combined, whichever proves to be most convenient.

The bismuth by-product cake solution is neutralized with 83.8 lb. of 50% caustic per 100 lb. of 60% nitric acid used to dissolve the cake. If convenient, this waste may be neutralized with half of the fluoride product precipitation waste, E-3WS. If the bismuth phosphate by-product waste (A-4BP) is treated alone, it is diluted with 6000 pounds of water per run before caustic addition, to make the reaction less vigorous.

The fluoride by-product waste, D-4BP, is an aqueous slurry. As a safety factor, 50 pounds of 50% sodium hydroxide is added per run to take care of residual acid from process solutions.

The fluoride product precipitation waste, E-3WS, is so large that it is received and neutralized in C-8 in two portions. The following factors are used to calculate caustic requirements:

For each 100 lb. of 60% HNO_3 (17-4P), add 84 lb. of 50% caustic
For each 100 lb. of HF added in D and E, add 400 lb. of 50% caustic
For each 100 lb. of 73.5% H_3PO_4 added in A, add 182 lb. of 50% caustic
For each 100 lb. of solid $H_2C_2O_4$, add 125 lb. of 50% caustic

The alkaline metathesis wastes, F-7WS and F-9WS, are jetted through Tank C-8 to the settling tank. If convenient, they may be combined with either or both of the by-product wastes. No account is taken of the free alkali in the metathesis wastes in calculating the caustic requirements of the by-product wastes.

Floor washings from Cell F are collected in Tank F-8, assayed for product and jetted to waste via Tank C-8.

ISOLATION (231) BUILDING WASTE DISPOSAL EQUIPMENT

Sump Tank (231-W-151)

A 39,000 gallon stainless steel sump tank (231-W-151) illustrated diagrammatically in Figure 85, Chapter II is provided for the collection of active waste solutions from the process cells and laboratories in the Isolation (231) Building, which are fed to this tank through two 3-inch stainless steel lines. This sump tank is provided with an agitator, and includes pipe connections at the grade level to sodium hydroxide drums for neutralization, and through a dip tube to a steam jet, whereby the contents may be emptied to the Reverse Flow Well 231-W-150 or buried sumps.

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Open Ditch

An open ditch is provided (See Figure 77, Chapter II) adjacent to the 231-W Building which is used for the disposal of innocuous wastes from the laboratories and process cells.

Tile Field

A tile field is provided (See Figure 77, Chapter II) near the 231-W Building for disposal of sanitary wastes.

WASTE DISPOSAL PROCESS AND OPERATIONS - ISOLATION (231) BUILDING

The active waste solutions form process cells and laboratory sinks which collect in the sump tank (231-W-151) are usually acidic in character, and contain product to the extent of a few micrograms per liter. These waste solutions are neutralized with sodium hydroxide, and then jetted to the dry wells or buried sumps.

[redacted]

[redacted]

REFERENCES

DECLASSIFIED

Operating Standard 221-81, Coating Waste

Operating Standard 221-82, Metal Waste

Operating Standard 221-83, Decontamination Wastes

Operating Standard 224-81, 224 Building Wastes

Report CN-2021
Coating waste, pp. 10, 11
Metal waste, pp. 24, 26
Decontamination wastes, pp. 38, 41, 48, 55-7
224 Building wastes, pp. 48, 67-8, 77-8

Report CN-1860, Site X Waste Disposal

Report CN-1418, Site X Waste Disposal

Geological
Letter and enclosures, 5/13/44, Chambers to Daniels
Letter, "HEW Class 4 Waste Disposal", 10/18/44, Chambers to Kay

Absorption of Activity by W Soils, Overstreet and Jacobsen, Berkeley, 10/3/44 (no report number); also, covering letter, J. G. Hamilton to N. Hilberry, 9/29/44

Report CN-2045

[redacted]

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DRY WELL DATA

The seven test wells in each area, 241-130 to 136 inclusive, are 150 feet deep. Data on the wells actually used for waste disposal are given in the following tables. All wells are of 6-inch iron pipe except the 222-110 wells, which are of 3-inch stainless steel.

Well	Depth, Ft.	Elev. at Grade, Ft.	Length Perforated, Ft.
241-T-361	287	707	62
241-T-361A	206	707	100
241-U-361	302	694	66
241-B-361	302	683	59
222-T-110	75	775	25
222-U-110	75		25
222-B-110	75	701	25
231-W-150	150		32

Logs of Dry Wells

The logs of the waste disposal wells are given below. Opposite each depth in feet the soil composition is reported from that point down to the next depth given. The symbols are explained by the following key.

Key

B - boulders	F - fine
Ba - basalt	G - gravel
Br - brown	Q - quartz
C - coarse	S - sand
Cl - clay	Y - yellow

241-T-361

Depth	Soil	Depth	Soil	Depth	Soil
[illegible]	G	105	Br Cl	212	B
[illegible]	S & G	120	Cl & S	214	S & G
[illegible]	G	125	G	245	G
17	CG	137	Br Cl	250	FG & S
19	S & G		S & G	270	FS
28	CG	160	G & S	275	CG
32	S	167	B	285	Wet S
36	G	175	G	287	
40	S	198	G & S		

[redacted]

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DECLASSIFIED

241-T-361A

Depth	Soil	Depth	Soil	Depth	Soil
0	S & G	77	G	183	G
2	S & G	85	S & G	205	
13	B	162	CG		
29	S & G	175	B		

241-U-361

Depth	Soil	Depth	Soil	Depth	Soil
0	S & G	117	Cl & S	224	S & G
18	FS	150	Y Cl	250	G
32	G, Ba & S	158	Sandy Cl	268	CG & B
45	S	184	CG	273	S & G
79	S & G	189	G & B	299	S
103	S	195	CG	302	
111	Y Cl & S	216	G		

241-B-361

Depth	Soil	Depth	Soil	Depth	Soil
0	S & G	60	S	290	S
38	S	205	S & G	295	CS
55	S & G	230	G	302	

222-T-110

Depth	Soil	Depth	Soil	Depth	Soil
0	B	24	G & B	53	G
6	G	34	CG	72	S
14	CG	39	G	75	
20	B	40	S		

222-U-110

Depth	Soil
0	G
23	S
40	S & G
54	S
75	

222-B-110

Depth	Soil
0	G
27	S & G
40	S
75	

[redacted]

241 BUILDING REFERENCE DRAWINGS

UNCLASSIFIED

Maps

2892, Sheets 10-11 "T"
2892, Sheets 18-19, "U"
W-72182, Plot Plan
2890, Sheet 33, "B"
2890, Sheet 27, "C"

75-Foot Diameter Tanks

BPF-73550
W-72743, Arrangement
W-71387, Concrete
W-74108, Details
D-64000, Riser Plugs
BPF-74353, Thermometer

20-Foot Diameter Storage Tanks

BPF-73550
W-72417, Concrete
W-74137, Pipe Supports
W-72742, Arrangement
D-64000, Riser Plugs
BPF-74353, Thermometer

Condensers

W-72927, Arrangement
D-62839, Details

Diversion Boxes

W-72183, Arrangement, Plan
W-72184, Arrangement, Plan
W-72185, Arrangement, Section
W-72186, Steel Framing
W-72461, Concrete
W-72484, Cover Slabs
D-62677, Jumpers
D-61117, Saddle Brackets
D-611194, Kick Plates
D-61114F, Flanges
D-61116C, Connector Blocks
D-60749B, Horizontal Steel Guide
D-60746A, Cast Steel Nut
D-60743, Screw and Key
D-60744, Dowel Pin & Jaw Guide
D-61446, Jaw and Pin
D-63149, Slab Lifting Beam

Retention Basin

W-73636, Concrete
W-73975, Arrangement

Settling Tank and Dry Well

BPF 74650, 20 Foot Diameter Settling Tank, 241-361
W-72902, Tank and Dry Well Arrangement

Catch Tank for Diversion Box Drainage

BPF-74650, 20 Foot Diameter Catch Tank, 241-301
W-72903, Arrangement

[redacted]

DECLASSIFIED

Electrical

Map 2928, Sheet 10, "T"
Map 2928, Sheet 18, "U"
W-75060, Flood Lighting
W-76157, Floodlight Towers
W-74875, 440V Receptacle and Wrench Control Details
Map 2906, Sheet 33, "B"
Map 2906, Sheet 27, "C"
W-71636, Distribution Diagram, E Area
W-72024, Distribution Diagram, W Area

Buried Sumps

231 Building H-2-346
221-T Building and 222-T Building H-2-353

[redacted]

Date_____
Run No._____

DECLASSIFIED

JTRALIZATION - WASTE FROM COATING REMOVAL

When notified by the dispatcher that coating removal waste solutions are ready for neutralization, turn on 15-7 jacket cooling water. — 15-7 Jacket water on_____

1. Beckman reading on 15-7 — Meter_____ Factor_____
2. Receive coating solution waste from 4-5L in 15-7 (TW 5300 lbs) — Time start____ Time end_____ 15-7 Wt. Ftr._____ Lbs._____
3. Receive wash water from 4-5L in 15-7 (Approx. 860 lbs from 4-5L) — Time start____ Time end_____ 15-7 Wt. Ftr._____ Lbs._____
4. Beckman reading on 15-7 — Meter_____ Factor_____
5. Beckman reading on 15-8 — Meter_____ Factor_____
6. Start 15-8 agitator and turn on 15-8 jacket cooling water — 15-8 Agitator on_____ 15-8 Jacket water on_____
7. Jet waste solution from 15-7 to 15-8 — Time start____ Time end_____
8. Receive acid wash solution in 15-7 from 4-5L. (TW 4425 lbs) — Time start____ Time end_____ 15-7 Wt. Ftr._____ Lbs._____
9. Beckman reading on 15-7 — Meter_____ Factor_____
10. Jet acid wash solution from 15-7 to 15-8. Hold temperature below 50°C. by adjusting rate of addition — Time start____ Time end_____ 15-8 Wt. Ftr._____ Lbs._____
11. Beckman reading on 15-8 — Meter_____ Factor_____
12. Turn off 15-7 jacket cooling water — 15-7 Jacket water off_____
13. Agitate the mixed waste in 15-8 for 30 minutes and cool to 35°C. — Time start____ Time end_____ Time 35°C._____

Date_____
Run No._____

DECLASSIFIED

TRALIZATION - WASTE FROM COATING REMOVAL (Continued)

Shut off 15-8 agitator and read Wt. FTR.,Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 15-8 agitator.	15-8 Agitator off_____ 15-8 Wt. Ftr._____Lbs._____ 15-8 Sp. Gr._____Temp._____ 15-8 Agitator on_____
Call dispatcher and obtain permission to jet neutralized waste from 15-8 to Bldg. 241 (Tank X-107)	Time called_____ Time start_____Time end_____ Lbs Transferred_____
17. Shut off 15-8 agitator and jacket cooling water.	15-8 Agitator off_____ 15-8 Jacket water off_____
18. Peckman reading on 15-8	Meter_____Factor_____

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DECLASSIFIED

Date_____
Run No._____

TRALIZATION - METAL WASTE SOLUTION - FIRST HALF

When notified by the dispatcher that metal waste solution is ready for neutralization, start 9-1 agitator. — 9-1 Agitator on_____

2. Turn on 9-1 jacket cooling water. — 9-1 Jacket water on_____

3. Beckman reading on 9-1 — Meter_____ Factor_____

4. Allow the 9-1 to 241 Bldg. jet to remain on air for 20 minutes after 9-1 is emptied to blow out line, then receive the first half of the metal waste solution from 8-3 into 9-1. (TW approx 16,700 lbs.) — Time start_____ Time end_____

5. Stop 9-1 agitator and read Wt. Ftr., Temp. and Sp. Gr when Sp. Gr. Meter constant, then start 9-1 agitator again. — 9-1 Agitator off_____
9-1 Wt. Ftr._____ Lbs._____
9-1 Sp. Gr._____ Temp._____
9-1 Agitator on_____

6. Beckman reading on 9-1 — Meter_____ Factor_____

7. Add 2760 lbs. of 50% Sodium Hydroxide (Line Q) to 9-1 via 9-1A. Hold the temperature below 75°C by adjusting the rate of addition. — Time start_____ Time end_____
Lbs. added_____
Maximum Temp._____

8. Add 10905 lbs. of 30% Sodium Carbonate solution (Line P) to 9-1 via 9-1A in 3 portions of 3635 lbs. each. adjust the rate of addition so that maximum pressure does not exceed 5 ins. water. — Time start_____ Time end_____
1st Port_____ Lbs. Time added_____
2nd Port_____ Lbs. Time added_____
3rd Port_____ Lbs. Time added_____
Total lbs. added_____

9. Cool 9-1 to 35°C and leave cooling water on. — Time 35°C_____

10. Stop 9-1 agitator and read Wt. Ftr., Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 9-1 agitator. — 9-1 Agitator off_____
9-1 Wt. Ftr_____ Lbs._____
9-1 Sp. Gr._____ Temp._____
9-1 Agitator on_____

11. Beckman reading on 9-1. — Meter_____ Factor_____

12. Call dispatcher and obtain permission to jet neutralized waste from 9-1 to 241 Bldg. (Approx. 32,000 lbs. to tank W-101) Be sure that 9-1 is emptied. Shut off 9-1 agitator and jacket cooling water when the Wt. Ftr. reaches 0.2. — Time called_____
Time approved_____
Time start_____ Time end_____
Lbs. jetted_____

Date_____
Run No._____

DECLASSIFIED

I TRALIZATION - METAL WASTE SOLUTION - SECOND HALF

	When notified by the dispatcher that metal waste solution is ready for neutralization, start 9-1 agitator.	9-1 Agitator on_____
2.	Turn on 9-1 jacket cooling water.	9-1 Jacket water on_____
3.	Beckman reading on 9-1	Meter_____ Factor_____
4.	Allow the 9-1 to 241 Bldg. jet to remain on air for 20 min. after 9-1 is emptied to blow out line, then receive the second half of the metal waste solution from 8-3 into 9-1. (TW from 8-3 Approx. 16,700 lbs.)	Time start____ Time end_____
5.	Stop 9-1 agitator and read Wt. Ftr., Temp. and Sp. Gr. when Sp. Gr. meter becomes constant, then start 9-1 agitator.	9-1 Agitator off_____ 9-1 Wt. Ftr.____ Lbs._____ 9-1 Sp. Gr.____ Temp._____ 9-1 Agitator on_____
6.	Beckman reading on 9-1	Meter_____ Factor_____
7.	Add 2760 lbs. of 50% Sodium Hydroxide (Line Q) to 9-1 via 9-1A. Hold the temperature below 75°C by adjusting the rate of addition.	Time start____ Time end_____ Lbs. added_____ Maximum Temp._____
8.	Add 10905 lbs. of 30% Sodium Carbonate solution (Line P) to 9-1 via 9-1A in 3 portions of 3635 lbs. each. Adjust the rate of addition so that maximum pressure does not exceed 5 ins. water.	1st Port____ Lbs. Time Added____ 2nd Port.____ Lbs. Time added____ 3rd Port____ Lbs. Time added____ Total lbs. added_____
9.	Cool 9-1 to 35°C and leave cooling water on.	Time 35°C_____
10.	Stop 9-1 agitator and read Wt. Ftr., Temp. and Sp. Gr. when Sp. Gr. Meter becomes constant, then start 9-1 agitator.	9-1 Agitator off_____ 9-1 Wt. Ftr.____ Lbs._____ 9-1 Sp. Gr.____ Temp._____ 9-1 Agitator on_____
11.	Beckman reading on 9-1	Meter_____ Factor_____
12.	Call dispatcher and obtain permission to jet neutralized waste from 9-1 to 241 Bldg. (Approx. 31,000 lbs. to tank X-101) Be sure that 9-1 is emptied. Shut off 9-1 agitator and jacket cooling water when the Wt. Ftr. reaches 0.2.	Time called_____ Time approved_____ Time start____ Time end_____ Lbs. jetted_____

[redacted]

DECLASSIFIED

Date_____
Run No._____

TRALIZATION - BY-PRODUCT AND PRODUCT WASTE SOLUTIONS - 1ST HALF
DECONTAMINATION

	When notified by the dispatcher that 1st decontamination waste solutions are ready for neutralization, start 15-8 agitator and turn cooling water on 15-8 jacket	15-8 agitator on_____ 15-8 jacket water on_____
2.	Beckman reading on 15-8	Meter_____ Factor_____
3.	Allow the 15-8 to 241 Bldg. jet to remain on air for 20 min. after 15-8 is emptied to blow out the line, then receive the by-product cake solution from 13-4 in 15-8. (TW from 13-4 approx. 3500 lbs.)	Time start_____ Time end_____ 15-8 Wt. Ftr.___ Lbs._____
4.	Beckman reading on 15-8	Meter_____ Factor_____
5.	Beckman reading on 15-7	Meter_____ Factor_____
6.	Turn on 15-7 air sparger and jacket cooling water.	15-7 air sparger on_____ 15-7 jacket water on_____
7.	Receive the first half of the product waste solution from 14-3 in 15-7.(TW from 14-3 approx. 14,750 lbs.)	Time start____ Time end_____ 15-7 Wt. Ftr.___ Lbs._____
8.	Beckman reading on 15-7	Meter_____ Factor_____
9.	Jet waste solution from 15-7 to 15-8	Time start____ Time end_____
10.	Shut off 15-7 air sparger and jacket cooling water.	15-7 Air sparger off_____ 15-7 jacket water off_____
11.	Add 5035 lbs. of 50% Sodium Hydroxide (Line Q) to 15-8 via 15-8B.(2160 lbs. for cake and 2875 lbs. for solution). Hold temperature below 50°C adjusting the rate of addition.	Time start____ Time end_____ Lbs. added_____
12.	Agitate 15-8 for 10 minutes	Time start____ Time end_____
13.	Shut off 15-8 agitator and read Wt. Ftr., Temp.and Sp.Gr. when the Sp.Gr. meter becomes constant, then start 15-8 agitator.	15-8 agitator off_____ 15-8 Wt.Ftr.____ Lbs._____ 15-8 Sp.Gr.____ Temp._____ 15-8 agitator on_____
14.	Call dispatcher, give charge data and obtain permission to jet waste solution from 15-8 to Bldg. 241 (Tank X-107).	Time called_____ Time start____ Time end_____ Lbs. transferred_____
15.	Shut off 15-8 agitator and jacket cooling water.	15-8 agitator off_____ 15-8 jacket water off_____

DECLASSIFIED

Date_____
Run No._____

TRALIZATION - PRODUCT WASTE SOLUTION - 2ND HALF - 1ST DECONTAMINATION

	When notified by the dispatcher, on 15-7 air sparger and jacket cooling water.	15-7 Air sparger on_____ 15-7 jacket on_____
3.	Beckman reading on 15-7	Meter_____ Factor_____
3.	Receive the second half of the product waste solution from 14-3 in 15-7 (TW from 14-3 approx. 14,750 lbs.)	Time start____ Time end_____ 15-7 Wt. Ftr.____ Lbs._____
4.	Start 15-8 agitator and turn cooling water on 15-8 jacket.	15-8 agitator on_____ 15-8 jacket on_____
5.	Beckman reading on 15-8.	Meter_____ Factor_____
6.	Allow the 15-8 to 241 Bldg. jet to remain on air for 20 minutes after 15-8 is emptied to blow out the line, then jet 15-7 to 15-8. Shut off 15-7 air sparger and jacket water on completion of transfer.	Time start____ Time end_____ 15-7 Air sparger off_____ 15-7 Jacket water off_____
7.	Beckman reading on 15-7.	Meter_____ Factor_____
8.	Beckman reading on 15-8	Meter_____ Factor_____
9.	Add 2875 lbs. of 50% Sodium Hydroxide (Line Q) to 15-8 via 15-8B. Hold temperature below 50°C by adjusting the rate of addition.	Time start____ Time end_____ Lbs. added_____
10.	Agitate 15-8 for 10 minutes.	Time start____ Time end_____
11.	Shut off 15-8 agitator and read Wt. Ftr., Temp and Sp.Gr. when the Sp. Gr. meter becomes constant, then start 15-8 agitator.	15-8 agitator off_____ 15-8 Wt. Ftr.____ Lbs._____ 15-8 Sp. Gr.____ Temp._____ 15-8 agitator on_____
12.	Call dispatcher, give charge data and obtain permission to jet waste solution from 15-8 to 241 bldg. (Tank X-107)	Time called_____ Time start____ Time end_____
13.	Shut off 15-8 agitator and turn cooling water off 15-8 jacket.	15-8 agitator off_____ 15-8 jacket water off_____
14.	Beckman reading on 15-8.	Meter_____ Factor_____

[redacted]

Date_____
Run No._____

DECLASSIFIED

UTRALIZATION - BY-PRODUCT AND PRODUCT WASTE SOLUTIONS - 1ST HALF -
D DECONTAMINATION

When notified by the dispatcher that 2nd decontamination waste solutions are ready for neutralization, start 15-9 agitator and turn cooling water on 15-9 jacket.

15-9 Agitator on_____
15-9 jacket water on_____

2. Beckman reading on 15-9. Meter_____ Factor_____

3. Allow the 15-9 to 241 Bldg. jet to remain on air for 20 minutes after 15-9 is emptied to blow out the line, then receive the by-product cake solution from 26-4 in 15-9. (TW approx. 1750 lbs.) Time start_____ Time end_____

4. Beckman reading on 15-9. Meter_____ Factor_____

5. Beckman reading on 15-6 Meter_____ Factor_____

6. Turn on 15-6 air sparger and jacket cooling water.
15-6 jacket water on_____
15-6 air sparger on_____

7. Receive the first half of the product waste solution from 17-3 in 15-6 (TW from 17-3 approx. 12,250 lbs.) Time start_____ Time end_____

8. Beckman reading on 15-6. Meter_____ Factor_____

9. Jet waste solution from 15-6 to 15-9. Time start_____ Time end_____

10. Shut off 15-6 air sparger and jacket cooling water.
15-6 air sparger off_____
15-6 jacket water off_____

11. Add 2860 lbs. of 50% Sodium Hydroxide (Line Q) to 15-9 via 15-9A. (930 lbs. for cake and 1930 lbs. for solution.) Hold temperature below 50°C by adjusting the rate of caustic addition.
Time start_____ Time end_____
Lbs. added_____

12. Agitate 15-9 for 10 minutes. Time start_____ Time end_____

13. Shut off 15-9 agitator and read Wt. Ftr. Temp. and Sp.Gr. when Sp. Gr. meter becomes constant, then start 15-9 agitator.
15-9 Agitator off_____
15-9 Wt. Ftr._____ Lbs._____
15-9 Sp. Gr._____ Temp._____
15-9 Agitator on_____

14. Call dispatcher, give charge data and obtain permission to jet waste solution from 15-9 to Bldg. 241 (Tank X-110)
Time called_____
Time start_____ Time end_____
Lbs. transferred_____

15. Shut off 15-9 agitator and jacket cooling water.
15-9 agitator off_____
15-9 jacket water off_____

Date_____
Run No._____

DECLASSIFIED

UTRALIZATION PRODUCT WASTE SOLUTION - 2ND HALF - 2ND DECONTAMINATION

.	When notified by the dispatcher, turn on 15-6 air sparger and jacket cooling water to cool solution as it is being received.	15-6 Air sparger on_____ 15-6 Jacket water on_____
.	Beckman reading on 15-6	Meter_____ Factor_____
3.	Receive the second half of the product waste solution from 17-3 in 15-6 (TW from 17-3 approx. 12,250 lbs.)	Time start_____ Time end_____ 15-6 Wt. Ftr._____ Lbs._____
4.	Start 15-9 agitator and turn cooling water on 15-9 jacket.	15-9 Agitator on_____ 15-9 Jacket water on_____
5.	Beckman reading on 15-9	Meter_____ Factor_____
6.	Allow the 15-9 to 241 Bldg. jet to remain on air for 20 minutes after 15-9 is emptied to blow out the line, then jet 15-6 to 15-9. Shut off 15-6 jacket water and air sparger on completion of transfer.	Time start_____ Time end_____ 15-9 Wt. Ftr._____ Lbs._____ 15-6 Jacket water off_____ 15-6 Jacket water off_____ 15-6 Air sparger off_____
7.	Beckman reading on 15-6.	Meter_____ Factor_____
8.	Beckman reading on 15-9	Meter_____ Factor_____
9.	Add 1930 lbs. of 50% Sodium Hydroxide (Line Q) to 15-9 via 15-9A. Hold temperature below 50°C. by adjusting the rate of addition.	Time start_____ Time end_____ Lbs. added_____
10.	Agitate 15-9 for 10 minutes.	Time start_____ Time end_____
11.	Shut off 15-9 agitator and read Wt. Ftr., Temp. and Sp.Gr. when the Sp. Gr. meter becomes constant, then start 15-9 agitator.	15-9 Agitator off_____ 15-9 Wt. Ftr._____ Lbs._____ 15-9 Sp. Gr._____ Temp._____ 15-9 Agitator on_____
12.	Call dispatcher, give charge data and obtain permission to jet waste solution from 15-9 to 241 Bldg. (Tank X-110)	Time called_____ Time start_____ Time end_____
13.	Shut off 15-9 agitator and turn cooling water off 15-9 jacket.	15-9 Agitator off_____ 15-9 Jacket water off_____
14.	Beckman reading on 15-9	Meter_____ Factor_____

[redacted]

DECLASSIFIED

Date_____
Run No._____

NCENTRATION BLDG. WASTES - DISPOSAL OF A-4 BP

	Beckman on C-8	Meter_____ Factor_____
	Add 720 Gal. of water directly to C-8 via sanitary water line.	Gal. water added___ Time_____
3.	Receive approx. 1750 lbs. of A-4 BP into C-8, starting C-8 cooling water and agitator.	Time start jetting_____ C-8 agitator on_____ C-8 water on_____ Time end jetting_____
4.	Beckman on C-8	Meter_____ Factor_____
5.	Stop C-8 agitator when temperature falls below 50°C and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ C-8 Wt. Ftr._____ Lbs._____ C-8 Sp. Gr._____ Temp._____
6.	Start C-8 agitator.	C-8 agitator on_____
7.	Add 1250 lbs. on 50% Sodium Hydroxide to C-8 via C-8A (Line Q). Keep temperature below 50°C. Stop cooling water at end of neutralization.	Lbs. sodium hydroxide add_____ Time start_____ Time end_____ Max. Temp._____ Time water off_____
8.	Ten minutes after all sodium hydroxide is added, stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ Wt. Ftr._____ Lbs._____ Sp. Gr. _____ Temp._____
9.	Start C-8 agitator.	C-8 agitator on_____
10.	Check with Cell F Supervisor and receive into C-8 any F-7-WS or F-9-WS which may be ready to go to waste.	F-7-WS run No._____ F-9-WS run No._____ Time start jetting___ Time end___
11.	Stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ C-8 Wt. Ftr_____ Lbs._____ C-8 Sp. Gr._____ Temp._____
12.	Start C-8 agitator.	C-8 agitator on_____
13.	Read gauge on 361 tank. When level is satisfactory and with C-8 temperature below 50°C, jet C-8 to waste. When Wt. Ftr. pen reaches 0.2, stop agitator. When jet gases, stop jet.	Tank 361 level_____ Time start jet_____ C-8 agitator off_____ Time stop jet_____
14.	Beckman on C-8	Meter_____ Factor_____

Date_____
Run No._____

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CENTRATION BLDG. WASTES - DISPOSAL OF D-4 BP AND E-3 WS

Beckman on C-8 — Meter_____ Factor_____

Note that C-8 is empty — Time empty_____

3. Receive approx. 3500 lbs of D-4BP and approx. 18,500 lbs of E-3WS into C-8. Run agitator continuously after receiving first waste, except when reading Wt. Ftr. Use cooling water as necessary to bring temperature below 35°C.

WASTE	RUN NO.	C-8 WT.FTR.	LBS	TIME START	DATE	TIME END	TEMP.
D-4BP							
E-3WS							

4. Start C-8 cooling water — C-8 water on_____

5. Beckman on C-8 — Meter_____ Factor_____

6. Add 2700 lbs of 50% sodium hydroxide to C-8 via C-8A (Line 4) Keep temperature below 35°C. Stop cooling water at end of neutralization.
   Lbs. sodium hydroxide add_____
   Time start_____ Time end_____
   Max. Temp._____
   Time water off_____

7. Ten minutes after all sodium hydroxide is added, stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.
   C-8 agitator off_____
   Wt. Ftr._____ Lbs._____
   Sp. Gr._____ Temp._____

8. Start C-8 agitator — C-8 agitator on_____

9. Check with Cell F. supervisor and receive into C-8 any F-7-WS or F-9-WS which may be ready to go to waste.
   F-7-WS Run No._____
   F-9-WS Run No._____
   Time start jetting__ Time end___

10. Stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.
    C-8 agitator off_____
    C-8 Wt. Ftr._____ Lbs._____
    C-8 Sp. Gr._____ Temp._____

11. Start C-8 agitator — C-8 agitator on_____

12. Read gauge on 361 tank. When level is satisfactory and with C-8 temperature below 35°C, jet C-8 to waste. When Wt. Ftr. pen reaches 0.2, stop agitator. When jet gases, stop jet.
    Tank 361 level_____
    Time start jet_____
    C-8 agitator off_____
    Time stop jet_____

13. Beckman on C-8 — Meter_____ Factor_____

[redacted]

Date_____
Run No._____

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I ENTRATION BLDG. WASTES - DISPOSAL OF D-4 BP

	Beckman on C-8	Meter_____Factor_____
	Add 720 gal. of water directly to C-8 via sanitary water line.	Gal. water added_____Time_____
3.	Receive approx. 3500 lbs of D-4BP into C-8, starting C-8 cooling water and agitator.	Time start jetting_____ C-8 water on_____ C-8 agitator on_____ Time end jetting_____
4.	Beckman on C-8	Meter_____Factor_____
5.	Stop C-8 agitator when temperature falls below 35°C and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ C-8 Wt. Ftr._____Lbs._____ C-8 Sp. Gr._____Temp._____
6.	Start C-8 agitator	C-8 agitator on_____
7.	Add 50 lbs of 50% sodium Hydroxide to C-8 via C-8A (Line Q). Keep temperature below 35°C. Stop cooling water at end of neutralization.	Lbs. sodium hydroxide add_____ Time start_____Time End_____ Max. Temp._____ Time water off_____
8.	Ten minutes after all sodium hydroxide is added, stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ Wt. Ftr._____Lbs._____ Sp. Gr._____Temp._____
9.	Start C-8 agitator	C-8 agitator on_____
10.	Check with Cell F supervisor and receive into C-8 any F-7-WS or F-9-WS which may be ready to go to waste.	F-7-WS Run No._____ F-9-WS Run No._____ Time start jetting_____Time end_____
11.	Stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ C-8 Wt. Ftr._____Lbs._____ C-8 Sp. Gr._____Temp._____
12.	Start C-8 agitator.	C-8 agitator on_____
13.	Read gauge on 361 tank. When level is satisfactory and with C-8 temperature below 35°C, jet C-8 to waste. When Wt. Ftr. pen reaches 0.2, stop agitator. When jet gases, stop jet.	Tank 361 level_____ Time start jet_____ C-8 agitator off_____ Time stop jet_____
14.	Beckman on C-8	Meter_____Factor_____

[redacted]

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Date_____
Run No._____

CENTRATION BLDG. WASTES - DISPOSAL OF E-3WS

	Beckman on C-8	Meter_____ Factor_____
	With C-8 empty, receive E-3WS (approx. 18,500 lbs) into C-8, starting C-8 agitator and cooling water when Wt. Ftr. pen starts to rise.	Time start jetting_____ C-8 agitator on_____ C-8 water on_____ Time end jetting_____
3.	Beckman on C-8	Meter_____ Factor_____
4.	Stop C-8 agitator when temperature falls below 35°C and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ C-8 Wt. Ftr._____ Lbs._____ C-8 Sp. Gr._____ Temp_____
5.	Start C-8 agitator	C-8 agitator on_____
6.	Add 2650 lbs of 50% sodium hydroxide from C-8A (Line 2) to C-8. Keep C-8 temperature below 35°C. Stop cooling water at end of neutralization.	Lbs sodium hydroxide add_____ Time start_____ Time end_____ Max. Temp._____ Time water off_____
7.	Ten minutes after all sodium hydroxide is added, stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ Wt. Ftr._____ Lbs._____ Sp. Gr._____ Temp._____
8.	Start C-8 agitator	C-8 agitator on_____
9.	Check with Cell F supervisor and receive into C-8 any F-7-WS or F-9-WS which may be ready to go to waste.	F-7-WS Run No._____ F-9-WS Run No._____ Time start jetting____ Time end____
10.	Stop C-8 agitator and read Wt. Ftr. and Sp. Gr. when constant.	C-8 agitator off_____ C-8 Wt. Ftr._____ Lbs._____ C-8 Sp. Gr._____ Temp._____
11.	Start C-8 agitator	C-8 agitator on_____
12.	Read gauge on 361 tank. When level is satisfactory and with C-8 temperature below 35°C, jet C-8 to waste. When Wt. Ftr. pen reaches 0.2, stop agitator. When jet gases, stop jet.	Tank 361 level_____ Time start jet_____ C-8 agitator off_____ Time stop jet_____
13.	Beckman on C-8	Meter_____ Factor_____

[redacted]

Date_____
Run No._____

CENTRATION BLDG. WASTES - DISPOSAL OF F-7-WS (For use when C-8 is empty and F-7 WS Waste must be disposed of alone.)

1.	Beckman on C-8	Meter_____ Factor_____
2.	Check that C-8 is empty.	C-8 WT. Ftr._____ Lbs._____
3.	With supervisor's approval, receive approx. 2800 lbs of F-7 WS from F-7 into C-8.	Time OK to proceed_____ Time start_____ Time end_____ C-8 Wt. Ftr.____ Lbs._____
4.	Beckman on C-8	Meter_____ Factor_____
5.	Start C-8 agitator.	C-8 agitator on_____
6.	Read gauge on 361 tank. When level is satisfactory, jet C-8 to waste. When Wt. Ftr. pen reaches zero, stop agitator. When jet gases, stop jet.	Tank 361 level_____ Time start jet_____ C-8 agitator off_____ Time stop jet._____
7.	Beckman on C-8.	Meter_____ Factor_____

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Date_____
Run No._____

[illegible]ONCENTRATION BLDG. WASTES - DISPOSAL OF F-9-WS (For use when C-8 is empty and F-9-WS Waste must be disposed of alone.)

1. Beckman on C-8	Meter_____ Factor_____
2. Check that C-8 is empty.	C-8 Wt. Ftr._____ Lbs._____
3. With supervisor's approval, receive approx. 2,500 lbs. of F-9-WS from F-7 into C-8.	Time OK to proceed_____
	Time start_____ Time end_____
	C-8 Wt. Ftr._____ Lbs._____
4. Beckman on C-8	Meter_____ Factor_____
5. Start C-8 agitator.	C-8 agitator on_____
6. Read gauge on 361 tank. When level is satisfactory, jet C-8 to waste. When Wt. Ftr. pen reaches zero, stop agitator. When jet gases, stop jet.	Tank 361 level_____
	Time start jet_____
	C-8 agitator off_____
	Time stop jet_____
7. Beckman on C-8	Meter_____ Factor_____

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CHAPTER IX - WASTE DISPOSAL

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SECTION C - SEPARATIONS

CHAPTER X - INSTRUMENTS

In carrying out the separations process, the requirements of remote operation and health protection necessitate the use of many specially designed instruments in addition to an extraordinarily large number of the usual industrial types. In this chapter more emphasis is placed on the description and use of the special purpose instruments than on the more common industrial types, since it is assumed that the reader is in some degree familiar with ordinary temperature and pressure instruments and the like.

In the 200 Areas, the instrumentation is divided among the various buildings but, except for the analytical instruments, the preponderance is contained in the Canyon (221) Buildings, with smaller amounts of similar equipment in the Concentration (224) Buildings, and Isolation (231) Building.

With the exception of a few health monitoring instruments, the permanently located instruments of the Canyon Building are mounted in the operating gallery on 325 feet of gauge board divided into 18 groups - one for each of the canyon sections of 2 cells each except for two sections which contain no equipment. Each group comprises a number (1 to 9) of standardized instrument panel units $2\frac{1}{2}$ feet wide and $7\frac{1}{2}$ feet high, chosen and arranged to suit the instrumentation needs of that particular section.

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CHAPTER X - INSTRUMENTS

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RADIATION MEASUREMENT

The material handled in the Canyon and Concentration Buildings is radioactive to various degrees, depending upon the stage of the process. Provision, therefore, is made to measure the radiation intensity for two purposes: (1) the intensity existing in each process vessel is constantly recorded to give valuable process information, and (2) the inhabited parts of the buildings and site are constantly monitored to give information pertaining to the health hazards. In the latter category only those instruments which are also of interest to operating personnel are described.

Process Cell Chambers

Means are provided for measuring the radiation intensity from each process vessel in the Canyon and Concentration Buildings. This radiation information:

a) indicates the approximate activity of the solutions inside the tanks;

b) permits the operator to "see" the solutions enter and leave the vessels;

c) gives an indication of the effectiveness of the decontamination steps, and

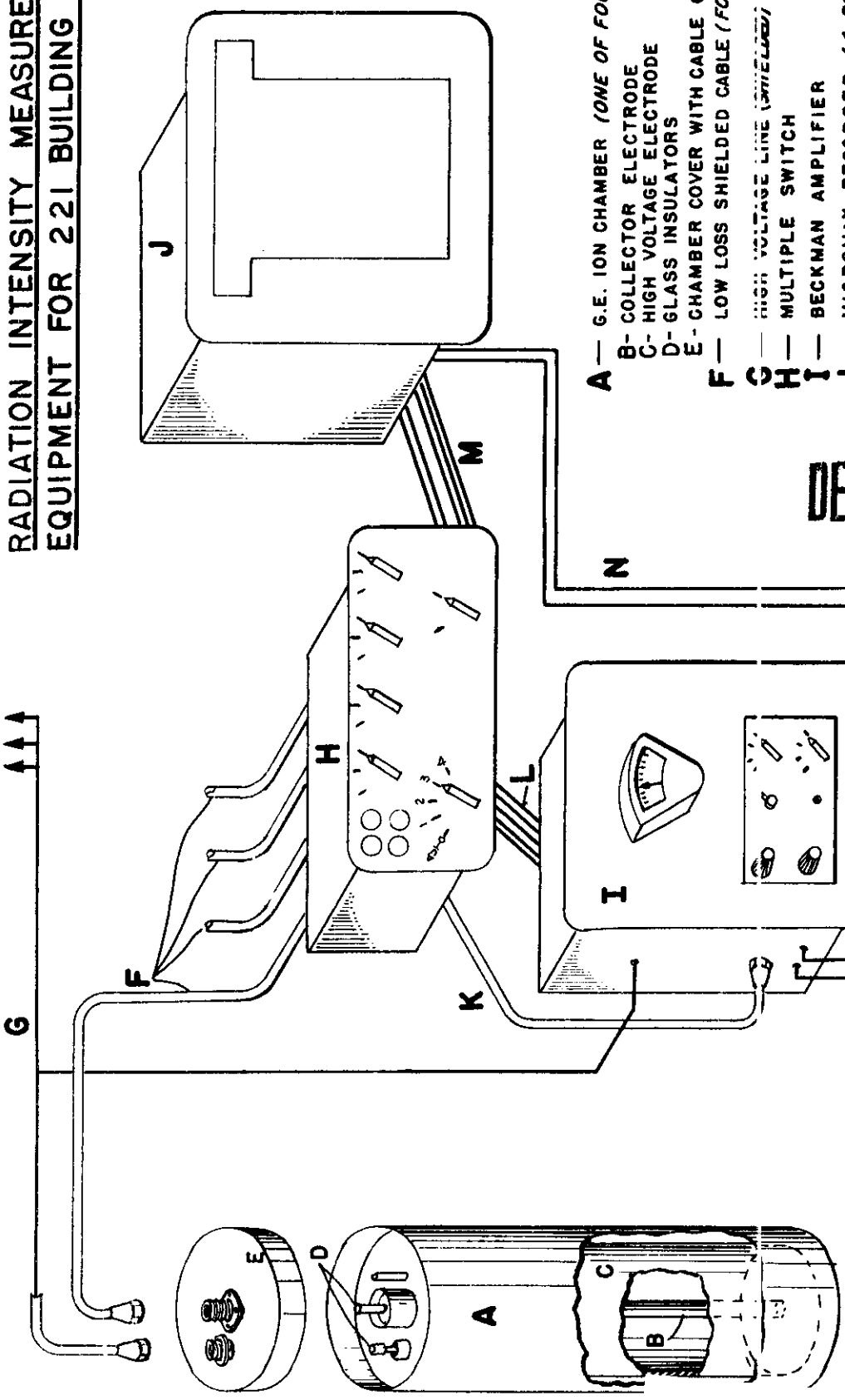
d) shows when, if ever, it is safe to approach the equipment in the cells for maintenance purposes.

The radiation sensitive elements in this application are ion chambers, made by the General Electric Company, which have the outward appearance of steel cylinders 5 inches in diameter and 27 inches in length. This cylinder contains a highly insulated center "collector electrode", and a concentric "high voltage electrode" near the outer shell. The entire cylinder is filled with argon gas at atmospheric pressure. Whenever gamma rays enter the chamber, some of their energy is expended in ionizing the argon. These ions are attracted to the electrode of opposite charge and cause a minute electric current to flow from one electrode to the other through a suitable external circuit. This current is proportional to the radiation intensity. A sketch of an ion chamber and its attendant equipment is shown in Figure 1.

Each section of the Canyon Building contains up to four G.E. chambers, one for each process vessel, located in 6-inch steel pipes embedded in the concrete cell partition. A "window", covered with a steel plate, is provided in the concrete opposite each chamber so that each chamber "sees" predominantly the vessel which it is intended to monitor. The collecting electrodes of each chamber in each section are individually connected to points on an automatic multiple switch which connects one point at a time to a Beckman micro-microammeter, a special amplifier capable of amplifying currents from 10^{-12} to 10^{-7} amperes to full scale meter deflection, depending upon which of six ranges is chosen. The reading of the Beckman amplifier is recorded on a

FIGURE 1

RADIATION INTENSITY MEASUREMENT EQUIPMENT FOR 221 BUILDING CELLS



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four-point Micromax recorder which is synchronized with the action of the multiple switch to print the radiation intensity at each chamber separately on the record chart. The switch can also be set to record continuously from any one chamber. The Beckman amplifier, multiple switch, and recorder for each section are grouped together on a single panel board.

In the Concentration Building an identical chamber-switch-amplifier-recorder system is used, but the location and mounting of the chambers is somewhat different. This is because of the different arrangement of the four tanks in each cell, and because of the lower activities present. In the case of each tank except the centrifuge, the chamber is contained in a heavy cast iron U-shaped shield which is supported from the concrete floor by steel legs. This assembly is placed near, but not touching the tank, with the open side of the shield facing the tank. The chambers are so oriented that the field of "vision" includes only the tank each is intended to monitor. The centrifuges are isolated by concrete partitions from the other tanks, so that the cast iron shields are unnecessary. The chambers for the centrifuges are mounted in niches located on the walls of the centrifuge enclosures.

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Health Monitoring Instruments

General Radiation Hazards - Intensity Measurements

The general activity level existing in the canyon deck area is monitored by special ionization chambers located beside each of the ten rear entrances to the canyon. These ionization chambers, known as H.M. (health monitoring) chambers, have fiber walls and contain almost no metal parts. The purpose of this type of construction is to obtain a radiation response comparable to that of the human body. Some of these H.M. chambers incorporate a movable door, or cover, which when open, allows beta particles as well as gamma rays to enter the chamber through thin plastic windows. Beckman amplifiers and automatic multiple switches, located outside the building under the rear stairs, transmit the chamber readings to four Micromax recorders located in the dispatcher's office in Building 271. The readings of these recorders are useful to the dispatcher in permitting access to the canyon.

An H.M. chamber installation with a Beckman amplifier and single point recorder is located in the operating gallery opposite section 9. Its purpose is to indicate immediately the presence of active solutions in the operating gallery piping. Such an occurrence is possible through improper operation of the transfer jet, spray, or sparger valves. This installation is equipped with an alarm.

Two H.M. chamber installations are located in the pipe gallery opposite Sections 4 and 13, with recorders located in the operating gallery. Their purpose is to indicate immediately the presence of active solutions in the pipe gallery, but these installations do not have alarms.

An H.M. chamber, located on the outside of the crane cab, operates a Beckman amplifier located inside the cab. This is for the crane operator's guidance when he wishes to leave the shielded cab. No recorder is used in this installation.

A 5-inch steel General Electric chamber is located in the railroad tunnel

at the upper corner in line with the operating gallery. The ion current is indicated on a Beckman meter located in the operating gallery, and repeated on another meter located in the dispatcher's office. This installation is useful to give assurance that no slugs were dropped or left in the tunnel after the loading operation.

Radiation Dosage Measuring Instruments - Victoreen Integrons

In addition to the radiation intensity measuring instruments described above, another type of instrument is in use which indicates the radiation dosage accumulated over a period of time. This instrument is the Victoreen Integron.

The Integron employs an ionization chamber whose collecting electrode is normally floating - free from any connection to the circuit - and well insulated. The walls of the chamber are coated with "Aquadag" and are at ground potential. The chamber is periodically (at least every 8 hours) automatically given a charge of about 250 volts. When the chamber is exposed to radiation, an ionization current flows in the collector circuit and causes its voltage to drop. Hence, over a period of time, the drop in the electrode voltage will be proportional to the integrated radiation (dosage) received by the chamber. Measurement of the collector voltage is accomplished without drawing any current from it by means of a motor driven rotating condenser; the highly insulated stator of the condenser is connected directly to the collector, while the rotor is connected to the input circuit of a vacuum tube amplifier. The capacity changes of the system caused by the rotation of the condenser cause an alternating voltage to appear across the input of the amplifier whose magnitude is proportional to the DC potential of the collector electrode. The onput of the amplifier is rectified and passed through a milliameter, whose reading expresses the charge of the chamber.

The rate of drift of this meter reading is indicative of the radiation intensity, and the instaneous reading indicates the accumulated dosage since the chamber was charged. The chamber is charged automatically every 8 hours, and also when 0.1 Roentgens of radiation is accumulated in less than 8 hours. A specially modified recorder is used to yield a record of the Integron reading.

Stack Gas Monitoring Instruments

The principal components of the stack gas which present a possible hazard are radioactive xenon and iodine. A sampling point is located at the stack 50 feet above the ground from which gas samples are drawn through the intensity measuring equipment. The gas is first passed through a soda ash solution scrubber to remove the iodine; the solution then is passed through a Saran tubing coil inside a special ion chamber which measures the activity of the iodine dissolved in the water. The gas, after passing through the scrubber, is first dried and then passed through a different ion chamber to measure the activity of the remaining components, principally xenon. The readings of two Beckman amplifiers (one for each chamber) are recorded on a two-point Micromax recorder. Since the stack gas-scrubbing water flow ratio is required to

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calculate the iodine concentration in the stack, it is measured both by rotometers and orifice meters which operate a recording ring balance meter.

Process Cooling Water Monitor

The cooling water discharge from the Canyon and Concentration Buildings join at a sewer man-hole, and then run to the retention pond in the waste disposal area. The water from either or both buildings may be continuously monitored as regards activity. At the manhole, water samples are continuously pumped through a small steel tank containing a glass Geiger-Mueller tube. Should any beta or gamma activity be present in the water, the GM tube will give rise to electrical impulses whose rate will be dependent upon the degree of activity present. These pulses are automatically collected and differentiated to give a meter reading expressing the rate of pulsing in counts/minute, which is proportional to the activity in the water.

The GM tube used in this instrument is a thin walled glass cylinder about 3/4 inch in diameter and 8 inches long. A sketch of this tube is shown in Figure 2. The central portion has walls thin enough to permit penetration by gamma rays, and all but the softest beta rays. The tube contains a very fine tungsten wire charged at high voltage; the glass wall is coated with silver and is at ground potential. The tube is filled with a mixture of argon and alcohol vapor at a pressure of 10 cm.of Hg. Whenever an ionizing particle enters, or is formed in the tube, an electrical discharge is initiated which causes the voltage of the central wire to drop until the discharge is quenched by the gas characteristics. This voltage drop constitutes a pulse which, after suitable amplification, is included in the counting rate.

The instrument used to amplify and differentiate the pulses or counts is an electronic circuit called a counting rate meter. The pulses received from the GM tube are first amplified, and then passed through a special "trigger" circuit which yields pulses of equal size regardless of the size of the input pulse. These pulses (one for each count) are impressed on a condenser which has a resistor connected across it. As the pulses are applied to the condenser, its voltage will increase until the current leakage through the resistor equals the current input from the pulses. Hence, the equilibrium voltage on the condenser is proportional to the rate at which the pulses are being received. The condenser voltage is measured by a suitable electronic circuit, indicated on a meter, and recorded on a recorder located in the Concentration Building.

In the Lag Storage (212) Buildings an instrument identical to that described above is used to monitor the activity of the water leaving the basin at any one of three points. It is also possible to sample the water in the tank cars used to bring the casks into the building. Appreciable activity in these waters indicate that some of the slugs have broken jackets.

Active Waste Detecting Instruments

An instrument has been provided for use in the Waste Disposal Area (241 Building) to detect any activity in the ground arising from leaks in the

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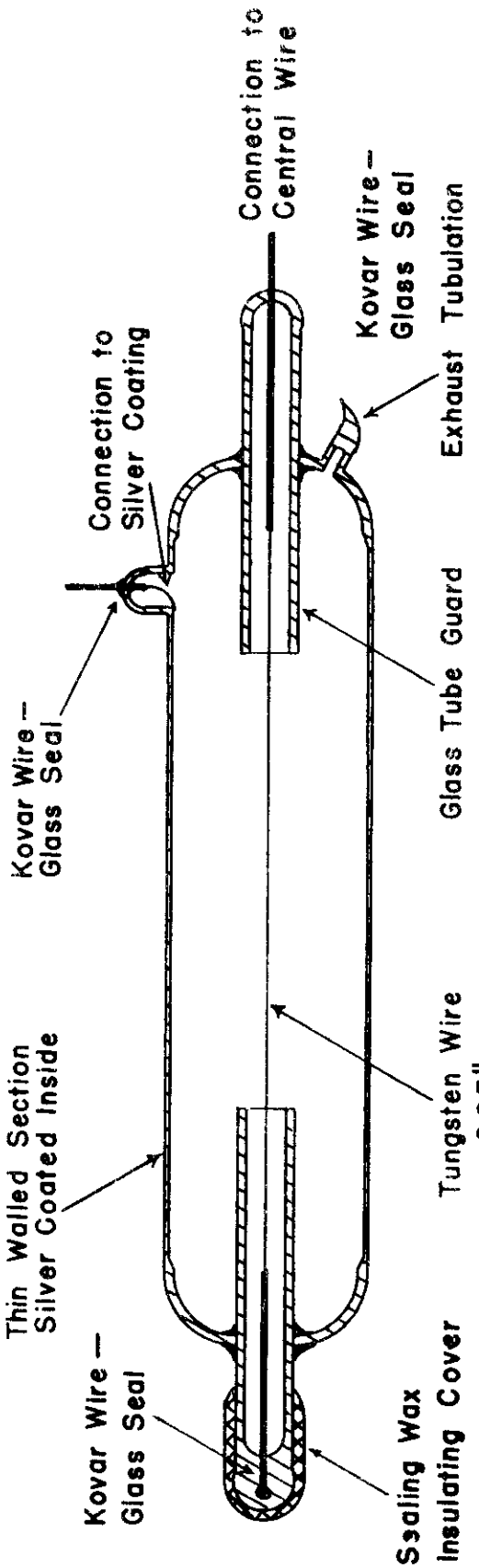
FIGURE 2



GLASS G-M TUBE

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twelve 75-foot diameter waste storage tanks. A total of seven test wells 6 inches in diameter and 150 feet deep have been drilled in the vicinity of each 241 Building area. One well is in the center of the group, one at either end, and two on each side. The device described in the following paragraph may be lowered into any well to survey the gamma activity of the surrounding earth.

The sensitive element of this device comprises a glass GM tube enclosed in a rugged waterproof brass case 5 inches in diameter and about 14 inches long. A one tube "cathode follower" circuit is also included in this case in order to convert the GM tube pulses to a form capable of being transmitted through the necessarily long cable to the earth's surface. The GM tube assembly is supported by a light chain and two cables, - one for the high voltage to the GM tube, the other containing the filament and plate voltages, and the signal lead.

The rest of the equipment comprises a portable rubber tired cart (See Figure 3) supporting:- (1) a reel for handling the chain and cables and (2) a scaler, timer and mechanical register. The scaler is a self contained electronic unit which (1) supplies high DC voltage to the GM tube, (2) amplifies the GM pulse, and (3) reduces the number of pulses by a factor of 64. The latter is necessary in order to obtain a counting rate slow enough to operate a mechanical recorder. The mechanical recorder is a magnetic-mechanical device which records the number of counts leaving the scaler. This figure times 64 and plus the number of counts "left in" the scaler (shown by neon interpolation lights) is equal to the number of GM pulses formed during the test.

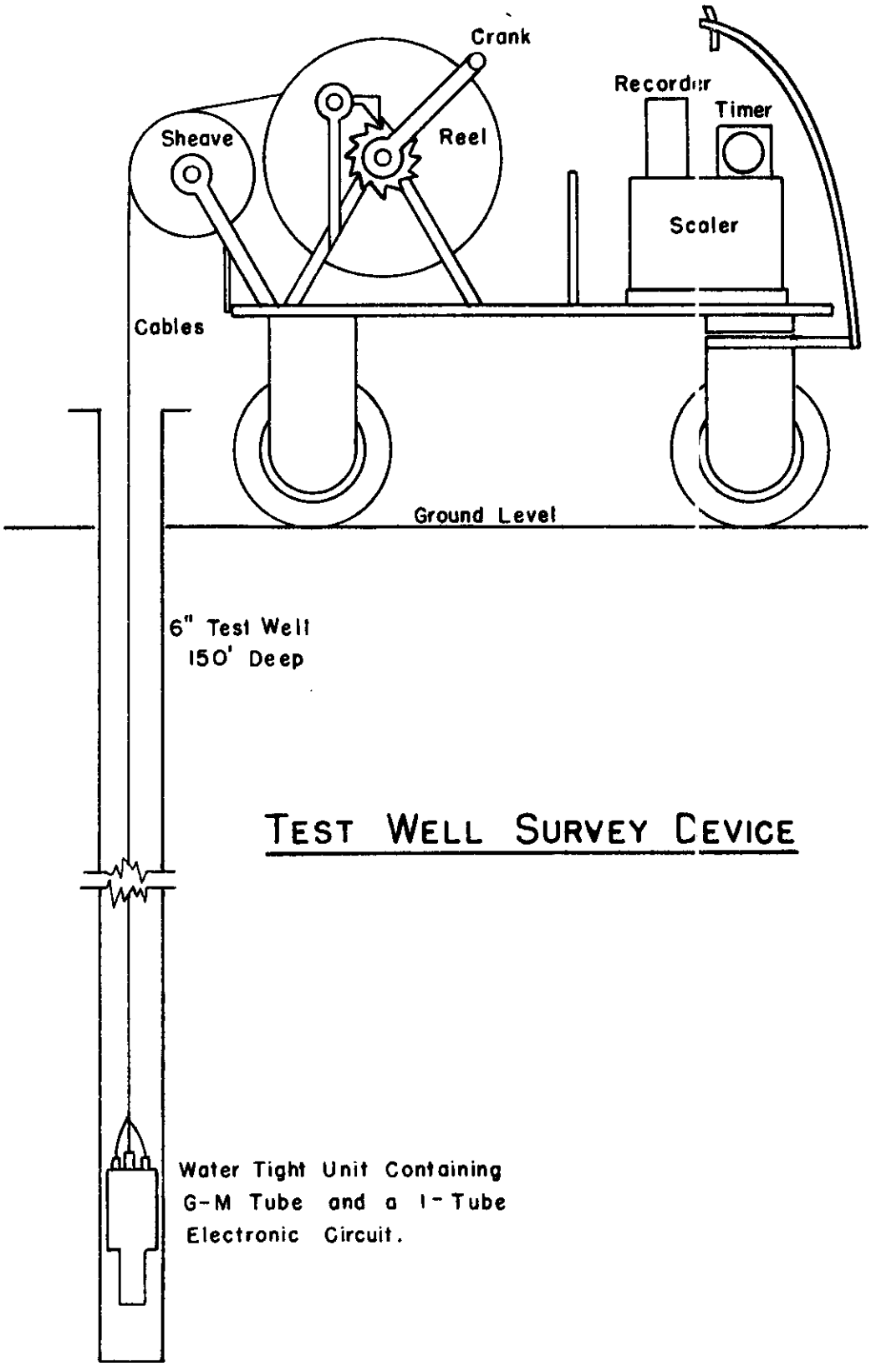
Another instrument used in the 241 Building area is a device to show the depth of settled solids in the 75-foot tanks. The fact that any solids settling out will carry most of the activity makes possible the use of a radiation sensitive sounding device. A 3-inch closed end pipe extending to the bottom of the tank is installed in one of the 4-inch tank openings. A small ion chamber may then be lowered down this pipe, and a sudden increase in ion current (indicated on a micro micro-ammeter) indicates that the chamber is passing through the interface between the sludge and the clear liquid.

VISUAL AND AUDIBLE AIDS

Centrifuge Wobble Meters

Each of the eleven centrifuges in the Canyon Building and the five in the Concentration Building is equipped with a wobble meter. The purpose of this instrument is to detect any unbalance of the centrifuge bowl due to uneven distribution of the slurry. Severe wobbling at high speed could, of course, damage the bearings or other mechanical parts. A magnetic pick up device, mounted on the centrifuge, generates modulated 60-cycle signal whose amplitude of modulation is proportional to the centrifuge shaft displacement. This signal, after amplification, is impressed on the vertical deflection plates of a cathode ray tube. The horizontal deflection plates are connected

FIGURE 3



TEST WELL SURVEY CEVICE

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to a 60-cycle source. The result is that the operator sees a closed pattern which ideally expands from a straight line to an ellipse and back. The magnitude of this fluctuation is proportional to the wobble of the centrifuge shaft.

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Cell Microphones

All process tanks are equipped with contact microphones. The operator, by listening to loud speakers connected to these microphones, may ascertain to some degree what is going on in the tank. He should be able to tell if the agitator is operating properly and hear solutions being loaded or unloaded. Operating experience is expected to increase the usefulness of this instrument.

The overall noise inside of most cells of the Canyon and Concentration Buildings is monitored by ordinary space microphones located within each cell. Any departure from the type of noise shown by experience to be characteristic of the cell indicates the possibility of some mechanical trouble.

The amplifiers for the contact and space microphones are located in the operating galleries opposite the gauge boards. They are arranged in relay racks containing up to four amplifiers with their power supplies. Some amplifiers handle more than one microphone; the required switches are located on the gauge boards. Loud speakers, one for each amplifier, are located on top of the gauge boards. The sound volume may be controlled by gain controls located on the amplifier and on the gauge board.

Crane Optical Aids

The crane cab in the Canyon Building is separated from the canyon by a concrete partition to protect the operator from radiation hazards. For this reason since the operator cannot directly watch the operation of the crane, recourse must be made to optical aids.

Two large periscopes, one on either side of the crane bridge permit the operator to view the crane hooks, impact wrenches and cell equipment with clarity. Although the field of vision is rather limited (to gain magnification), scanning may be obtained at the operator's discretion by electric motors which "telescope" the periscope across the canyon, and rotate it on its axis. The eyepieces are automatically synchronized with the viewing direction of the objective ends of the periscopes so that the operator is always looking in the true direction.

Large negative lenses have been installed on the outboard side of each periscope objective so that when the objective is rotated in this direction, a tremendously increased field of view is obtained. This allows the operator, in one glance, to get a general view of the canyon to see whether or not all cover blocks are in place and to see if anyone is on the deck.

A television camera also is installed under the near, right hand corner

of the crane bridge. The viewing angle of this instrument is varied by electric motors under the operator's control which rotate the camera on horizontal and vertical axes. The "picture" picked up by the camera is transmitted by wire to the crane cab where it appears on the screen of a cathode ray tube. Although the television lacks the definition of the periscope, it has a broader field of view, is not as tiring to the eye, permits quicker scanning of the situation, and affords an additional view from another angle.

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Portable Periscopes for Viewing the Interior of Centrifuges

Two portable periscopes have been provided for inspecting the interior of the centrifuges. One of these instruments is 2-3/4 inches in outer diameter and 48 inches long, while the other is 1-1/2 inches in diameter and 31 inches long. The larger diameter instrument is suitable for viewing the interior of both the 40-inch and 26-inch centrifuges, while the smaller one is suitable for viewing the interior of either the 26-inch or 12-inch centrifuges.

The 2-3/4 inch diameter periscope has an outside brass case and is provided with a scanning head and self contained illumination enclosed within a watertight glass window. The optical train consists of six basic elements; the scanning prism, an erecting prism, an objective, outer erector lens, inner erector lens and eyepiece. (See Figure 4) The scanning prism, erecting prism, and objective manufactured by the Kollmorgan Optical Company, are mounted as a unit. The objective has a focal length of 2-1/2 inches. The scanning prism has a sweep of 90 degrees. The erecting prism is employed to keep the image of the system from turning on its side as the scanning prism moves. The outer erector is about 1-1/2 inches in diameter and has a focal length of 23.8 cm. The inner erector has a diameter of 2-1/4 inches and a focal length of 29.9 cm. Eyepieces were provided to give 1, 1-1/2, and 2-1/2 times magnification. Scanning is accomplished by rotating the prism through as much as 90° sweep and rotating the instrument through 360°. The prism is rotated by a gearing arrangement which in turn connects to a push-pull rod. This push-pull rod runs the length of the instrument and is moved by a rack and pinion action at the eye end of the viewer. Lighting is provided by two special general electric 6.4 volt, 21 candle power bulbs.

The 1½ inch diameter instrument has an aluminum exterior. The head on this instrument is of the fixed 90° viewing type, but includes self illumination consisting of a 32 candle power lamp within a watertight glass window. Scanning with this instrument may be accomplished by rotation of the instrument itself. The periscope has an angular magnification of unity and a field of view of 30°.

INDUSTRIAL INSTRUMENTS

pH Measurements

It is desirable to make occasional pH measurements chiefly of waste

FIGURE 4

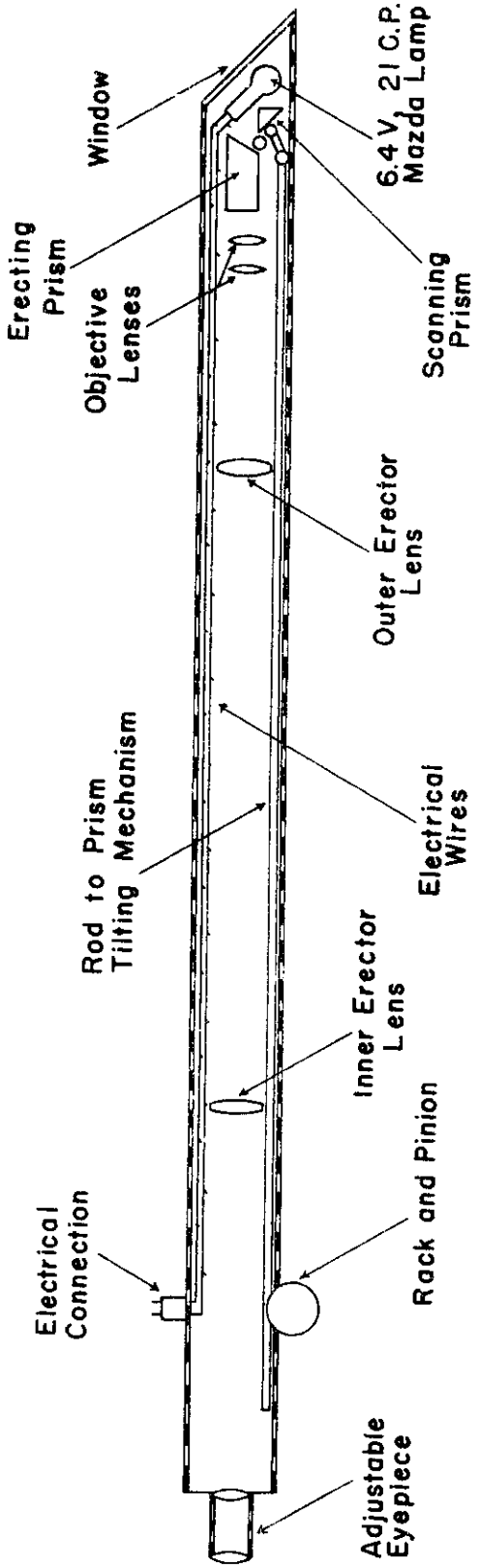

DIAGRAM OF 48" PERISCOPE.

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liquors, in the Canyon, Concentration, and Waste Disposal Buildings. In the Canyon Building one portable pH instrument is considered sufficient. Samples for determination of pH are secured by means of special samplers, also used to secure samples for analysis. Whenever a pH determination is desired, a special glass-calomel electrode is inserted through an opening in the sampler provided for this purpose, and the pH reading is made with the portable instrument.

Temperature Measurements

Temperature measurements are made on all process solutions in the Canyon Building. In the case of the dissolvers, the temperatures of the off-gas, the condenser water and the cell itself are also recorded. In all cases the temperature sensitive elements are 100-ohm nickel resistance thermometers located in special wells at the desired point. The temperatures are all recorded on L&N recording meters with 0-100 °C ranges. The recorders are of the single, two or four point type depending upon the application. The L&N instrument is an automatic Wheatstone bridge circuit with two slide wires so arranged that changes in the resistance of the leads due to temperature effects do not affect the reading. The current for this bridge circuit is supplied from an AC rectifier built into the instrument. Unlike the potentiometer-thermocouple type of instrument, no standard cell or dry cell is required.

Pressure Measurements

Standard types of dial gauges are used to indicate the pressures of process steam and air, and instrument air. Combination pressure-vacuum gauges are used on the steam jets which transfer the solutions from one tank to another and on spargers and spray distributors. These gauges indicate the steam pressure on the jet, but are also capable of showing if a vacuum is created on the line due to condensation after the jet has been turned off. This vacuum is undesirable since active solution may be drawn into the operating gallery.

The ventilation system of the Canyon Building is so designed that the cells are maintained under a slight vacuum with respect to the inhabited parts of the building. This is to prevent any radioactive gases from seeping into the operating gallery or deck area. In order to indicate whether this vacuum is being maintained, each cell is equipped with an inclined draft gauge having a range of 0-1 inch of water. The inclining feature of this instrument results in a magnification of the normal vertical pressure head so that more accurate measurements of the cell pressure can be made. Similar draft gauges are located in the operating gallery to indicate the differential pressure between that gallery, and the canyon above the deck, the outside of the building and several other points within the 221 and 271 Buildings.

Liquid Level and Specific Gravity Measurements

The basic principle involved in the measurement of liquid level in process

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tanks is that the liquid depth is proportional to the hydrostatic pressure at the bottom of the vessel divided by the specific gravity of the liquid. Two factors, therefore, must be measured:- a) hydrostatic pressure or "weight factor", and b) specific gravity.

The weight factor is found by measuring the differential pressure between the air space in the vessel and a point in the liquid near the bottom of the tank. The specific gravity is determined by another differential pressure measurement made at two points in the liquid at a constant vertical distance apart. If air is slowly bubbled through a pipe terminating at one of these points, the air pressure necessary to allow air to escape from the bottom will equal the hydrostatic pressure at that point. This air pressure (or differential pressure between two points) can easily be measured by a manometer or any other suitable pressure measuring device, such as that described below.

The differential pressures involved in the two measured factors are impressed upon the two rings (No. 1 and No. 2) of the Dual Ring-Balance meter. (See Figure 5) Both rings are enclosed in the same case and activate two pens on the same circular recording chart. Each hollow ring is suspended on a knife edge above the center of gravity, as shown. The free space in the ring is divided into two parts by a metal bulkhead at the top and by a liquid seal at the bottom. Flexible tubing connects each chamber with one of the pair of bubbler lines. A pressure difference in these lines gives rise to an equal differential pressure between the two chambers of the ring. This causes an unequal pressure on each side of the bulkhead and a shift in the liquid seal. Since the liquid seal itself can exert no rotational force on the ring, the differential pressure on the bulkhead is all that need be considered in this analysis. The pressure difference on the bulkhead causes the ring to rotate on its axis until this force is equalled by the restoring force of the unbalanced ring. A pen motion, attached to the ring through a cam, records the differential pressure on a circular chart. Calibration is effected by adjusting lead weights at the bottom of the ring.

The ring balance instruments have weight factor ranges from 0-10 arbitrary units, and the sensitivity may be adjusted to fit nearly any size tank. Specific gravity ranges are 1.0 to 1.4, or 1.0 to 2.5, depending upon the application.

Simple manometers are used for liquid level measurements in some applications such as centrifuges, Cell 4 and in the Semi-Works of Building 221-T. No specific gravity measurement is made in this case; a single dip tube supplied by a single sight bubbler gives a single hydrostatic pressure which is indicated by the manometer.

Flow Meters

Indicating flowmeters are used in the dissolver cells to record the flow of cooling water through the condensers and vessel coils. These are Foxboro instruments which indicate the flow by measuring the pressure drop across an orifice.

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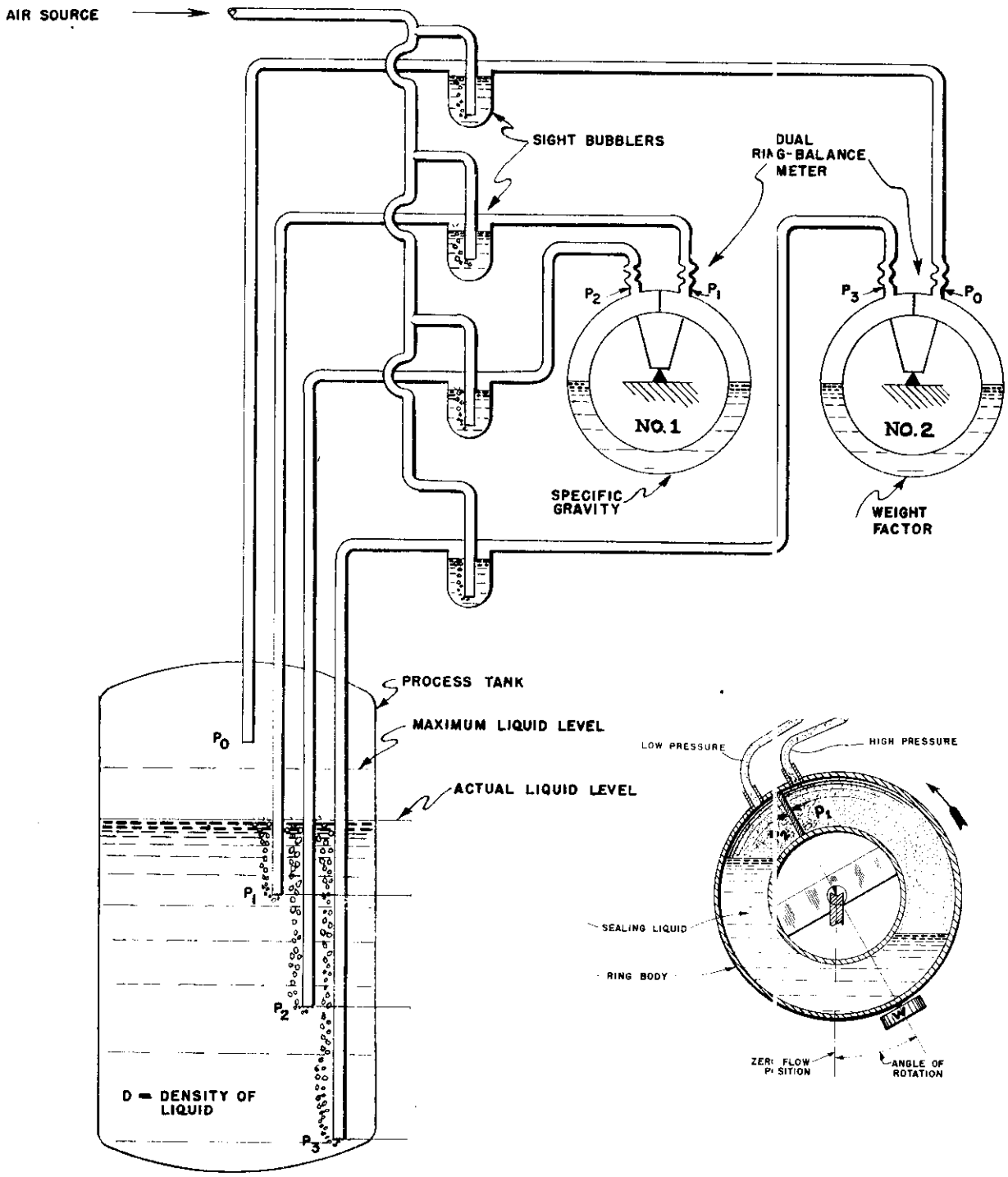
FIGURE 5

LIQUID LEVEL MEASUREMENT WITH SPECIFIC GRAVITY AND WEIGHT FACTOR



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Centrifuge Instruments

All centrifuges are equipped with electric tachometers to indicate the speed of rotation. The tachometer comprises two parts:- (1) a small AC generator mounted on the centrifuge shaft, whose voltage output is proportional to the speed, and (2) an indicating tachometer which is actually a DC Voltmeter (using a rectifier) calibrated in rev./min.

An automatic control attached to the centrifuge tachometers prevents inadvertent reversal of the centrifuge in case of prolonged "plugging". (When a centrifuge is plugged, the power phases to the motor are reversed so that the motor torque is applied in the reverse direction. This causes the speed to decrease rapidly, but if the plug control is held in too long the centrifuge will actually reverse its direction of rotation resulting in serious damage to the centrifuge.) A photo tube and lamp are mounted in a small case on the face of the meter in such a manner that the light is reflected to the photo tube by a mirror when the meter needle is up scale, but the light beam is broken when the needle is at 0 rev./min. The photo tube controls an electronic circuit which breaks the plugging circuit to the motor. As a result when the speed is reduced almost to 0 rev./min. during plugging, the light beam is interrupted, the power to the motor is switched off automatically so that motor reversal cannot occur.

The motors on all agitators and centrifuges are connected through ammeters so that the operator may have an indication of the load on these machines, and determine to some degree if they are operating properly.

ANALYTICAL INSTRUMENTS

The unique nature of the product and the intermediate process substances require analytical equipment of a very special nature. Since most of the substances involved are radioactive, nearly all analytical procedures involve quantitative measurements of the various types of radiations. Alpha, beta, and gamma rays are the radiations with which the analytical work is concerned. Each can be quantitatively measured by various types of instruments.

Alpha Counting Sets

Alpha particles, which are heavy atomic fragments with a double positive charge, are the characteristic emission of the product. Hence, satisfactory instruments to count alphas are extremely necessary. Three types of alpha counting instruments are in use at H.E.W.

The Standard Alpha Counting Set, one of the first types of satisfactory alpha counting chambers, comprises (1) a counting chamber containing a preamplifier, (2) alpha amplifier, and (3) scaler and register. The chamber consists of a brass or steel cylinder which contains a pair of parallel plates in the bottom between which the sample disk is placed. The lower plate is charged with high voltage, while the upper plate is connected to the grid

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of a vacuum tube in the upper portion of the chamber. Alpha particles emitted from the sample ionize the air between the plates; the positive ions thus formed are attracted to the upper plate causing an electrical pulse in the first vacuum tube. This pulse is subsequently amplified and counted by the scaler and mechanical recorder. The alpha amplifier circuit contains a pulse height selector circuit which passes the strong alpha pulses, but stops the smaller beta pulses. The Standard Alpha Set is very reliable, but has several limitations:- (1) the counting losses are rather high at high counting rates because of the slowness of the circuit, (2) it will not tolerate a very high beta background, and (3) it is quite subject to microphonic disturbance.

The Low Geometry Vacuum Alpha Set is designed to count alpha samples which are too active to be counted on the Standard set, but the percentage of alpha particles that cause counts (geometry factor) is greatly reduced by increasing the distance between the sample and the collecting plate and making the alphas pass through one of several small holes before reaching the collecting plate. Since the alpha range in air is extremely short, most of the alpha path is under vacuum. The evacuated portion is separated from the collection plate (surrounded by air to form ions) by a thin mica window through which the alphas can pass. Although the Low Geometry Set permits the use of "hotter" samples, the beta background and microphonic limitations of the Standard type are still present.

The Proportional Alpha Counter (Simpson Counter) is somewhat more complicated than the two instruments described above, but its different principal of operation eliminates to a large extent the limitations of the Standard and Vacuum types. The counting chamber is a horizontal brass cylinder with a slide opening at the bottom by means of which the sample is introduced. A very fine tungsten wire, charged at high voltage is stretched along the inside of the chamber parallel to its axis. A small, continuous flow of methane gas keeps the inside of the chamber filled with this gas. Ionizing particles arising from the sample cause rapidly quenched electrical discharges in the counter, whose magnitudes are proportional to the energy of the ionizing particles; hence the name "proportional" counter. The electrical pulses thus formed are first amplified linearly then passed through a pulse height selector where the small pulses due to betas are eliminated. The alpha pulses then, after passing through a scale-of-128, actuate a mechanical recorder. Because of the extremely sharp pulses formed by the Simpson counter, extremely high counting rates are possible. For the same reason, an extremely high beta background can be tolerated. A low geometry-vacuum attachment can be used with Simpson counters, if it is desired to count still "hotter" samples.

Beta and Gamma Sets

Beta particles emitted by chemical samples are counted with a special type of GM tube called a mica window tube. It operates on the same principle as the glass GM tube previously described in connection with certain health instruments, but is different in construction. The mica window tube comprises a copper cylinder closed at one end and flanged at the other. A thin mica window is waxed over the open flanged end; a stiff tungsten wire enters

through an insulator in the closed end and extends nearly to the mica window. The end of the wire is covered with a glass bead. The tube is filled with a mixture of argon and alcohol vapor at low pressure. When arranged to count beta samples, the tube is mounted above an aluminum rack in which the samples are placed for counting. Also mounted on this rack is a "Neher-Harper" quenching circuit which improves the characteristics of the tube. The entire assembly is contained in a lead shield to reduce the background count. A standard scaling and recording circuit is used to register the number of counts.

Gamma emission can be measured by the same equipment if metallic screens are placed between the sample and the GM tube to screen out the betas.

Gamma activity of liquid samples can be measured with a specially constructed ion chamber. This chamber is made from 10-inch steel pipe about 12 inches long. The top is closed with a welded plate containing a sample tube. A highly insulated collecting electrode, surrounding the sample tube is brought out through a removable flange at the bottom of the chamber. The chamber is filled with argon under 600 lbs./sq.in. pressure to increase the chamber's ionizing sensitivity. Lead, 4 inches thick, surrounds the chamber on all sides to decrease the effect of cosmic rays. The shell if charged to a positive potential of 180 volts. When a measurement is made, the collecting electrode, starting at ground potential, acquires a positive potential due to ionized gas particles, at a rate proportion to the gamma activity of the sample. This rate of drift of potential of the collecting electrode is observed with an electrometer and stop watch.

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SECTION C - SEPARATIONS

CHAPTER XI - SHIELDING PROVISIONS IN THE SEPARATION PLANT

The fundamental principles upon which shielding from radioactive radiation is accomplished, as well as a discussion of the shielding requirements in the Separation Plant, are discussed in Chapter I. This outlines briefly the provisions which have been included in the design for the purpose of shielding the operating personnel from radioactive radiation.

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CHAPTER XI - SHIELDING PROVISIONS IN THE SEPARATION PLANT

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DESIGN BASIS OF SHIELDING

The fact that the decontamination factors desired through the separations process are of the order of 10^7 to 10^8 makes it desirable to break the process down into several stages which can be handled with progressively lighter shielding. During the storage of new metal from the 100 Areas and the extraction and decontamination portions of the process very heavy shielding against gamma rays is required. The concentration process requires concrete shielding of little more than ordinary structural thickness, while the final purification and isolation process is carried out with essentially no shielding against gamma rays, but with great precautions against product hazards.

In the early stages of the separation process, concrete shields of the order of 5 feet in thickness are required, and these are designed to serve also as structural elements of the building. Wherever possible, a large factor of safety in shielding has been provided at the cost of a relatively small additional thickness of concrete. The 221 Building, for instance, is shielded so that metal freshly discharged from the pile could be handled without having the gamma dosage rate in any working area exceed about 1 mr/hr, or roughly one-tenth of the tolerance dosage rate. Hazards against which the shields offer no protection are those of suckbacks, spills, downdrafts from the stack, and product contamination. Such hazards are detected by various monitoring devices, and appropriate corrective action is taken.

METAL STORAGE BASINS

Metal from the piles is stored in the Metal Storage Basins for a period of the order of 40 days before being sent on to the dissolvers. This decay period permits substantially all of the $_{93}U^{239}$ to decay into product, so that the activity of this isotope does not exceed 20 millicuries per batch after extraction and two decontamination cycles. It also effects a substantial reduction in the amounts of radioactive iodine and xenon which are vented to the stack during dissolving.

Metal is handled and stored in stainless steel buckets which hold approximately one-half ton of slugs. The buckets are transported in shielded casks (Fig. 34 Chapter II). The walls of the cask are of lead, 12 inches thick, which is sufficient to reduce the gamma intensity outside the cask to something of the order of 1 mr/hr. New casks must be carefully monitored, in order to determine whether or not blowholes in the lead casting are present which may permit dangerous amounts of radiation to leak out. In the event of blowholes in the casting these should be repaired before the cask is put in service. The cask lid fits into a staggered recess so that there is no direct path for the escape of radiation. Curved pipes in the top and sides of the cask allow water to circulate and cool the metal by thermal siphon action without permitting appreciable radiation leakage. The lid is locked in place by a pair of sliding bars driven by racks and a pinion. This locking feature minimizes the danger of spills during handling or

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transportation of the cask.

Casks are transported between areas on a special railroad car. Two water tanks, each capable of carrying one cask, are mounted on each car. The function of the water is primarily to keep the metal cool. The shielding effect of the water added to the cask is negligible. The heat capacity of the water in the tank car is sufficient to limit the initial temperature rise of a bucket of freshly discharged metal to a value no greater than 2 °C/hour, and heat losses to the surroundings are such that under the worst conditions the water will not boil. In contrast, the cask and bucket alone heat at a rate of the order of 5 to 15 °C/hour, and boiling of the water retained in the cask can occur with freshly discharged metal, but not with aged metal.

In case it becomes necessary to handle damaged slugs, enough metal may go into solution to raise the radiation intensity outside the tank car above the tolerance level. Replacement of the water and decontamination of the car would then be required. Evidence so far indicates that this hazard is not likely to occur.

The arrangement of the 212 Building is shown in Fig. 32 in Chapter II. The principal features are the transfer pits, in which the buckets of metal are removed from the casks and transferred to a monorail system, and the storage area, where they are stored under water. Radiation hazards during the transfer operation are minimized in the following way: the locked cask is picked up from the railroad car and swung over the transfer pit, where the lid is unlocked. The cask is then lowered into the water, being guided by rails at the sides of the pit. At a point where the cask is under about ten feet of water, the four projecting arms of the lid are caught on steel supports. The cask and its bucket of metal then move down and forward to the bottom of the pit. At this point the bucket is lifted from the cask by means of a yoke, transferred to the monorail system, and moved to storage. A minimum depth of about 16 feet of water is maintained over the buckets at all times. The procedure is reversed to remove aged metal for shipment to the separation plant.

The depth of water in the storage pool provides adequate shielding for the worst conceivable condition, namely, the case in which the entire pool is filled with metal freshly discharged from the piles. There is no danger of a self-sustaining chain reaction getting started in the stored metal, regardless of how the buckets are arranged. The mass of water is great enough and is changed frequently enough so that heating effects are negligible. The radiation from undamaged slugs does not induce activity in the water, so that the appearance of activity in the water can only be caused by damaged slugs. In this case the offending slugs must be located and moved to special storage in the Canyon (221) Building. As previously indicated, such failures are unlikely to occur.

Since the lead cask and the water in the pool provide adequate shielding, the building itself is of conventional construction with 8-inch thick concrete block walls and roof constructed from 2-3/4 inch thick cement tile with 1 inch of insulation. This construction provides no protection against an accident in which a cask is broken open at ground level or the water

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is drained from the storage pool. In either of these improbable events, the affected building would be unapproachable. The 212 Buildings are spaced half a mile apart so that in the event of such an accident in one building, the remaining ones could still be used.

CANYON BUILDING

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In the 221 Building, aged metal is dissolved and subjected to an extraction and two decontamination cycles. The fission product activity is thereby reduced by a factor of 10^5 or more. Until this degree of decontamination is achieved, it is necessary to carry out the process in heavily shielded equipment which is operated and maintained by remote control. The necessity for remote maintenance places a high premium on uniformity and interchangeability of equipment, in comparison with which any savings that might be obtained by the use of lighter shielding at the low activity end of the separation line are unimportant. Consequently, the shielding is uniformly heavy throughout the length of the separation line.

To prevent a mishap to any single 221 Building from rendering the others inoperable, these buildings are separated by a minimum distance of 1 mile.

A typical section of a 221 Building is shown in Fig. 35 in Chapter II. In general, the thickness of the structural members which form the shields has been chosen so that the gamma ray dosage rate is less than 1 mr/hr in any populated area when a bucket of fresh metal is present in the building. Aged metal, which is normally the only active material brought into the building, is only about one-tenth as active as fresh metal. Under ordinary conditions, therefore, the gamma leakage through the shields is of the order of 1% of the tolerable dosage rate of 12.5 mr/hr.

The walls of the canyon are 5 feet thick up to a height of 26 feet above deck level, the walls are 3 feet thick and the roof, for structural reasons, tapers from a thickness of four feet at the sides to a thickness of three feet at the center. The lower part of the wall shields personnel outside the building or on adjacent buildings from direct gamma radiation at times when metal is being brought into the canyon. The upper part of the wall and the roof eliminate "sky shine" or radiation scattered from the air above the canyon, which would otherwise produce a hazard in the neighborhood of the building.

The various process vessels are located in a row of cells which are sunk below deck level and normally shielded by concrete cell covers 6 feet thick. Each cell cover is built up of four concrete blocks to facilitate handling. The recess into which the cover fits has stepped sides, and the adjoining faces of the individual blocks are also stepped to eliminate narrow beams of radiation which otherwise would escape from the cells. Active solutions are transferred from one cell to another by way of a pipe trench running the length of the canyon. The pipe trench is covered by stepped concrete covers, 4-1/2 feet thick, similar to those used on the cells.

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Significant amounts of radiation are present in the canyon only when the covers have been removed from cells or pipe trench, or when a bucket of metal is being brought in. At all other times the canyon may be entered for the purpose of taking samples or maintaining such equipment as is accessible.

Labyrinths in the 5-foot concrete wall on the pipe trench side of the canyon provide access to the deck. The labyrinth walls are 5 feet thick as far as the second bend. From this point to the outside entrance, the intensity of the scattered radiation is low enough so that walls 1 foot thick are adequate.

A special feature of the 221-T Building is the "Head End". This is a special pair of cells used for development work and located at the head end of the canyon. The Head End is separated from the canyon proper by a concrete barricade 5 feet thick and 13 feet high. The purpose of this barricade is to shield personnel on the Head End deck from direct gamma radiation in case of an accident in the main canyon, and vice versa.

The wall which shields the various galleries is 10 feet 10 inches thick opposite the pipe gallery, 7 feet thick opposite the operating gallery, and 5 feet thick opposite the crane cabway. Pipes which run through these walls are curved to minimize radiation leakage. The pipe gallery handles only inactive materials (active process solutions are transferred in the pipe trench on the other side of the canyon) and the piping is designed to minimize the chance of sucking active solutions back into the pipe gallery. The few suckbacks that have occurred have been promptly detected and remedied. The roof of the operating gallery, which also forms the floor of the crane cabway, is 4 feet thick. This thickness is more than adequate inasmuch as this floor is exposed only to radiation which has been scattered from the canyon ceiling. The cabway is entered through a row of labyrinths on the operating gallery side of the building.

The crane cab is shielded from direct gamma radiation by the concrete parapet behind which it runs. Protection against scattered radiation is afforded by the steel shielding of the cab, which is 4-1/2 inches thick on the top and upper part of the sides, 3 inches thick on the lower part of the sides, and 1-1/2 inches thick on the bottom. The cab is entered through steel doors 3 inches thick. Steel is used in this structure rather than lead because of the danger that lead might eventually sag and open up crevices through the shield. The shielding is adequate to hold the radiation intensity in the cab below 1 mr/hr for the extreme case when a bucket of fresh metal is raised as high as the crane will lift it. The periscopes are brought in through a labyrinth on the front wall of the cab, and ventilating air is supplied through a shielded duct from a blower mounted on top. The objective lens systems of the periscopes are exposed to moderately severe gamma bombardment while the crane is loading the dissolver or moving contaminated equipment. As a result, the lens elements gradually become discolored. With normal usage, however, this effect is expected to be negligible for a period of at least 10 years.

Metal is brought in through a railroad tunnel which enters below deck

level near the head end of the canyon. Hatches over the tunnel are normally closed by covers of steel plate, but standard cell covers can be used if needed. The tunnel extends away from the building for some distance to reduce the hazard of backscattering to adjacent areas such as the tank farm, and is closed by a door of conventional construction. When the dissolver is to be charged, a train of the special tank cars mentioned previously is backed into the tunnel, the locomotive withdraws to a safe distance, and the buckets are picked out of the casks by the shielded crane. The canyon and the area in front of the railroad tunnel are evacuated during this operation.

Sampling devices are located in boxes shielded with 4 inches of lead and sunk flush with deck level. Built-in sprays are provided to wash out the inside of these boxes. Samples are taken by means of special instruments described in Chapter V and are transported and stored in stainless steel containers with walls 2 inches thick. These containers reduce the radiation from the most active samples to the tolerance level of 12.5 mr/hr at a distance of 1 to 2 feet.

Ionization chambers which indicate the activity of process solutions are located in wells which run from deck level down to the level of the process vessels. These wells are plugged to stop the radiation scattered up the tube.

CONCENTRATION BUILDING

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The process solutions which are piped into the Concentration Building have been decontaminated by a factor of 10^5 and can be handled with comparatively light shielding. In many cases the distance between process vessels and operating areas is sufficient in itself to reduce the radiation intensity below the tolerance value, so that the shields provide an added margin of safety.

The walls of the operating cells are of concrete, 12 inches thick. The same thickness of concrete is used in the sampling vestibules. A walkway around the cells is shielded by an 8-inch concrete parapet, and the centrifuges are mounted on platforms shielded by 8 inches of concrete. The sampling vestibules also serve as labyrinths through which the centrifuge platforms may be entered. The cells may be observed from the operating gallery through ports in the wall. When not in use, these ports are closed by sliding shutters of 4-inch concrete. The ports are also equipped with plate glass windows in a hinged sash as protection against splashes.

The remainder of the building is of conventional concrete block construction. A fence keeps casual bystanders at least 15 feet from the cell side of the building.

ISOLATION BUILDING

Process material enters the Isolation Building after decontamination by a factor of 10^7. At this stage the dangers of product contamination are enormously greater than the radiation hazards, so that although no special shielding is required, an elaborate ventilation system is maintained and great precautions are taken to prevent contamination of working areas and personnel.

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ADDITIONAL FACILITIES

Certain other facilities which branch off from the main process line may be mentioned briefly. These include the 291 Building, which is the fan house for the 221 Building; the 241 Building waste disposal facility; and the 222 Building analytical laboratories.

The 291 Building comprises the fan house and stack through which ventilating air and waste gases from the 221 Building are discharged. The fans are located in the open and are not shielded. Activity which is believed to enter the ventilating air stream as spray or mist from the process vessels deposits in the fan housings. At the present time the radiation intensity has reached a level of 1000 mr/hr at the most active spot on the fans, and falls to the tolerable value about 15 feet away. An earthen barricade of 4-foot minimum thickness is being constructed 6 feet from the fans. The off-gas lines from the dissolver run underground to a set of steam jets located in a concrete pit at the base of the stack. The activity in this pit is too high to permit entry during dissolving, and residual activity at times when the dissolver is not operating is high enough so that the pit can be entered only for limited periods.

The 241 Building consists of a buried tank farm for the disposal of active wastes. The tanks are buried under a minimum depth of 9 feet of earth, which reduces the radiation intensity at ground level to 1 mr/hr or less. Tanks which handle the most active wastes are fitted with air-cooled reflux condensers. The condensers are connected to the tanks through a buried concrete labyrinth.

The 222 Building is of conventional concrete block construction. The counter room in this building is shielded by 2 feet of concrete. Samples are brought in by means of stainless steel carriers, and are stored on shelves behind an inch of lead for additional protection. The small amounts of solution used in the actual analyses are handled with special instruments designed to shield the analysts from beta rays. Gamma rays from such small samples do not constitute a serious hazard.

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SECTION C - SEPARATIONS

CHAPTER XII - HEALTH PROTECTIVE PROCEDURES

Because of the radioactive nature of the materials processed in the separations areas, even though adequate provision has been made in equipment and buildings for the safe handling of materials, it is essential that constant vigilance be exercised to insure that no individual is exposed to excessive radiation. This chapter discusses the general health protective procedures employed.

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SPECIAL HEALTH HAZARDS IN THE SEPARATION (200) AREAS

As discussed in Chapter II, the various operational areas have been laid out with the various units being widely separated from each other, such that any accident occurring in one unit would not cause damage to the other units. The previous chapters have also discussed the radioactive nature of the product and fission elements associated with the uranium metal coming from the piles in the 100 Areas.

It has been pointed out that the product emits alpha rays with a range of 3.7 centimeters, while the various fission elements emit beta and gamma rays of various intensities. In Chapter I the nature and penetrating power of these radiations, the safe radiation limits for personnel, and the general principles of shielding against their effects were discussed. Here it was reported that the maximum permissible dosage from exposure has been established at 12.5 milliroentgens (mr.) per hour for gamma radiation, and 12.5 milliroentgen equivalents (mrep) per hour for beta radiation for a total of not more than 8 hours in any 24-hour period. The total permissible mixed fission products which may be taken into the body was stated as 10^{-2} to 10^{-3} curies. The tolerance limit in air for continuous exposure (8 hrs. per day) is 3×10^{-14} curies per cubic centimeter. Xenon which does not deposit in specific body locations is considered tolerable in concentrations up to 10^{-11} curies per cubic centimeter. The lifetime tolerance dose for product, which is retained in the bone marrow of the body, is considered about 10^{-6} grams, and the tolerance concentration in air set at 5×10^{-16} grams per cubic centimeter. Due to the low tolerance limit it is considered unsafe for any product whatever to enter the blood stream through a cut or abrasion.

The various elaborate provisions in building and equipment design and operation for protection against these hazards are described in Chapter II to XI inclusive.

Even though extensive provisions have been made for safe handling of processing solutions, it is considered essential that a thorough and constant program for monitoring the various operating areas and equipment be maintained, and that a constant check be made upon the exposure to these hazards experienced by the personnel entering the various operating areas, in order that accidental overexposure may be avoided.

The chief items requiring constant careful monitoring in order to insure safe operating conditions are included in four main categories, namely stack gas and ventilation hazards, fission product contamination of operating areas and equipment, product contamination of working areas and equipment, particularly in the laboratories and in the Concentration (224) and Isolation (231) Buildings, and contamination of hands and clothing of the operating personnel. Constant alertness must be exercised to detect process accidents such as "suckbacks" of process solutions into the pipe and operating galleries, "blowing" of samplers during the sampling operation through plugging of the jet lines, or accidental spilling of solutions.

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While it is beyond the scope of this chapter to attempt to outline the detailed monitoring procedures or equipment used for health protection, the following discussions cover the general features involved in control of hazards of each of the four categories.

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STACK GAS MONITORING METHODS

As described in Chapter IV, during the dissolving operation considerable quantities of radioactive iodine (I^{131}) and xenon (Xe^{135}) are liberated through the 291 Building stack. The gamma emitting iodine has an 8-day half life and is absorbed fairly quantitatively into the lungs, of which about 20% is believed to be deposited in the thyroid. Iodine inhalation can be readily detected by checking the gamma emission from the thyroid externally with a small Geiger - Mueller counter probe attached to an electronic (Chicago) scale circuit. At present 750 gamma counts/minute above background is considered a tolerance level for radioactive iodine.

The xenon (half life of 5.4 days) hazard is primarily one of external radiation, since it is not believed to be retained within the body over about 1 hour. The tolerance dose is set at 1.4×10^{-11} curies/cc. for breathing over an 8-hour day.

In 292 Building is provided equipment for the collection of samples of the 291 Building stack gases, and for determination of the radioactivity due to iodine and xenon present. While the method of determination of these activities present is still in the development stage, it consists essentially of the following steps: (See Flow Diagram in Figure 1)

1) Removal of a sample of the gases from the stack area entrance into 292 Building at a fixed flow rate.

2) Passage of the stack gases through a water cooler and thence through a scrubber where iodine is removed with a refrigerated aqueous solution of soda ash.

3) Passage of the soda ash solution containing the iodine through an ionization chamber where the activity is measured and recorded using a standard Beckman amplifier and Micromax hook-up.

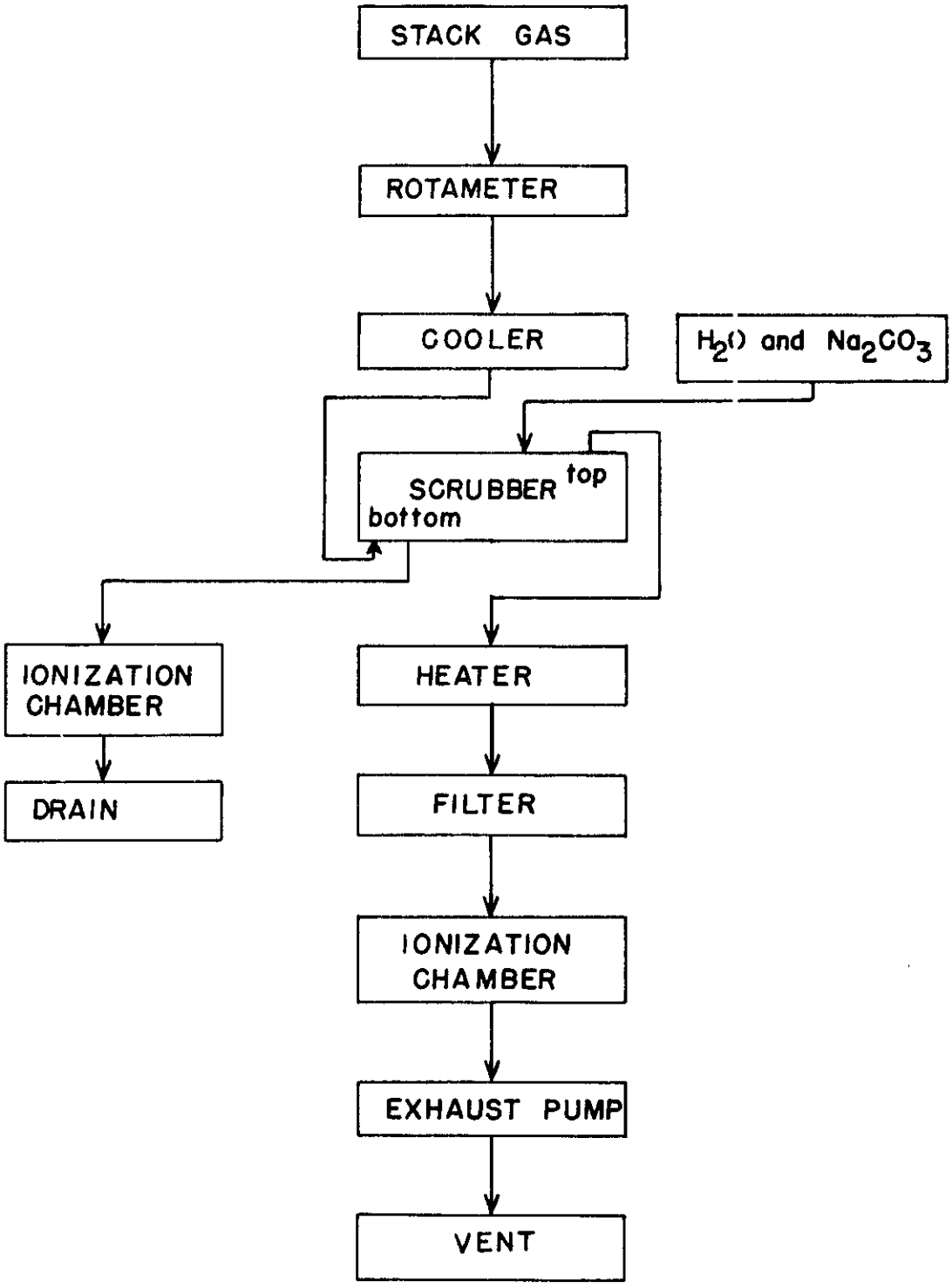
4) Passage of the xenon through a heater, followed by a silica gel filter and then through an ionization chamber where radioactivity is measured and recorded.

5) The gases remaining are exhausted to the atmosphere through a roof vent.

The main difficulties encountered at present in the use of this equipment consists in the difficulty of maintaining a uniform rate of flow of water through the scrubber and contamination of the ionization chambers. (This stack gas monitoring equipment is described further in Chapter X.)

FIGURE 1

FLOW SHEET FOR STACK GAS MONITORING EQUIPMENT, 292 BUILDING.



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VENTILATION MONITORING METHODS

While careful provision has been made for adequate ventilation of all operating areas, in order that any accidental unsafe conditions may be detected promptly frequent monitoring of the air in the operating areas is also carried out. This is especially important in the Isolation (231) Building where product may be present in the air. Portable electrostatic precipitators which collect the radioactivity present on metal tubes, as well as portable filtering equipment which collects the activity on filter paper, are used, and the activity determined by counting in a special counting chamber.

Frequently the paper filtering media in the ventilating equipment supplying the operating areas in the Canyon (221) and Concentration (224) Buildings are monitored, using a portable Beckman direct reading meter.

Precipitrons

The precipitrons used are manufactured by the Mine Safety Appliance Co. or locally in the instrument shops and consist of a suction head comprising a high voltage electrode supported by appropriate insulators and extending axially through a removable 1 1/2 inch diameter by 7 1/2 inch long cylindrical metal tube. This unit is connected to a high voltage source adjusted just below the sparking voltage and a suction fan, whereby the air may be drawn through the tube at a known rate and hence between the charged surfaces. The removable metal tube is lined with paper or preferably aluminum foil on which the precipitated material is deposited. The exposed paper or aluminum foil is counted without removal from the metal tube in a "Long Tom" chamber assembly. In using this instrument a sampling time must be used which is long enough to give a sufficient volume of air for the desired degree of sensitivity in product determination required. An upper limit of 1 hour for the sampling time is imposed since breakdown of the equipment often occurs when periods greater than this are used. Other long life alpha particle emitters such as uranium metal or its fumes or natural radioactivity in the air cannot be distinguished from product, except by counting after proper decay periods.

Alpha Counters

The counters for alpha particles are similar to those used for analytical purposes and are described in detail in the Manual for Analytical Instruments. Essentially these instruments measure the electrical impulses caused by an alpha particle ionizing the air through which it passes in an electric field set up between two specially constructed electrodes which are mounted within a chamber. While these instruments (amplifiers, scalers and recorders) used in the alpha counters for health monitoring purposes are substantially identical with those used for analytical purposes the actual construction of the chambers is often varied in order to accomodate the object being monitored. In the so-called Standard Chamber the grid electrode (collectrode) is a brass plate fixed within the chamber, and the high voltage electrode is a parallel brass plate about 1/2 inch below it and fastened to a small hinged door which

swings outward for loading, breaking the high voltage connection when the door is opened. This chamber is used for counting the small paper air filters (4.25 to 5.5 cm. in diameter). In the "Long Tom" chamber which is used for counting the precipitron tubes and larger paper air filters, the collectrode is a small 3/8 inch diameter tube positioned axially in the chamber, and the metal precipitron tube becomes part of the high voltage electrode.

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Beckman Radiation Meter

This portable instrument consists essentially of an ionization chamber, direct current electronic amplifier, and a sensitive ammeter. The ammeter is calibrated directly in mr/hour, and may be adjusted to measure over four ranges of radiation through a selector switch. The ranges covered are 0-20 mr/hr, 0-80 mr/hr, 0-400 mr/hr, and 0-2000 mr/hr. A fiber shield with removable slide is provided around the ionization chamber. By subtracting the shielded chamber reading from an unshielded chamber reading an approximate value for beta radiation may be obtained.

MONITORING OF EQUIPMENT AND OPERATING AREAS FOR CONTAMINATION

While the equipment and facilities provided are quite adequate for handling the active process solutions under normal operating conditions, in the event of operational accidents, or should it be necessary to enter any of the equipment cells or areas for inspection or maintenance, it is necessary that the areas and equipment be surveyed for contamination due to process solution or condensate fumes.

Methods for Gamma and Beta Radiation Monitoring

Where it is possible for the person conducting the survey to approach the equipment or area in person, the portable Backman Radiation Meter is usually used for initial survey work for beta and gamma radiation because of its direct reading nature and because of the wide range of radiation intensities over which it indicates.

A Victoreen radiation meter, of the same general type as the Beckman Meter, which operates only over a single range, is also used for the above surveys. These meters are available in three different ranges of activity - 0-70 mr/hr, 0-100 mr/hr, and 0-50 r/hr; in each case the dosage rate can be estimated up to twice the rated limit. In the high range instrument the chamber is mounted on the end of an aluminum pole, and thus may be used in initial surveys of very active areas.

A third instrument used for survey purposes is a portable Geiger-Mueller tube counting meter. (See Chapter X for a description of this tube). This instrument is calibrated to read in mr/hr and covers ranges of 0-0.12 mr/hr and 0-2.5 mr/hr. This instrument is useful in locating very small beams of radiati and is especially useful for monitoring tools.

The Lauritsen quartz fiber electroscope is also used for survey purposes and is one of the most accurate available. It covers the range of about 0-1000 mr/hr. In using this instrument after charging the electroscope, the time required for the quartz fiber to move over a measured distance during the process of discharging the electroscope is determined. A calibration chart is provided from which the intensity of the radiation amy be determined after measuring the rate at discharge. This instrument is used where most accurate measurements of the radiation intensities are required.

A special Geiger-Mueller tube counter (described in Chapter X) with the pulse amplifier at the end of a cable is used to monitor the test wells in the 241 Building (Tank Farm) area. This arrangement permits the use of a standard scaler and counting procedure. In order to check waste tank leakage and seepage from dry wells further, all of the pumping wells on the reservation are sampled weekly. In addition, several stagnant ponds around the area are sampled at regular intervals. The abandoned 241-361 Dry Well is also checked each week for activity. These samples are evaporated and counted by the regular analytical procedures.

The Laundry Ditch, the 200 North area ditches and the disposal ditches in the 241 Area are checked frequently with the portable Geiger-Mueller counter, and occasionally samples of the mud from these areas are counted for activity.

Deposition of the active material from the 291 Building stacks is monitored over the areas with the above portable meters and through the use of ionization chambers located at strategic spots around the areas. Two types of ionization chambers are in use. One type having an approximately 1/8 inch thick Bakelite wall is relatively insensitive to soft beta radiation. In the second type, one half of the wall is cut away and is covered with 0.001-inch thick aluminum foil. These chambers, which are used in duplicate, are used principally outside the 200 Areas. A set of 29 Integrons chambers (See Chapter X) is maintained outside of the process buildings in the operating areas, and at Pasco, Kennewick, Benton City, Riverland Yards, Richland and Hanford.

Methods for Monitoring Alpha Activity

The principle survey instruments used for monitoring alpha activity or product are the Sandy Zeuto and Pappy meters. The Sandy meter consists essentially of a small ionization chamber, with one face of wire screen to permit penetration of alpha particles, connected to an electronic amplifying circuit and meter to measure the ionization current in the chamber. The ionization chamber consists of a 3 inch by 5 inch by 1-1/2 inch metal box with 14 to 16-mesh wire screen on one of the large surfaces. An electrode constructed of metal rods of a double cross form is mounted in insulators parallel to the wire screen. A potential of about 50 volts is maintained between the metal box or wire screen and the electrode through a high resistance of about 2 x 10 inch ohms. When ionization occurs in the chamber due to the entrance of alpha radiation, the change in current flow through the chamber is taken as a measure of the ionization and is reflected by an increased voltage drop across the high resistance. In measuring this ionization current, a two stage DC amplifier is used (illustrated in the wiring diagram, Figure 2). Since this instrument indicates

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FIGURE 2

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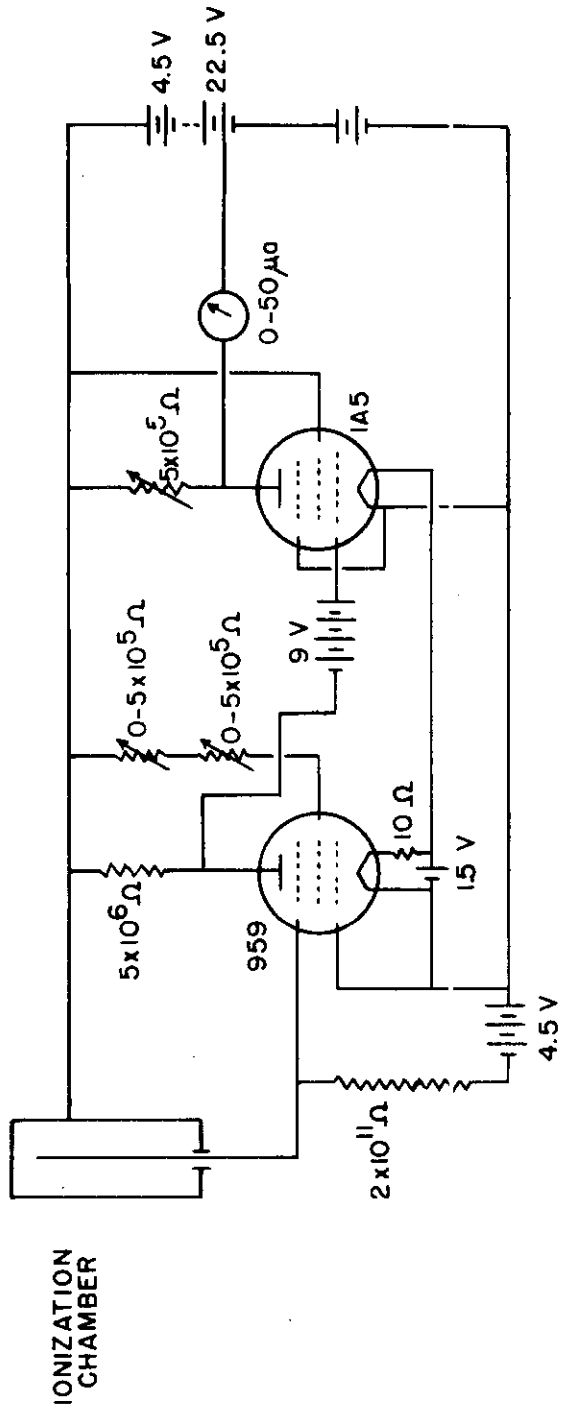
SIMPLIFIED CIRCUIT DIAGRAM OF "SANDY" ALPHA SURVEY METER



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chamber ionization, it is sensitive to any ionizing radiation. Flames, drafts, or mechanical disturbances of the chamber also affect its readings. This instrument is useful in obtaining a rough measure of fairly high levels of alpha radiation. Since the range of the alpha particles from product is 3.7 cm. its radiations are readily absorbed by thin layers of solids or liquids such as a sheet of paper or a film of water. In order to monitor a surface for product with this instrument it is necessary, therefore, to move it completely over the surface with the face of the screen a short distance away from the surface being monitoring, and it is also important that the surface being monitored be dry. Since the zero setting of this instrument is not very stable, it is necessary to readjust the rheostat frequently or to use the change in deflection from an initial reading as the criterion of the contamination present. Drafts must generally be shielded from the sensitive screen by eliminating the source or by deflecting the air stream away from the instrument. If doubt exists as to whether or not the contamination being monitored is alpha radiation, interposing a sheet of paper between the contamination and the meter will cut out that portion due to the alpha radiation. Calibration of the instrument is carried out against known sources of product. The full scale reading of an average production model "Sandy" meter is about 20 - 80000 alpha disintegrations/minute, which represents about 1500 disintegrations/minute/scale division and in practice the useful detection sensitivity is about 2 - 3000 disintegrations/minute.

There are a number of attachments for the "Sandy" meter which extend the usefulness of this meter. In addition to the regular ionization chamber, other ion chambers based on the same principle, but differing in shape, may be constructed, thus permitting a more convenient shape for monitoring in certain locations. Outside of geometry corrections the shape of the ion chamber does not appear to affect appreciably the sensitivity of the instrument. Two main types of auxiliary ion chambers are available for use with this instrument:

a) Probe. The overall dimensions of this chamber are about 2 inches diameter by 10 inches long. The sensitive portion of this chamber is represented by the outer 5 inches of its length. This is quite useful for monitoring in vessels or other tight places.

b) Floor Monitoring Chamber. This is a large ion chamber, in overall dimensions about 4 inches by 18 inches by 2 inches thick, with a tray to hold the regular "Sandy" meter, all mounted on casters and provided with a convenient handle. This permits monitoring a strip of floor 18 inches wide at the rate of about 20 feet/minute.

In some cases it is necessary to locate contamination on some surfaces which are not readily accessible to the above meter, or when a measure of the "looseness" of the contamination is desired. In practice a small 1 1/4 inch by 1 1/4 inch square of paper is pressed down and wiped across the surface under study by means of a 3/4 to 1 inch diameter rubber stopper wrapped with friction tape. The paper which adheres to the tape is removed with forceps, and counted in a regular alpha counting chamber.

The electrical precipitators or Precipitrons described above under ventilation monitoring methods are particularly useful in monitoring air for alpha

radiation, and are used regularly in monitoring the exhaust air from the filter boxes for the ventilating system exhausting air from the laboratories and processing areas in the Isolation (231) Building.

The mud samples from waste disposal ditch from the Isolation (231) Building are counted weekly for alpha activity.

METHODS FOR MONITORING OF CLOTHING AND PERSONNEL

In addition to careful monitoring of the equipment and area for radiation hazards, a constant check is made upon the clothing and hands of the operating and maintenance personnel for contamination. Each individual while in the operating area also is required to wear individual instruments for indicating the radiation to which he may become exposed.

Clothing Monitoring Procedures

Every operator in the separation area is required to wear coveralls and caps where needed. This clothing is regularly laundered and monitored for contamination. Shoes, overshoes, towels, cloth shoe covers, and gloves are also supplied where needed and are also regularly monitored. Routine monitoring of incoming laundry for beta and gamma radiation is carried out with Islip counting rate meters. Finished laundry is monitored with Geiger-Mueller tube chambers connected to scaler circuits. Four Geiger-Mueller tubes are used on each side of the garment. The tubes are arranged so that with a point source the minimum reading is 50% of the maximum. Measurements from each side of the garment are made independently. Gloves, rubber overshoes, shoe covers, caps, and towels are monitored several at a time.

Alpha monitoring of clothing is carried out, using the Pappy instrument referred to above.

In the laundry, Igepal C A is used for washing contaminated items. Acetic acid is generally used for the beta and gamma decontaminating agent, and citric acid for the alpha decontaminating agent.

Shoes and leather gloves are checked by using a thin walled glass Geiger-Mueller tube mounted in a rectangular box and connected to a scaler circuit.

Hand Monitoring Procedures

Hand monitoring is especially important since contamination is most likely and can readily lead to contamination of food. The hand monitoring device used for detecting beta and gamma radiation contamination is called the "four-fold hand counter". It employs eight glass Geiger-Mueller tubes so arranged that two tubes are exposed to each of the four surfaces of the hand. Each unit of two tubes is connected to a separate scaler circuit and register, such that each surface of the hand is monitored simultaneously.

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The hand monitoring device for alpha (product) activity is called a "Handee" counter. This consists of a sound insulated box containing a pulse collecting unit connected to a scaler circuit, recorder, and oscilloscope. The pulse collecting unit consists of a cylindrical screen in the center axis of which, and insulated from the screen, is positioned a brass electrode. A high electrostatic field is maintained between the screen and center electrode. A wire screen handgrip surrounds the cylindrical screen electrode. In counting, only the palm surface of the hand can be satisfactorily monitored. This collecting unit has a geometry of about 11%. Counting values corresponding to 500 disintegrations/minute are considered the upper safe limit for product contamination.

In addition to the hand counting procedures described above, finger packets consisting of two discs of sensitive photographic film enclosed within a waterproof cover are supplied to individuals suspected of being exposed to abnormal amounts of radiation, and those who have been observed to have high beta and gamma hand counts. Determination of the radiation exposure is made by measurement of the blackening with a densitometer following development of the film simultaneously with an unexposed sample.

PERSONNEL EXPOSURE MONITORING PROCEDURES

All personnel entering the 200 Areas are required to wear two pocket meters or "pencils", and a "film badge". These instruments are intended to indicate the degree of beta and gamma radiation exposure which each individual experiences.

Pocket Meters

The pocket meter, which is essentially an ionization chamber, is illustrated diagrammatically in Figure 3. In use a predetermined charge is placed on the inner electrode and the extent to which the charge is dissipated by exposure of the meter to beta or gamma radiation is measured by means of a Victoreen Minometer. This latter instrument consists essentially of a potential source and an electrometer, the fiber of which casts a shadow across a scale calibrated in milliroentgen. In use the instrument is adjusted to a zero reading, then a pocket meter from which the end cap has been removed is placed in a slot provided. If a deflection of the electrometer results, the number of milliroentgens exposure is read from the calibrated scale. The pocket meter is then recharged with a known potential by depressing a button provided. Upon replacing the end cap the meter is ready for re-use. A dessicant is included in the cap of the pocket meter in order to prevent leakage of the charge due to moisture. The pocket meter may be discharged by mechanical shock; therefore, in use, dropping them or otherwise subjecting them to mechanical shock should be avoided. These meters record radiation exposure up to 200 mr. In order to minimize error, two meters are worn by each worker, and are monitored daily.

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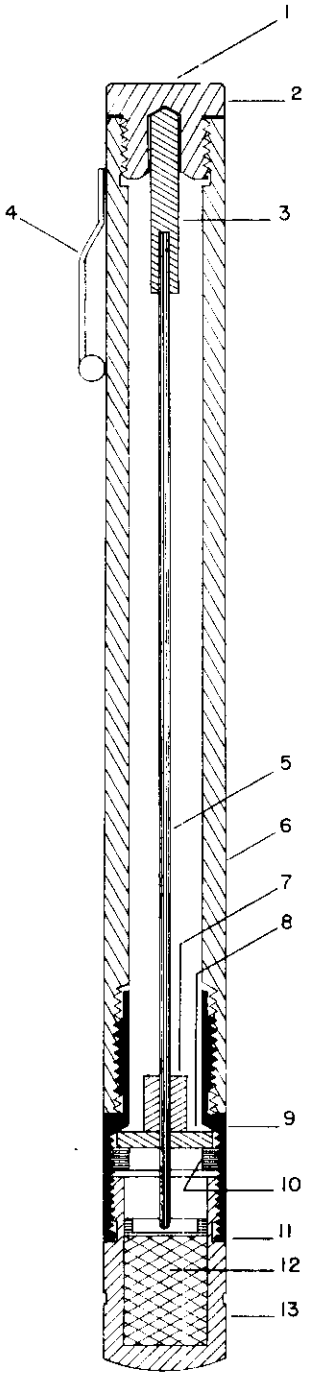
FIGURE 3

DIAGRAM OF POCKET METER



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LEGEND

1. IDENTIFYING NUMBER
2. TOP CAP, ALUMINUM
3. POLYSTYRENE INSULATOR
4. POCKET CLIP
5. ALUMINUM ROD, AQUADAG COATED
6. BAKELITE BARREL, AQUADAG LINED
7. ALUMINUM REINFORCING SLEEVE
8. POLYSTYRENE INSULATOR
9. ALUMINUM ADAPTER
10. ALUMINUM RETAINER RING
11. GASKET
12. SILICA GEL DESICCANT
13. BOTTOM CAP, ALUMINUM

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Film Badges

The film badge is illustrated diagrammatically in Figure 4. This badge is loaded as indicated in Figure 4 with two different kinds of film, which are sensitive to beta and gamma radiation. A silver shield covers approximately three fourths of the film surface such that it is possible to obtain an indication of the relative exposure to beta and gamma radiation Perforations in the silver shield are used for permanently recording the wearer's badge number by exposing the shielded film to a known source of X-rays. With each lot of film used a series of standards are prepared by exposure to known radiation sources, as well as blank having no exposure. The degree of exposure experienced by the wearer is determined by comparison of the blackening of the film present with the standard by means of a sensitive densitometer. Over the range of 0 to 20 roentgens of radiation good accuracy and reproducibility is obtained. The film batches are worn for a week, after which they are monitored and reloaded. The developed film from these badges comprises a permanent record of each employee's exposure to beta and gamma radiation.

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FIGURE 4

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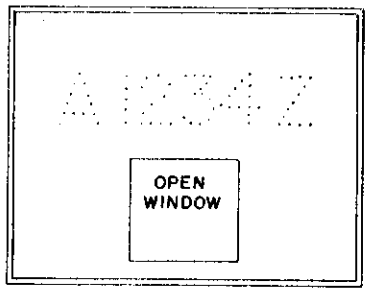
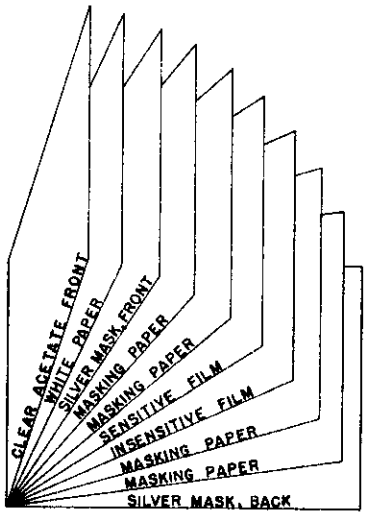
DIAGRAM OF FILM BADGE

FRONT SILVER MASK

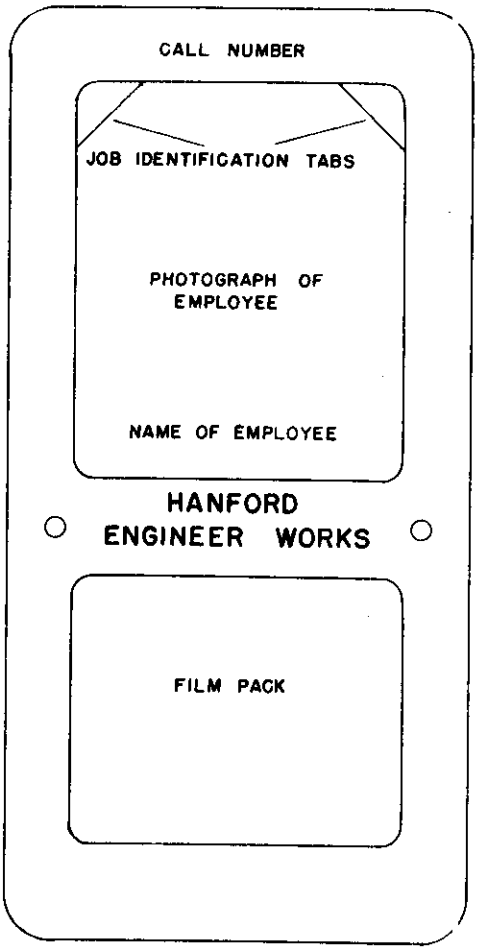


(BACK SILVER MASK IS THE SAME EXCEPT IT DOES NOT HAVE THE EMPLOYEES PAYROLL NUMBER DRILLED THROUGH THE SILVER.)

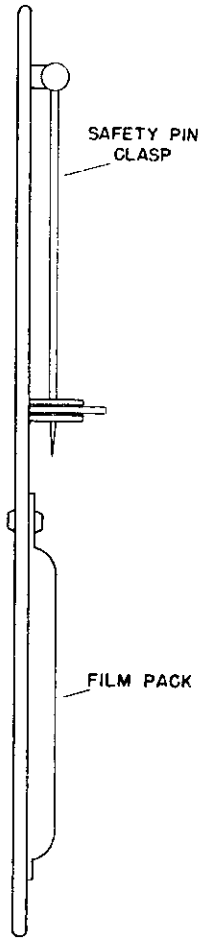


MAKE-UP OF FILM PACK

BADGE, FRONT VIEW



BADGE, SIDE VIEW



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CHAPTER XIII - EQUIPMENT DECONTAMINATION

To avoid complete dependence on remote maintenance, it is desirable to be able to decontaminate Canyon Building process equipment from by-product activity so that it can be approached for short periods. In the Concentration and Isolation Buildings product decontamination of process equipment and work areas is necessary to eliminate any possible health hazard. This chapter describes means of removing product and by-product contamination from process equipment.

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BY-PRODUCT DECONTAMINATION

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Decontamination with Nitric Acid

The first step in by-product decontamination is to wash out the equipment with nitric acid. This treatment removes some of the activity and also dissolves accumulated bismuth phosphate which might otherwise interfere with subsequent treatments. The nitric acid wash also removes essentially all of the product and may need to be processed for product recovery.

The acid is about equally effective at concentrations from 35% to 60%. This permits a charge of 60% nitric acid to be circulated and jetted through a series of tanks without losing its effectiveness until jetting dilutions reduce its strength below 35%.

The acid is heated to around 60°C to increase the rate of solution of the active deposits. The wash has no significant corroding action on the heat-treated 25-12 stainless steel vessels.

Decontamination with Solutions other than Nitric Acid

A substantial part of the contamination in process equipment is not removed even by repeated nitric acid treatments. Analytical data on the chemical identity of this activity are not available. It is believed to be associated with the insoluble bonding compound present in the metal solution, by-product precipitates formed during extraction, and zirconium phosphate scavenger.

Operating experience at Hanford on the removal of nitric acid-insoluble activity is confined to the use of nitric acid/ammonium silicofluoride washes. At concentrations of 5% ammonium silicofluoride and 15-25% nitric acid, the cleaning solution is not seriously corrosive to process equipment and can be used safely under such conditions as 1-hour circulation or agitation at 50°C.

Cleanup experiments in the pilot plant, generally based on laboratory treatment of test pieces exposed in the extraction precipitator or catch tank, indicated that alkali treatments remove a good deal of the nitric acid-insoluble activity. Of all the treatments studied, including both acid and alkaline washes, the best was sodium hydroxide/sodium tartrate solution. This was dropped from consideration for use at Hanford only when sodium tartrate in the required quantities was found to be unavailable.

The tartrate solution is effective at 5% sodium hydroxide/1.25% sodium tartrate. There is little improvement at higher concentrations, but the degree of decontamination drops off sharply if the concentration of either ingredient is lower than these minimum values. A half-hour treatment at 65°C was found sufficient to give maximum decontamination.

Sodium hydroxide at concentrations of 5-20% also has marked decontaminating action, although only about a fifth as effective as the tartrate solution

in the pilot plant tests. Addition of sodium citrate, potassium oxalate, "Calgon", borax, mannitol, or glycerine to the caustic offered no improvement.

Neither sulfuric nor hydrochloric acid was found to have significant decontaminating action as a follow-up to nitric acid washes.

Inactive fission product solution and such mixtures as 10% zirconyl nitrate/5% nitric acid were also tested, with unpromising results.

Mixtures of nitric and hydrofluoric acids decontaminate quite well. At high concentrations or temperatures, this type of solution is seriously corrosive. However, 10% nitric acid with 1% hydrofluoric acid is quite safe at room temperature and decontamination is still good.

Equipment Cleanups in 221-T Canyon Building

Based on experiments in the pilot plant and limited operating experience at Hanford, the preferred cleanup solutions are 60% nitric acid followed by 25% nitric acid/5% ammonium silicofluoride. Temperatures are held near, but not above, 60°C for the acid and 50°C for the acid/silicofluoride during treatment of the various tanks.

In catch tanks and precipitators, the cleanup solution is circulated through the distributor for 20 minutes, allowed to soak for an hour or more, then circulated again for 20 minutes.

The solution tanks are agitated for at least 1 hour with the wash at the prescribed temperature. Since these tanks have no distributors, it is necessary to use 3000-5000 pounds of wash to reach all of the normally wet surface.

The centrifuge is the most difficult piece of equipment to clean properly. One or two initial washes are made with the bowl stopped, over-flowing into the catch tank. This procedure reaches the outside of the bowl and the central portion of the casing, which are not normally wet, but on which spatter deposits accumulate. The bowl is then brought up to speed and the wash is recycled through two or three times.

A water wash after the chemical cleanups effects further decontamination. Also, it is important that no silicofluoride wash remain in tanks where it could interfere with oxidations and cause by-product losses; 8-4, Section 12, 13-1, 14-4, 16-1 and 17-4.

It appears to be good practice not to allow washes used in cleaning highly contaminated equipment to get into relatively clean vessels. The latter are likely to be further contaminated, rather than decontaminated, and furthermore, insolubles thus introduced into vessels where they do not normally accumulate may interfere with subsequent process runs.

The decision on the sequence in which a given group of tanks should be

[redacted]

treated and how far down the line it is advisable to go with a particular batch of wash solution is based on the relative contamination of the tanks as shown by the Micromax-Beckman readings. For example, Section 8 and 13-1, 13-2, and 13-4 are from one hundred to some thousands of times more active than 13-3 and succeeding process tanks. Consequently, Section 8 and 13 washes should be sent to waste, not to Section 14. To minimize transfer of active solids from 8-4 or 13-1 to 13-3 during washing operations, solution is jetted from 13-1 only when the bowl is running. Washing of the stationary centrifuge is accomplished by running in fresh solution from a gallery scale tank.

For example, in cleaning out Section 13, washes are added, with the bowl stopped, through Scale Tank 13-2C. The contents of the bowl are jetted to the precipitate dissolver and the centrifuge brought up to speed. The bulk of the wash, in the catch tank, is recycled through the precipitator and the centrifuge twice. After the second centrifugation, the catch tank is cleaned out using the spray distributor. The solution is then brought back to the precipitator, which is also cleaned out with the distributor. The wash from 13-1 is moved over to 13-4 through 13-2 in a series of slurrying operations, as in cake removal, to take out insoluble material that has been caught in the bowl. After heating and agitation in 13-4, the wash is sent to waste, unless product recovery is required.

Results of equipment washes in 221-T Canyon Building and Hot Semi-Works are given in the appendix at the end of this chapter.

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PRODUCT DECONTAMINATION

Product decontamination of equipment and work areas in the Concentration (224) and Isolation (231) Buildings is necessary to eliminate the possible health hazard involved in the maintenance of the equipment and the safe occupancy of the work areas.

No definite method or procedure for product decontamination has been established, since the treatment is dependent upon the nature of the equipment and the extent to which it is to be decontaminated. Product decontamination of the interior surfaces of process tanks, transfer lines and transfer vessels can be obtained satisfactorily by washing with nitric acid. At other points where decontamination may be required, such as flange leaks, small spills, hoods, etc., the method varies with the individual case and the type of surface to be decontaminated. Although a contaminated surface should be washed with the most corrosive chemical that the surface will tolerate to obtain maximum decontamination, the hazards to personnel make the choice of less corrosive agents more desirable. In the use of most of the decontamination agents which have proved successful, the technique followed in the application of the cleaning agent is often more important than the effect of the cleaning agent itself.

In dealing with product contamination there are several basic principles which should be followed:

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1. Take all possible precautions to limit the contamination to the smallest possible area.

   a. Do not flood with water or any decontaminating agent.

   b. Protect all the surrounding area which might be spattered during the decontamination treatment by spreading paper and/or hanging troughs under overhead flanges.

   c. Do not set down contaminated material such as tools, rags, swabs or gloves on un-contaminated areas. Use paper bags or other suitable containers.

2. The most effective decontaminating solutions which have been used are listed below.

   a. Chromic acid cleaning solution - This solution is very corrosive to stainless steel and is used only as a last resort.

   b. Strong nitric acid (60%) - Do not use on paint or concrete surfaces. Strong acid leaves concrete very porous and susceptible to additional contamination.

   c. Citric acid solution (20%) - This is the safest and easiest to use although it is not always the most effective.

   d. Various organic solvents - Effective on painted surfaces where the contamination can be removed along with the surface of the paint film. Care must be exercised to avoid merely softening the paint and allowing the contamination to soak into the paint so that it is not readily observed.

   e. Spindle oil (Pyrorex C) - Recommended from tests at the pilot plant for cleaning hood surfaces. It has not been used here since it is felt that the chief effect of such a treatment is the masking of the contamination with an oil film which prevents detection of contamination by existing instruments.

   f. Bon Ami or similar abrasive agents - Extensive hand scrubbing with abrasive paste has been successful in the plant, but extremely time consuming.

   g. Permanganate-bisulfite - Scrubbing with saturated $KMnO_4$ solution, removal by wiping, followed by scrubbing with 10% $NaHSO_3$ solution, and water rinse is comparable to citric acid treatment.

None of these cleaning agents are perfect decontaminating agents and it is to be emphasized that careful application and scrubbing is an important factor.

The following procedure which was effective in reducing by 95% the contamination resulting from a small flange leak is given as an example:

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PRODUCT DECONTAMINATION

1. The surrounding area which might be spattered during the treatment is protected by spreading heavy brown paper.

2. Wring out an asbestos swab in the cleaning solution and rub the contaminated area. Place swab in paper bag.

3. Dry with a clean swab. Place swab in a paper bag.

4. Repeat 2 and 3 several times depending on the degree and extent of the contamination.

5. Wring out a rag in soap and water and rinse cleaned area. Place rag in paper bag.

6. Dry with a clean rag. Place rag in paper bag.

7. Repeat 5 and 6 at least three times.

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REFERENCES

Report CN 2208	"Equipment Decontamination"
File No. 6264	"200 Area Report for January 1945" (Pilot plant)
File No. 6555	"200 Area Report for February 1945" (Pilot plant)
File No. 540-S	Acid Wash "T-5-01-AW-1,-3"

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APPENDIX

CORROSION RATES OF WASH SOLUTIONS

(Maximum reported rates on heat-treated 25-12)

Solution	Temperature	Corrosion Rate
60% nitric acid	75°C	0.00002 inches/mo.
60% nitric acid/1% hydrofluoric acid	Room	*0.004 " "
10% nitric acid/2% hydrofluoric acid	Room	0.0009 " "
10% nitric acid/1% hydrofluoric acid	Room	0.0001 " "
14% fluosilicic acid	75°C	0.027 " "
14% fluosilicic acid/6% nitric acid	75°C	0.006 " "
14% fluosilicic acid	40°C	0.0002 " "
29% ammonium bifluoride	Room	0.0004 " "

* also pits welds in 24 hours

EQUIPMENT CLEANUP DATA, 221-T, JANUARY, 1945

(Micromax-Beckman values after each wash, in units of 10^{-14} amperes)

Tank	Start	Wash No. 1	Wash No. 2	Wash No. 3	Intensity at Vessel Surface
B-1	3600	2800	820		50-100
B-2	1700		430*		
B-3	720		110		
B-4	10000		260		
B-1	50000		1000	1000	
B-2	3300		450	48	40-60
B-3	350		100	100	100-150
B-4	13000			1000	

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* Reduced to 120 after washing with sprays and hose

Wash No 1: 3000 lb. of 60% nitric acid

Wash No 2 and 3: Each, 5000 lb. of 25% nitric acid/5% ammonium silicofluoride

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APPENDIX

EQUIPMENT CLEANUP DATA, 221-T HEAD END, JANUARY, 1945

Tank	Start	Wash No. 1	Wash No. 2	Wash No. 3	Intensity at Vessel, mr/hr
T3-1	1000	170	6	3	13-34
T3-2	10000	12000	1100	6	15
T3-3	1000	630	5	3	30-64
T3-4	1000	340	6	5	23-40
T4-1	200	100	2	1	7-12
T4-2	300	280	3	2	8
T4-3	200	50	3	1	12-18
T4-4	200	120	3	1	12-90

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Wash No 1: 60% nitric acid, 1 hour at 50°C
Wash No 2: two washes, 15% nitric acid/5% ammonium silicofluoride, one hour at 50°C
Wash No 3: 60% nitric acid/1% hydrogen peroxide, 1 hour at 65°C

EQUIPMENT CLEANUP DATA, 221-T HEAD END, MARCH 1945

Tank	Start	Wash No 1	Wash No 2	Wash No 3	Wash No 4
T3-1	690	20	12	4	1
T3-2	6600	310	76	20	13
T3-3	5600	230	35	11	8
T3-4	920	40	16	5	4
T4-1	480	26	26	4	1
T4-2	190	15	5	2	1
T4-3	170	15	5	2	1
T4-4	550	40	16	8	4

Wash No 1: 40% nitric acid, 1 hour at 50°C
Wash No 2: 14% ammonium silicofluoride, 1 hour at 50°C
Wash No 3: 15% nitric acid/5% ammonium silicofluoride, 1 hour at 50°C
Wash No 4: Water, 1 hour at room temperature

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APPENDIX

EQUIPMENT CLEANUP DATA, 224-T, AUGUST, 1945

Reagent	Treatment	%Remaining Contamination Removed		
		Treat. #1	Treat. #2	Treat. #3
	A. Stainless Steel Surfaces			
60% HNO_3	Scrubbing with asbestos wicking followed by damp rag wipe-up	90	90	90
Sat. $KMnO_4$ - 10% $NaHSO_3$	Scrubbing with $KMnO_4$ for 5 min.; wiping up; scrubbing with $NaHSO_3$ for 5 min.; wiping up; rinsing with water	75	75	75
Bon Ami Paste	Scrubbing (hard) with paste and wiping up with damp rag	60	60	60
	B. Painted Concrete Surfaces			
Sat. $KMnO_4$ - 10% $NaHSO_3$	Same as for stainless steel	75	50	25
20% Citric Acid	Scrubbing with conc. citric acid solution and then rinsing and wiping with damp rags	50	30	20

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CHAPTER XIII - EQUIPMENT DECONTAMINATION

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