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ADAPTABILITY OF NEUTRON COUNTING TO THE REDUPLEX PROCESS

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ADAPTABILITY OF NEUTRON COUNTING TO THE RECUPLEX PROCESS

By

L. E. Bruns

FINISHED PRODUCTS TECHNOLOGY OPERATION
RESEARCH AND ENGINEERING OPERATION
CHEMICAL PROCESSING DEPARTMENT

August 5, 1957

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ADAPTABILITY OF NEUTRON COUNTING

TO THE RECUPLEX PROCESS

Introduction

In July, 1956, a program was initiated concerning the feasibility of applying a neutron counter to various Recuplex process streams. The program originated as the result of a suggestion by C. F. Setbecken and D. J. Brown of the 234-5 Manufacturing Instrument Department. The suggestion recommended that the CAW (Recuplex extraction column waste) and the CCW (Recuplex organic column effluent) critical mass control be monitored by a neutron counter as a replacement for the originally installed photoelectric cell (the latter did not work under Recuplex conditions). With the development of the CAW-CCW neutron detector other possible Recuplex applications became apparent, such as, neutron counting of powders to increase batch size, transferring Pu solutions to critically unsafe tanks without laboratory analysis, in-line monitoring of Pu product solutions (CCP), and accountability sampling of Pu slurry solutions. This report summarizes the work carried out to date and recommends future studies.

Summary

1. Initial work was carried out with CAW spiked samples using the "Big Bertha" Final Inspection Chamber. Favorable results led to a program which involved fabrication of two new counting chambers (see Figures II and III).

2. Additional CAW samples spiked with Pu and impurities, CCW samples, CCP (Recuplex solvent extraction product stream) samples, Slag and Crucible filtrate samples, slurry samples, and Task I supernate were some of the solutions studied using the new chambers. Results were favorable (see Tables I, II, III, and IV, and Figure I). Accuracy of CAW samples with their variable impurity levels was set at ± 25% in the range of 1 g/l Pu. SC filtrate samples could be detected within ± 25% in the 0.1 to 0.5 g/l Pu range. CCP samples at the 10 - 20 g/l Pu range could be detected within ± 10%.

3. Task II, III, and IV powders, as well as other miscellaneous dry solids, were checked in the neutron counter. As a result of these studies, more solids per run could be charged to a Recuplex dissolver without exceeding critical mass limitations.

4. Factors affecting the accuracy of neutron counting are background, light element impurities, and moderation between neutron probe and sample.

Discussion

Theory

The theory behind neutron counting is explained in DUH-10,012 (1) and LAMS-934 (2). Unlike charged particles, neutrons have very little interaction between them-

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selves and, in order to produce sufficient ionization, advantage is taken of the following reaction:

$$B^{10}_{5} + N^{1}_{0} \longrightarrow Li^{7}_{3} + He^{4}_{2}$$

BF_3 gas is placed in an ionization tube (BF_3 probe) and the above reaction takes place, giving charged particles which induce ionization.

Neutron emissions are usually accompanied by gamma and beta emissions. The latter can likewise produce ionization in a BF_3 tube. To distinguish between these different energy level particles, the beta and gamma induced ionization must be minimized. This is done by varying the voltage range so as not to accept gamma or beta pulses to the count rate meter (CRM).

Solutions and solid material containing Pu give off two types of neutrons, the spontaneous emissions from the Pu itself and secondary emissions from alpha-neutron reactions. Alpha particles contacting light elements such as boron, fluorine, oxygen, etc., cause reactions resulting in secondary neutron emissions. It is this latter emission which limits the accuracy of the neutron counter unless the amount of light element impurities remains relatively constant or is known.

Equipment

Initial work was carried out using the Final Inspection facilities. Seebacken and Brown, with the advise of R. E. Isaacson, Product Inspection Supervisor, tested 20 ml samples of Recuplex CAW spiked to a 1 g/l Pu concentration in the "Big Bertha," the Final Inspection neutron counting chamber. The "Big Bertha" is a paraffin-water moderated chamber. A definite count above background was noted and results were reproducible.

Based on these encouraging results, a neutron counting program was initiated and powders, simulated CAW solutions, CCW solutions, CCF solutions, SC filtrate solutions were counted in the chamber as sketched in Figure II. However, Task I supernates did not give comparable results with the cadmium and paraffin used as moderators (see Figure II). A new chamber constructed as in Figure III essentially eliminated moderation between sample and probe. With this second chamber, a good correlation was obtained between supernates and other type samples, for example, SC filtrates. The reason reduced moderation between tube and sample was necessary is not known. It is postulated that the moderation present in the original chamber allowed only a small percent of the neutrons caused by the α,n reactions to eventually become ionizations. However, SN's, with their high gamma energies, activated the α,n reactions sufficiently to product ionization. Removing the moderation caused the α,n reactions to induce ionizations when testing filtrate samples as well as SN samples.

The BF_3 tubes were assembled at the Hanford 300 Area shops. The tube consists of a standard aluminum slug-can filled with enriched BF_3 gas (96% B^{10}, 4% B^{11}) at a pressure of 25 cm Hg. The positive electrode is in the center of the tube and is 1 mil in thickness. The negative electrode is the aluminum can.

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In the set-up used here, the BF_3 tubes are subjected to 2100 volts by means of seven 300 volt batteries. The impulse from the ionization in the BF_3 tube is pre-amplified by means of a cathode-follower pre-amp. The signal was then further amplified by a Detectolab (DA 5) amplifier and sent to a Victoreen (670) count rate meter where the reading was made either by use of the indicator on the CRM, or from a Brown recorder connected to the CRM.

Results

CAW. Figure I depicts the data from Table I concerning the spiked CAW samples. Three different settings of gain and discriminator gave the 3 parameters shown. Simulated CAW samples were placed in ½-inch stainless steel tubes holding about 40 cc of sample. The original chamber, as shown in Figure II, was used in all CAW experiments.

Figure I shows good linearity for constant purity results except for the 3 g/l sample, and this sample was believed to be low in make-up. Where the CAW solutions were spiked with impurities, the following conclusions can be made:

1. 0.35 M of excess fluoride increases the C/M reading 20 - 50% (see points b on Figure I).

2. Addition of 0.5 M $Mg(NO_3)_3$ to the 0.35 M fluoride solution gave little change in C/M (see points a on Figure I).

3. 0.1 M of excess fluoride increases the C/M by 15 - 30% (see points d Figure I).

4. Doubling the normal amount of ANN present in a CAW batch increases the C/M by ca. 20% (see points c Figure I).

5. 1 g/l Pu in 5 M HNO_3 solution gives about the same reading as synthetic CAW containing 1 g/l Pu.

The 0.35 M fluoride is a larger excess than should ever occur in Recuplex. It can be concluded that impurities present in Recuplex CAW should not effect the neutron monitor accuracy of a 1 g/l CAW solution by more than ± 25% if calibrated properly.

Table II summarizes the CCW and CCP data. Samples were placed in 3/4-inch stainless steel tubes (75 cc) and were counted in the chamber as shown in Figure II. With 75 cc of sample, good linearity is evident not only in comparing organic samples but in comparing organic with aqueous samples (1 g/l CCW sample gave 360 C/M and 10 g/l CCP sample gave 3750 C/M).

Table III indicates the ability of the neutron counter to monitor an actual process stream. Standard analytical samples were taken from the Recuplex Slag and Crucible filtrate tanks. Before sending the sample to the laboratory the 25 cc polythene sample bottle sealed in plastic was placed in the neutron counting chamber (as in Figure II). A 1.44 g/l sample was used as a standard. All results checked within 0.1 g/l of the laboratory result. All of the filtrate solutions monitored originated from aged solids. When Task I supernates were blended with aged solids in the Recuplex dissolvers, SC filtrate neutron counting results were no longer comparable. As explained earlier

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"Equipment" above, the trouble was too much moderation. A new chamber (Figure III), containing a minimum of moderation between BF_3 probe and sample, was constructed and reasonably good correlations were made.

Table IV shows results when comparing Task I supernates against the same standard used in SC filtrate tests (see Table III). The accuracy (± 0.14 g/l average) was not as good as that indicated in Table III but still sufficiently accurate for critical mass control.

Many other Recuplex samples were tested, such as slurry samples, organic samples, and feed samples. All of these compared favorably with laboratory results, however, it was necessary to establish a new standard in the case of slurry samples, the standard being a normal slurry sample with a known Pu value. Since only sparse amounts of data were available at the time of this writing, no tables were reported.

Waste solids from the Pu purification plant were also monitored in the neutron counter in an effort to charge larger quantities of powders to the Recuplex dissolvers (Ref. HW-46518 (3)) and to mix powders with Task III fragments in a single charge. The method of counting was based upon a "fail-safe" principle. Pure Pu emits only spontaneous neutrons. If the same amount of Pu was in a PuF_4 form, not only the spontaneous emissions, but also the α,n reactions would induce ionizations. Therefore, if relatively pure Pu is used for a standard, any waste from the Pu purification plant must always count on the high side since these wastes contain light element impurities.

To test this "fail-safe" arrangement was adequate for critical mass control, it was desired that the neutron counter might be used for actual accountability of powders originating from the Pu purification plant. For this reason a comparison study was made of monitored values vs. actual values. The actual values were determined by material balance of the solutions from dissolved powders and their residues.

It was apparent that the major variables affecting C/M were the amount of Pu present and the ratio of Pu to total weight of powder. Hence a plot of monitored value vs actual value was constructed with parameters of total powder weight/monitored (or "fail-safe") Pu value. Figure IV depicts curves based on about 50 powder runs. Type of powder was also considered as a variable. After comparing many Task II, Task III, and Task IV powders of similar total weights, it was noted that the portion of the "fail-safe" value due to α,n reactions was approximately the same regardless of type of powder, hence this variable was neglected.

Approximately ten powders were checked using Figure III. Accuracy was within ± 25%. Accuracy by the present method (by difference estimates) varied from a -70 to a +12%.

Despite the favorable results noted in this report, potential obstacles in neutron counting are evident. They are (a) background, (b) light element impurity valuations, and (c) degree of moderation between sample and BF_3 probe. Geometry is incorporated with item c.

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Background variations are caused by a varying neutron flux in the area surrounding the counting chamber and by instrument imperfections. Sufficient moderation between probe and ambient neutron flux can minimize background effects. If this is not possible, frequent background checks using water or some other standard must be made. Faulty BF_3 probes, noisy amplifier tubes, etc., can increase the background. Instrument checks should be made about once per month to keep this source of background at a minimum. In approximately eight months usage of the equipment described in this report, only two BF_3 probes and 3 instrument tubes were replaced.

Varying impurity levels can result in harmful inaccuracies. For critical mass control these inaccuracies are not serious, but for powder accountability or solution analysis (in place of laboratory analysis), varying light element impurity concentrations could minimize the value of the counter for these services.

As noted in the discussion concerning CAW samples, the addition of 0.1 M of a light element, such as fluorine, can increase the C/M reading by 15 - 30% if no fluorine is present to begin with. Increasing the fluorine concentration from 0.1 M to 0.35 M further increases the C/M reading but not linearly (only about 10% average increase). Heavier elements such as aluminum and magnesium have a lesser affect on CAW C/M readings. Fortunately, many Recuplex streams which could benefit by neutron monitors (as a substitute for laboratory analysis) have a constant purity level (e.g., the CCW, organic stripping column effluent).

Purity level in powders is a variable, but the effect of the variable is different from that encountered with solutions. In powders, the Pu concentration varies widely with the total weight of material being counted. Straight PuF_4 powder, for example, is 75% Pu, whereas scrapings from one of the hoods may contain only 0.1% Pu. In solutions, the ratio of Pu to total weight is almost constant for any one type of solution, and even between different type solutions the ratio is small and varies only a few percent.

In pure PuF_4 with the Pu surrounded by the light element fluorine the number of ionizations resulting from α,n reactions is large compared to the ionizations resulting from spontaneous emissions. In powders containing mixtures of fluorides and oxides at low Pu concentrations (0.1% range), for example, the chances of an α,n reaction are limited and the net result is more ionizations from spontaneous emissions than from α,n reactions. Observation of Curve II shows this effect. If the ratio of total powder weight to monitored Pu value ("fail-safe" value) is less than one, about 80% of the C/M reading is due to α,n reactions and 20% due to spontaneous emissions. If the weight/monitored value is 5, about 67% of the C/M is due to spontaneous emissions and 33% to α,n reactions.

Moderation and geometry between probe and sample can change the C/M reading. A liter of powder in a gallon container will give a lower C/M reading than the same powder in a quart container. Both geometry and moderation play a part in creating this difference. Uniformity of sample geometry minimizes this effect.

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Too much moderation can give false readings since many α,n induced ionizations could be eliminated unless (as postulated under discussion on "Equipment") sufficient gamma energy was present to "activate" the α,n reactions. Reduction in moderation between probe and sample minimizes the gamma effect.

Future Work

The work discussed in this report has been of an exploratory nature and more development work is required to substantiate results found thus far.

Definite plans for future experiments with the neutron counter in Recuplex are enumerated below:

1. An experimental probe will be placed in the D-10 slurry tank. A special stainless steel thimble will be made that will be surrounded by at least 3 inches of solution. Only one probe will be used. Slurry solutions of known composition will be placed in D-10 to calibrate the instrument. Later, neutron counter readings will be compared with actual laboratory samples. If this experiment proves successful, probes will be placed in the other slurry tank D-11, the SC accountability tanks, D-8 and D-7, and in a special built tank between the H-3 column and the K-1 and K-2 tanks which will monitor the Recuplex organic streams. It is hoped that the probes in D-10 and D-11 will take the place of analytical samples. The D-8 and D-9 readings will be used for a critical mass control and the probe between H-3 and K-1, K-2, should serve both as a criticality control and as a substitute for analytical samples.

2. Supernates from Task I will be monitored by an in-line neutron counter. This instrument will be set up in Task I along with an alpha counter (already under test) for comparison studies.

3. Further experiments using the present test set-up (chambers as in Figure II and Figure III) will be made. Higher Pu concentrations of CCP solutions will be tested to evaluate the feasibility of substituting a neutron counter for the present gamma absorptometer.

Other items that should be investigated are:

1. The ability of the neutron counter to replace the present system of determining the Pu in all dry solids.

2. The effect of spiking the neutron chamber with an impurity such as an aluminum fluoride complex. A procedure such as this may minimize the effect of impurities.

3. Further background and moderation studies should be made.

Acknowledgements

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Department, and Process Control Development, Hanford Laboratories Operation in their assistance as to equipment, working area, and advice. Instrument Technician work was done by C. F. Setbacken. Data was compiled and studied by H. P. Maffei of the Finished Products Technology Operation and the writer.

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3. Smith, R. E., "Multiple Batching of Recuplex Powders by a Neutron Counting Technique," HW-46518.

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TABLE I

NEUTRON MONITORING OF RECUPLEX SIMULATED EXTRACTION WASTE SAMPLES -

CAW (0.8 M ANN, 2.5 M HNO_3)

Gain Setting / Discriminator Setting		Not Recorded / 68			5 / 68			2 5/8 / 68		
		Counts per Minute			Counts per Minute			Counts per Minute		
SOLUTION	Pu (g/l)	Background	Total	Sample	Background	Total	Sample	Background	Total	Sample
CAW	0.1	-	-	-	144	161	17	-	-	-
CAW	0.5	96	105	9	144	186	42	56	70	14
CAW	1.0	84	105	21	144	207	63	56	92	36
CAW	1.0	102	126	24	144	216	72	52	90	38
CAW	1.0	-	-	-	-	-	-	50	93	43
CAW	2.0	87	126	49	144	276	132	54	134	80
CAW	2.0	126	158	52	-	-	-	-	-	-
CAW	3.0	90	147	57	-	-	-	54	180	126
CAW	3.0	99	162	63	-	-	-	52	162	110
CAW + 1.0 M ANN	1.0	90	117	27	141	225	84	51	100	49
CAW + 0.1 M NaF	1.0	119	93	26	144	237	93	52	109	57
Same	1.0	-	-	-	-	-	-	51	103	52
CAW + 0.35 M NaF	1.0	87	114	27	144	249	105	51	109	58
Same + 0.5 M $Mg(NO_3)_2$	1.0	90	117	27	141	228	87	52	116	64
Same	1.0	-	-	-	-	-	-	51	103	52
5 M HNO_3	1.0	81	102	21	138	198	60	51	91	40

Equipment: Chamber as in Figure I.

Samples: Contained in ½-inch stainless steel tubes, 14 inches long. Volume of sample - 40 cc.

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TABLE II

NEUTRON MONITORING OF RECUPLEX ORGANIC AND PRODUCT SAMPLES

Equipment: Chamber for counting samples as in Figure II.

Samples: Contained in 3/4-inch, 14-inch long stainless steel tubes. Volume of samples - 75 cc.

Solutions: (a) Recuplex organic spiked with Pu.

(b) Synthetic low concentration CCP.

SOLUTION	Pu (g/l)	BACKGROUND (C/M)	TOTAL C/M	SAMPLE C/M
15% TBP - 85% CCl_4	0.08	27 (a)	54	27
15% TBP - 85% CCl_4	0.1	36	75	39
15% TBP - 85% CCl_4	0.5	36	210	174
15% TBP - 85% CCl_4	1.0	277 (b)	637	360
100% CCl_4	1.0	277	700	423
CCP	10.0	600 (c)	4250	3750
CCP	20.0	600	8300	7700

NOTES: ( ) Scale was 0 - 300

(b) Scale was 0 - 1500

(c) Scale was 0 - 10,000

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TABLE III

NEUTRON MONITORING OF RECUPLEX SC FILTRATE SAMPLES

Equipment: Chamber as in Figure I.

Samples: 10 - 25 cc. of sample in polythene bottles.

Solutions are actual samples of Recuplex filtrate solutions.

Standard: 25 cc. of a 1.44 g/l SC filtrate solution.

Lab. Result (g/l)	Monitor Result (g/l) (a)
0.298	0.25
0.400	0.41
0.368	0.31
0.526	0.53
1.44	1.44 (b)

NOTES: (a) All samples were corrected for background and size of sample.

(b) Standard 1.44 g/l SC filtrate solution.

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TABLE IV

NEUTRON MONITORING OF TASK I SUPERNATE SAMPLES

Equipment: New chamber as in Figure III.

Samples: 10 cc. samples in polythene bottles.

Standard: 25 cc. of a 1.44 g/l SC filtrate solution.

Lab. Result (g/l) (a)	Monitor Result (g/l)
0.28	0.35
0.13	0.29
0.18	0.37
0.34	0.51
0.28	0.42
0.27	0.42
0.34	0.42
0.17	0.33
0.31	0.44
1.44 (b)	1.44

NOTES: (a) All samples were corrected for volume and background; however, background deviation was small.

(b) Standard 1.44 g/l SC filtrate solution.

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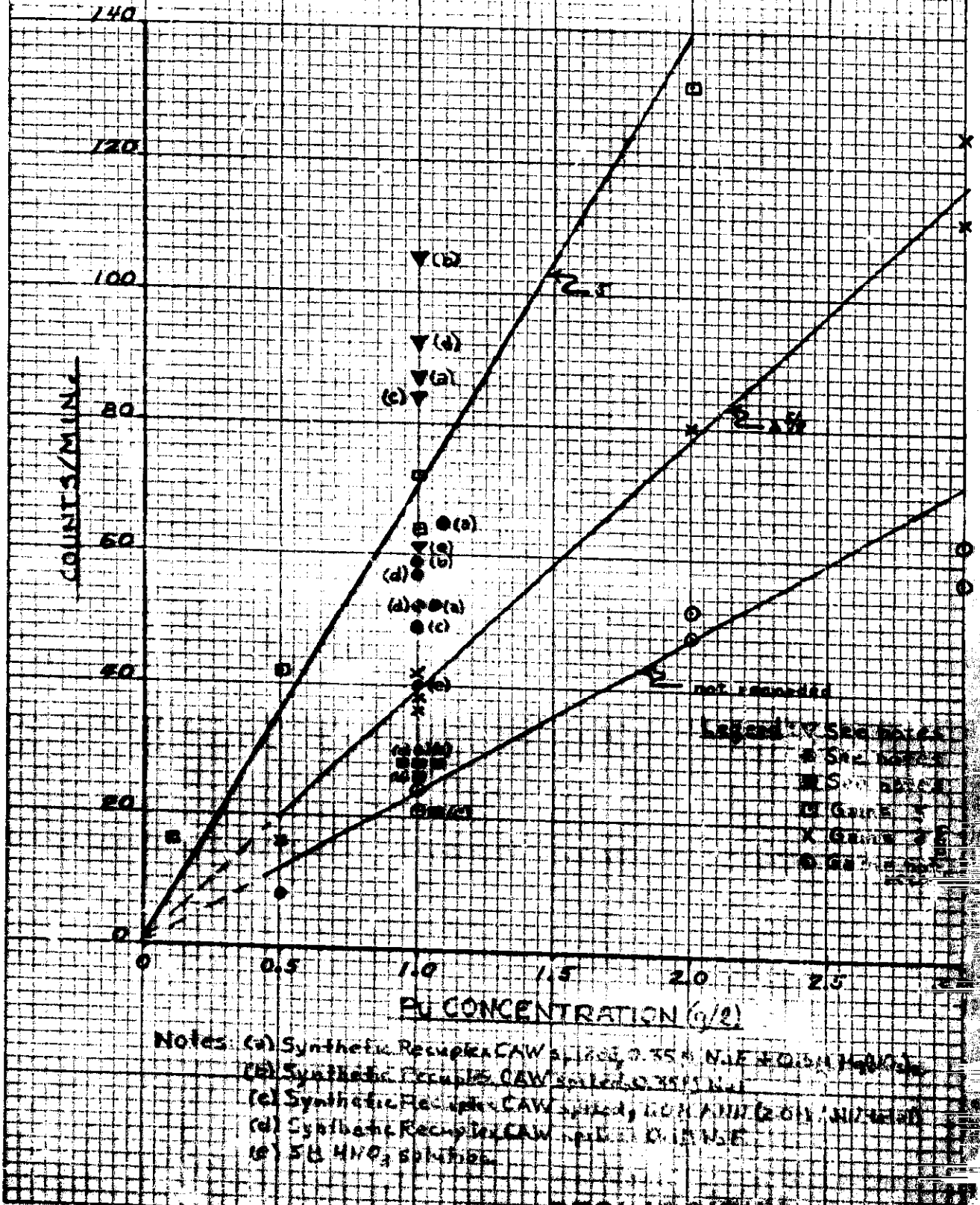
FIG. 1

Pu AND IMPURITY (SYNTHETIC SOLUTIONS) VS. COUNTS/MIN.



Notes: (a) Synthetic Recuplex CAW spiked 0.35% N.I.E. in 0.5M HNO_3

(b) Synthetic Recuplex CAW spiked 0.35% N.I.E.

(c) Synthetic Recuplex CAW spiked 0.01% N.I.E. in 0.5M HNO_3

(d) Synthetic Recuplex CAW spiked 0.01% N.I.E.

(e) 5M HNO_3 solution

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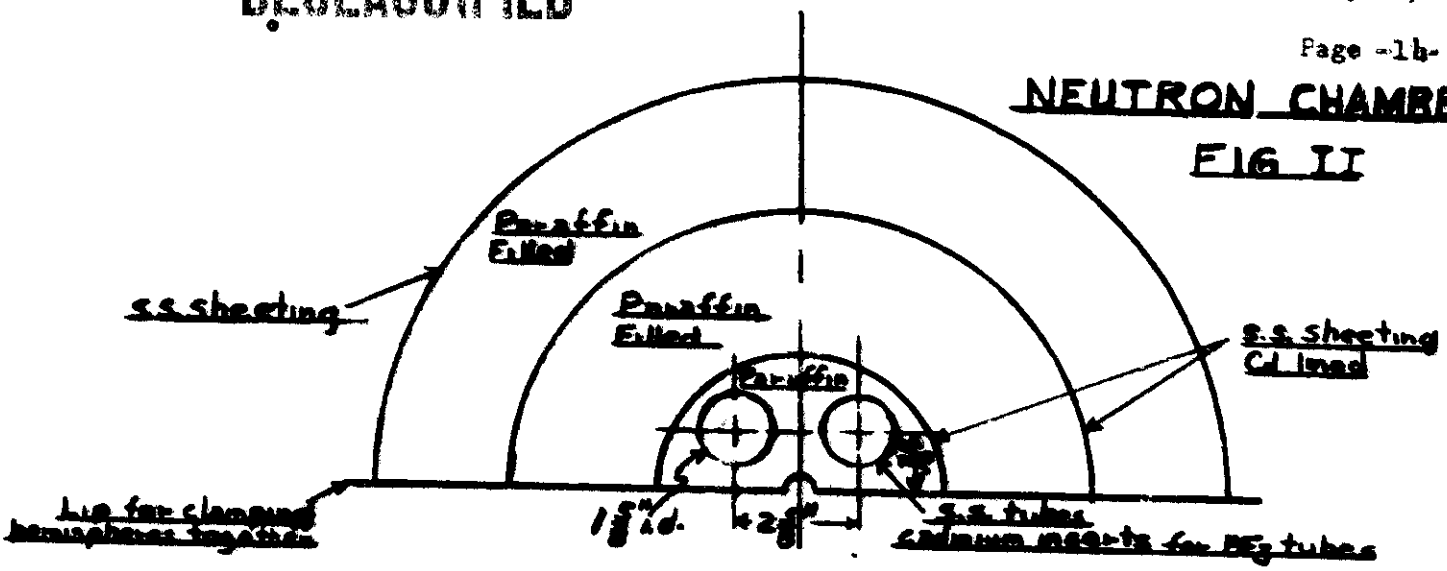
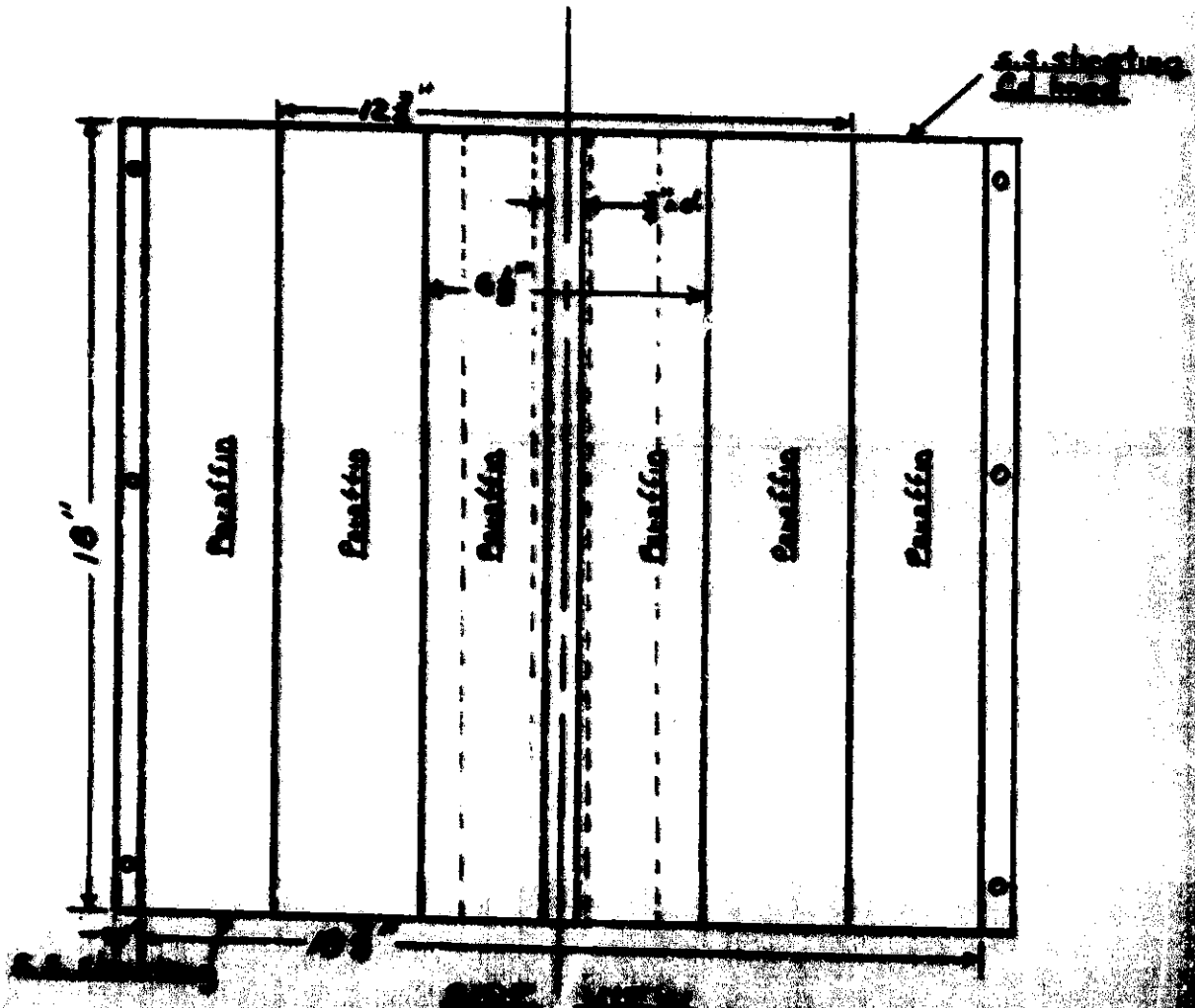
NEUTRON CHAMBER

FIG II



TOP VIEW
ONE OF TWO IDENTICAL HEMISPHERES



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NEUTRON CHAMBER
FIG. III

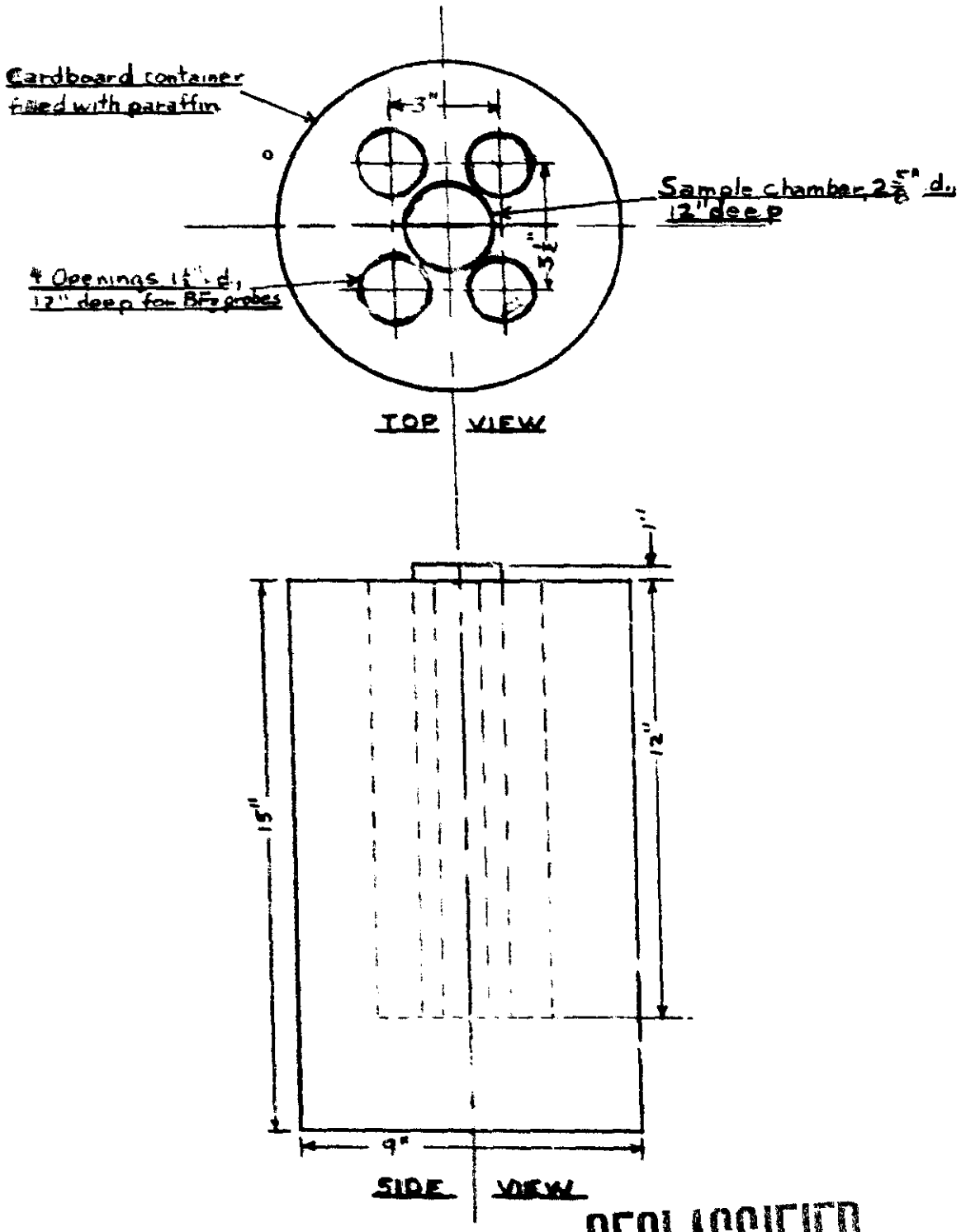

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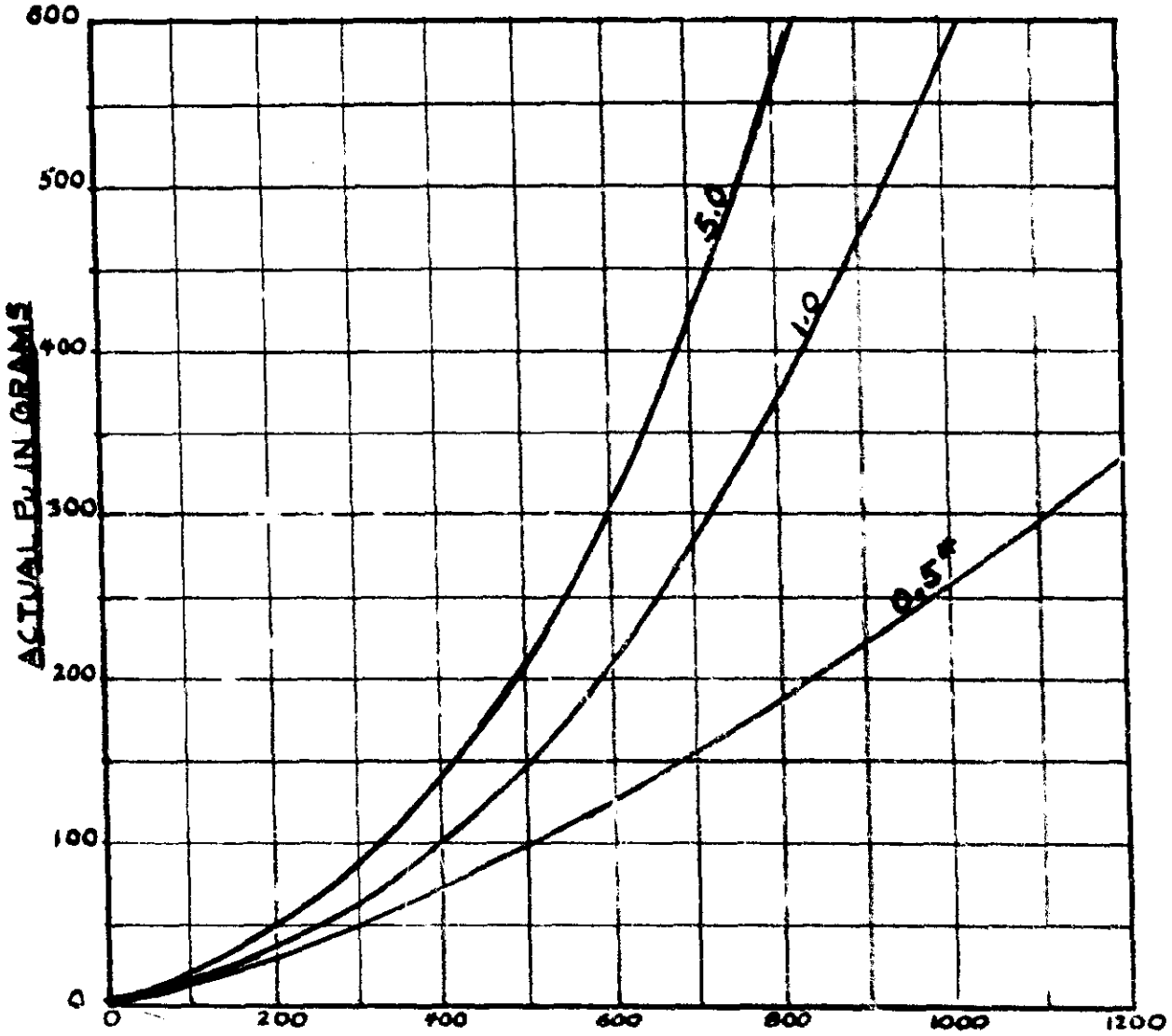
FIG IV

ESTIMATED Pu IN DRY SOLIDS AS DETERMINED BY THE NEUTRON COUNTER



* Parameters are total weight of solids in grams divided by the monitored Pu in grams.

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