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RECUPLEX FEASIBILITY REPORT

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PROCESS ASSISTANCE
SEPARATIONS TECHNOLOGY UNIT
TECHNICAL SECTION, ENGINEERING DEPARTMENT
HANFORD WORKS

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I. INTRODUCTION.

The Recuplex Process is proposed by the Process Assistance group of the Separations Technology Unit as a feasible method for the simultaneous and efficient recovery of waste materials in the 234-5 Building and direct coupling of the product streams from the Redox and Bismuth Phosphate Processes to the 234-5 Building operations. Demonstration of the feasibility of the process has been accomplished through the cooperative efforts of the Chemical Research, Chemical Development, and Process Assistance groups of the Separations Technology Unit.

A. Historical Background.

The Recuplex Process originated as a possible solution to the problem of recovering product from one of the waste streams in the Purification and Fabrication Building. Metallic plutonium is obtained in the 234-5 Building by the reduction of plutonium tetrafluoride in a magnesium-oxide crucible. After removal of the metal "button", the crucible and its associated slag contain approximately three percent of the total plutonium in the processed batch and should therefore be prepared for recycling through the main processing stream. At the start of 234-5 Building operations, however, a suitable means of treating the slag and crucible for product recovery had not been developed, and the decision was made to store the material until such a process was available. This procedure is still in effect.

An investigation of the methods of treating the reduction crucibles and their associated slag was undertaken by the Chemical Research group of the Separations Technology Unit in the fall of 1950.(7,8,14) It was recognized that the preparation of a recycle solution suitable for blending with the regular plant process-streams involved a dissolution step and a separations step. Of the four dissolving procedures tested in the laboratory, the most desirable appeared to be the total dissolution of the slag and crucibles in nitric acid followed by the addition of aluminum nitrate to complex the fluoride ion. Although it was chemically feasible to recycle the dissolved slag-and-crucible solution to the Redox 2A column or to the Product Precipitation step in the Second Decontamination Cycle of the Bismuth Phosphate Process, an independent extraction operation presented the advantage of returning the stream directly to the 234-5 Wet-Chemistry step with a low plutonium loss. The proposal was therefore made to purify and concentrate the plutonium solution by a solvent-extraction process utilizing tributyl phosphate in carbon tetrachloride as the selective solvent.

The growth of the proposed slag-and-crucible recovery process to a process involving five plutonium-bearing streams resulted from problems associated with the anticipated start of Redox operations. The question was raised as to the most advantageous method of introducing the Redox product stream into the present Hanford operating plants. Among the possible coupling processes considered feasible were plutonium peroxide precipitation, plutonium oxalate precipitation, solvent extraction, ion exchange, and modifications of the current Bismuth Phosphate Process.(21) The similarity of the product streams from the Redox and Bismuth Phosphate Plants to the slag-and-crucible dissolver solution presented the possibility of employing a separate solvent-extraction facility for the simultaneous performance of the recovery and coupling operations. The proposed unit was further expanded by the addition of two recycle streams - the supernates from the 234-Building Wet-Chemistry step, and the skulls and miscellaneous wastes from the 235-Building operations, including such materials as grit blast, hand cleaning wastes, impure buttons from hood sweepings, etc. The simultaneous purification and concentration of the two product streams and three recycle streams to provide a solution suitable for the 234-5 Building Wet-Chemistry operations was termed the Recuplex Process.

B. Description of the Recuplex Process.

The proposed Recuplex Process may be divided into five phases of operation: (1) feed preparation, (2) feed reception and blending, (3) solvent extraction, (4) solvent treatment, and (5) waste treatment.

(1) Feed Preparation.

Of the five plutonium-bearing streams treated in the Recuplex Process, only one required special development work for the preparation of a suitable feed solution. The 2BP stream, product of the Redox Plant, can be received directly into the Recuplex Process. The F-10-P stream, product of the Bismuth Phosphate Process, contains an oil emulsion and solid contaminants which appreciably increase the disengaging time of the aqueous and organic phases in the extraction column. The solution must therefore be subjected to a clarification step prior to its delivery to the extraction column. This can be accomplished by contacting the F-10-P stream with carbon tetrachloride or by a filtration operation similar to that presently employed in the 231-Building. Solution preparation of the 234 supernatant stream and the 235 skulls-and-waste stream has previously been developed, the former involving treatment with hydrogen peroxide followed by an evaporation step,(1) and the latter requiring dissolution in a nitric acid-hydrofluoric acid mixture.(3) A research-and-development program conducted with slag-and-crucible material has established that a satisfactory aqueous feed stream can be prepared by dissolving the solid material in nitric acid, adding a fluoride-ion complexing agent, and filtering the resultant solution to remove the undissolved silica. The slag-and-crucible filtration step is performed with a 20% dilution (using prepared supernatant solution) and must take place at a temperature of approximately 80°C in order to reduce the viscosity of the solution and prevent the formation of solid magnesium nitrate.

(2) Feed Reception and Blending.

Prior to actual delivery to the extraction column, the two product and three recycle streams are blended and the concentrations adjusted in two separate make-up facilities: one having a plutonium concentration of approximately 10 grams per liter and the other having a plutonium concentration of approximately 0.3 grams per liter. Although the two streams are later mixed and enter the extraction column as one feed stream, the blending operation is divided into two phases to provide the maximum feed-tank capacity and operating facility within critical mass considerations. The concentrated stream comprises the 2BP, F-10-P, and skulls and metal-fabrication waste solutions, blended in the approximate ratio of 24:12:1 (depending upon relative plant production rates). The two constituents of the dilute stream, the slag-and-crucible solution and Wet-Chemistry supernatants, are blended in the

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approximate ratio of 32:7. Blending of the solutions must be accompanied by the addition of suitable reagents to adjust the solution acidity and the plutonium valence state. Aluminum nitrate nonahydrate, commonly termed ANN, is added to the concentrated stream as a salting agent to insure optimum column operation. The two streams are then proportionately pumped to the extraction column where they enter as one combined feed stream.

(3) Solvent Extraction.

The plutonium feed stream rises through a pulse column twenty-nine feet high, in which the extraction and scrubbing operations are performed, and is then pumped to a second twenty-nine foot pulse column for the stripping operation. The three operations serve to remove undesirable contaminants from the solution and increase the plutonium concentration to 50-60 grams per liter to permit its transfer directly to the 234-5 Wet-Chemistry step.

In the extraction step, the feed stream is intimately contacted with a selective solvent (15 per cent by volume of tributyl phosphate in carbon tetrachloride) resulting in the mass transfer of plutonium from its aqueous solvent to an organic solvent. The major portion of the solution contaminants remains in the aqueous phase and is discharged as waste. Prior to its passage to the stripping column, the organic phase is scrubbed with an aqueous solution of nitric acid to remove some of the contaminants originally carried over from the feed stream. The operation of the extraction-and-scrubbing column is continuous and countercurrent. The organic solvent enters at the top and descends through the column as the discontinuous phase. The aqueous scrub solution is fed into the bottom of the column, while the plutonium feed stream enters immediately above the scrubbing section. Intimate mixing of the two phases is achieved at each of a series of perforated plates. This is accomplished by an external pulse generator, which serves to repeat the passage of a given liquid volume through each plate. The number of passages is determined by the amplitude and frequency of the pulse stroke. The immiscibility of the two phases and the difference in density result in the discharge of the aqueous waste stream at the top and the organic product stream from the bottom.

The stripping operations serves to transfer the plutonium from its organic solvent to an aqueous solution with a simultaneous increase in plutonium concentration. The continuous aqueous discharge from the stripping column is the product of the proposed Recuplex Process and does not require further treatment prior to its shipment in batch quantities to the final Purification and Fabrication Processes.

(4) Solvent Treatment.

The organic waste from the stripping column may be recycled through the process approximately 100 times without effecting column performance. The gradual accumulation of DBP from alpha bombardment and hydrolysis of tributyl phosphate, however, necessitates periodic treatment of the solvent. It has been determined that effective removal of the organic contaminants may be accomplished by contacting the waste discharge from the stripping column with a dilute aqueous solution of sodium hydroxide.

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(5) Waste Treatment.

The proposed Recuplex process discharges three waste streams: the silica cake from the filtration step in the slag-and-crucible solution preparation, the aqueous waste stream from the extraction-and-scrubbing column, and the dilute caustic stream from the solvent-treatment tower. If the plutonium content of the silica cake is 10 per cent, or less, of the total plutonium in the original material, the cake will be slurried and transferred to underground storage. If the loss exceeds 10 per cent, the cake can be treated with hydrofluoric acid in the platinum-lined skull dissolver and the resulting solution returned to the Recuplex Process. It is believed that the proposed dissolving procedure will result in greater than 90 per cent recovery and that additional processing of the silica cake will not be necessary. The upper limit of waste loss to the aqueous discharge from the extraction column is 0.2 per cent. Material having a higher plutonium content can be recycled through the extraction and scrubbing steps, although such a procedure would require a temporary column shutdown. Waste extraction solutions having a lower plutonium content are neutralized and sent to underground storage. The proper disposition of the dilute caustic waste stream from the solvent-treatment tower has not as yet been determined. It is believed that a procedure for the recovery of plutonium from this stream can be developed although the anticipated loss is extremely small.

C. Advantages of the Recuplex Process.

The Recuplex Process has five inherent advantages, each of which materially effects the operating efficiency and economy of the Hanford Works.

(1) Rangeability of Operation.

The character of the present design and development proposals for the Recuplex Process is such as to provide extreme operating flexibility and permit extensive process changes. The objectives of the proposed process are dynamic rather than static, and recognize possible variations in the quantities and compositions of the streams to be treated in the event of future plant developments. Among the possible process changes that could be accommodated by the Recuplex Process are: (1) plant capacity expansion to the production level of 100-120 Kg/mo., (2) complete shutdown of the $BiPO_4$ plants, (3) direct coupling of Redox to 234-5 Building Wet Chemistry, and (4) improvements in the 234-5 Building operations that will effect the nature and amount of process wastes. Decisions relative to these process changes are not necessary for the successful operation of the proposed recovery-and-coupling facilities. Furthermore, the process is of such a nature that it can be brought into the line of production processing in a step-wise manner. Production commitments can be accommodated by existing facilities and do not have to be bypassed or shutdown until good assurance of success following shakedown is evident.

(2) Increased Efficiency in 234-5 Building Operation.

Product recovery from dry wastes (slag and crucible, off-standard metal, skulls and miscellaneous cleaning residues) would permit the conversion of

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incoming plutonium to final output form to increase from 94 to 99+ per cent. The continuous processing of such material would also eliminate the gross storage problem that presently exists. In fact, it is proposed that the Recuplex processing equipment be contained in the room now allocated to the storage of waste materials.

(3) Independence of 234-5 Building Operation.

Recovery of all the 234-5 Building waste materials by the one process would result in the efficiency gains inherent with independent plant operation. Continual operation of the 234-5 Building would not be contingent upon uninterrupted operation of the $BiPO_4$ or Redox Plants, as it is with respect to the present practice of recycling the oxalate supernatants to 224 Building.

(4) Reduction of Fixed Overhead.

The batchwise filtration and precipitation steps presently carried out in the 231 Concentration Building can be entirely eliminated. The simultaneous concentration of two product streams and purification of three recycle streams represents an efficient consolidation of separate operations with the accompanying reduction in processing time and materials handling.

Placement of the operating areas in the 231 Building on a standby basis would result in an appreciable reduction of fixed overhead in the plant operating costs. In the event of complete shutdown of the 231-Building, the annual saving is estimated to be $1,200,000 less the direct operating cost of the Recuplex Process.

(5) Minimum Process Design Effort.

The Recuplex Process achieves efficiently and simultaneously a multiple process design goal with a minimum amount of design effort.

D. Recuplex Process Development Program.

The demonstration of the Recuplex Process as a feasible method for the recovery of waste materials in the 234-5 Building and the coupling of the Redox and 224 Building product streams to the 234-5 processes was completed with respect to major points within a three-month period ending November 1, 1951. Such action was essential due to the anticipated start of Redox operations and the consequent necessity for providing an increase in the capacity and efficiency of various operations in the 231 and 234-5 Buildings. It was felt that, in the event the Recuplex Process should prove impracticable, a development-and-design program for other facilities could not be deferred indefinitely beyond this date.(2)

(1) Program Goals.

In order that a decision as to the feasibility of the Recuplex Process might be reached within the allotted period of time, a process development program was initiated having three principal goals: (1) definition of the process flowsheets to a degree which would demonstrate the yield and purities to be expected, (2) definition of the process equipment requirements with respect to size, type, and materials of construction, and (3) demonstration by equipment-arrangement studies of the adequacy of the space available within the 234-Building to accommodate the processing equipment.

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(2) Division of the Development Work-Load.

The comprehensive nature of the investigation and the brevity of the available time necessitated the cooperative efforts of three groups within the Separations Technology Unit: Chemical Research, Chemical Development, and Process Assistance, the sponsoring group. By delegating the work-load among the three groups, the principal topics of the investigation requiring laboratory, pilot-plant, and engineering studies were satisfactorily accomplished.

II. SUMMARY.

The Recuplex feasibility investigation comprised five major studies: (1) the dissolution and filtration of slag-and-crucible material; (2) the extraction and stripping of the combined plutonium-bearing feed streams; (3) the treatment and disposition of the process waste streams; (4) the anticipated corrosion effects; and (5) the equipment required and the adequacy of the available space in the 234-5 Building to accommodate the processing facilities. The Recuplex Process was designed to process virgin material, with its associated recycle streams, at an instantaneous production rate of 100 Kg/mo. Sufficient flexibility was incorporated into the design to permit efficient operation at lower rates or, with minor equipment changes, at production rates up to 120 Kg/mo. Design goals were: (a) the recovery of not less then 90 per cent of the plutonium present in the slag-and-crucible residues, (b) a minimum recovery of 99.8 per cent and a normal recovery of 9> 99 per cent of the plutonium in the streams treated in the solvent-extraction columns, (c) the preparation of a low acidity (<2.0 N) purified and concentrated product stream suitable for direct shipment to 234-5 Wet-Chemistry operations, and (d) the installation of the requisite processing equipment within the space available in the 234-5 Building.

Two methods for the dissolution of the plutonium present in the slag-and-crucible material were investigated. The first, based upon the recovery flowsheet presently employed at the Los Alamos Scientific Laboratory, necessitates essentially three separate procedures to recover 90 per cent of the plutonium in the material: (1) dissolution in $Al(NO_3)_3 \cdot 9H_2O$ and HNO_3 followed by filtration; (2) treatment of the undissolved silica cake with HF and dissolution of the residue with HNO_3; and (3) periodic dissolution of the accumulated heel in the dissolver vessel with hydriodic acid. The second method, developed at the Hanford Works, accomplishes greater than 90 per cent recovery by the dissolution of the plutonium in a HNO_3-HF mixture. This method eliminates the dissolver heel and the need for any further treatment of the silica residues. The dissolving procedure, however, presents a corrosion problem that has not as yet been fully investigated. There are presently three satisfactory approaches to the problem: 1) adjustment of the chemistry to provide satisfactory dissolution in a non-corrosive environment; 2) development of special materials of construction for increased resistivity to chemical attack under dissolving conditions; and 3) periodic replacement of the dissolver. Preliminary data indicate that the corrosion rates with presently available stainless steels would make dissolver replacement economically feasible. The operating conditions developed in the Hanford Works laboratories are based upon dissolvings performed on the scale of one reduction crucible and its associated slag. Development of the optimum conditions for multi-crucible dissolvings can best be performed in the final process equipment, since the construction of the necessary semi-works facilities would entail an installation essentially the same size as the plant unit with the associated problem of space location.

Laboratory batch-equilibration tests have demonstrated that the combined, plutonium feed streams to the Recuplex Process can be recovered with a solvent-extraction waste loss of less than 0.2 per cent, purified, and concentrated to the degree necessary for direct transfer to the 234-5 Wet-Chemistry operation. Two chemical flowsheets were developed from the laboratory tests and are presented as alternate and satisfactory processing schemes for meeting the Recuplex design goals. The difference between the two solvent-treatment procedures is the manner in which the plutonium is stripped from the organic phase: in the first method a plutonium-complexing sulfate salt is employed, while in the second the plutonium is concentrated in the extraction-and-scrubbing column by a reflux operation which permits adequate stripping without the use of a complexing or reducing agent. The latter procedure permits greater flexibility in column operation and is especially desirable in view of the fact that the feed streams to the Recuplex Process will vary in their quantities and composition.

Pilot-plant investigations of contactors having suitable operating and physical characteristics involved the Fenske stacked extractor and pulse columns. Test data demonstrated that both types of contactors can successfully meet the design requirements for product concentration and purity, and for solvent-extraction waste losses. The more reproducible capacity exhibited by the latter and the greater flexibility in handling streams varying in quantities and contents indicated the pulse columns to be more suited for use in the Recuplex Process. Moreover, the relatively complex structure of the Fenske assembly would result in greater costs and a larger time-consumption for maintenance operations or actual replacement. Although the use of the Fenske contactors would not require extension of the units through the ceiling of Room 221 in the 234-5 Building, the space necessary for the pulse columns can be provided without undue construction difficulties and without interfering with present processing operations. On the basis of these considerations, the pulse column is recommended as the more satisfactory and reliable unit.

Studies of the probable plutonium losses to the three waste streams discharged by the Recuplex Process indicate that the process design waste limits will not be exceeded. It is believed that a one-step slag-and-crucible dissolution procedure can be developed which will permit a recovery yield of greater than 90 per cent and eliminate the need for further processing of the silica residues. In this event, the silica cake from the solution filtration step will be slurried and transferred to underground storage. If the process should be economically unsound, there are three available alternatives for processing the silica residue: (1) storing the silica cake for later treatment by modifications to the Recuplex Process, (2) processing the cake in the Hot Semi-Works Plant, and (3) treating the cake with hydrofluoric acid on a semi-routine basis in the platinum-lined skull dissolver and returning the resultant solution to the Recuplex Process. Proposed improvements in 234-5 Building operations would alleviate the silica residue problem, as for example, the reduction in the amount of SiO_2 resulting from the use of thin-walled crucibles and the possible change in crucible composition.

Batch equilibration data indicate that the plutonium loss to the aqueous waste stream discharged from the extraction-and-scrubbing column will be in the order of 0.02 to 0.1 per cent. Neutralization of the solution results in the formation of a large volume of gelatinous solids. Since separation of these solids (by centrifugation) does not decontaminate the supernatant solution sufficiently to permit cribbing, it is recommended that the alkaline slurry be transferred directly to underground storage. Approximately 10,000 gal/mo. of the neutralized extraction waste stream (volume of unneutralized waste 8,000 gallons) will be sent to storage. Facilities have been included in the preliminary equipment design for recycling the stream in the event that the Pu loss exceeds 0.2 per cent.

Laboratory tests have demonstrated that continual exposure of the organic solvent to alpha bombardment results in the gradual decomposition of tributyl phosphate and the fixation of small amounts of plutonium. The plutonium compound formed in this manner is extremely insoluble in water and can not be stripped from the organic phase by either nitric acid or a sulfate salt. The anticipated plutonium loss to the organic waste stream discharged from the stripping column is approximately 0.03 per cent. Test data indicate that after 100-hours contact time or approximately three months operation at 100 Kg/mo. the organic solvent may be efficiently treated with 5 per cent sodium hydroxide and returned to the process system without any detrimental effects upon column operation. Due to the extremely small volume ratio of aqueous caustic to organic solvent (1:20) necessary for effective treatment, the concentration of "unstrippable" plutonium in the caustic waste stream will be approximately 4 g/1. Although this represents an extremely low waste loss, it is believed that suitable methods for its recovery and final disposition can be readily developed.

A study of the corrosion effects to be anticipated in the Recuplex equipment demonstrated that the resistance of available stainless steels is sufficient throughout the thermally cold portion of the system. The corrosion problem under slag-and-crucible dissolution conditions has not as yet been fully investigated. Preliminary data indicate that the corrosion rates will available stainless steel materials are not overly excessive (0.0008 - 0.0037 in/mo) and that periodic replacement of the dissolver vessel would be economically sound.

The engineering design basis chosen for sizing of the Recuplex equipment was a production rate of 80 Kg/mo. with an allowance of 20 per cent for down-time, or an instantaneous processing rate of 100 Kg/mo. The capacities of the process vessels were based upon a minimum of 12-hours hold-up and an allowance of at least 20 per cent freeboard. Greater hold-up capacities (12-48 hours) were incorporated at potential "bottlenecks" within the system. To insure continuity of operation, some of the equipment pieces were duplicated. The storage and processing of the concentrated plutonium solutions necessitated special design of certain vessels to prevent the possibility of exceeding critical-mass conditions. Vessels designed to accommodate batch solutions containing plutonium at concentrations in the order of 10 g/1 were limited to eight inches in tank diameter or a volume capacity equivalent to 1000 grams of plutonium. The minimum edge-to-edge distance between these tanks is twelve inches. Containers for the purified and concentrated product stream were designed to be infinitely safe, id est, the geometry of the tanks is that criticality conditions cannot be exceeded regardless of the plutonium concentration or the total quantity of liquid present. Such tanks permit sufficient neutron escape through the sides to insure critically safe conditions. The tanks have been designed with a rectangular shape, one dimension of which is a maximum of eight inches and the other two unlimited. The tanks can be arranged within the hood with no restrictions as to their edge-to-edge distance if they are contained within two parallel planes separated by the eight-inch dimension. Plutonium-bearing vessels located outside the infinite volume defined by these planes must be protected from neutron absorption by twenty-four inches of air or its equivalent.

The contamination hazards associated with handling plutonium solutions necessitated housing the process equipment in such a manner as to protect operating personnel. The two types of process hoods, Barrier and Greenhouse, which are presently employed in the Separations Area were evaluated with respect to relative construction costs, utilization of floor space, accessibility of process vessels, ease of equipment maintenance, operating convenience, operating efficiency, and personnel protection. The analysis was made on the basis of operating experience obtained with both types of hoods and established that the Greenhouse pattern was the more advantageous. Although the design of housing for the Recuplex Process was based primarily upon the Greenhouse plan, an attempt was made to incorporate the desirable features of both patterns in an improved hood design.

Four engineering flow sketches have been prepared based upon the process equipment requirements and the chemical flowsheets. Each diagram depicts one or more phases of the Recuplex Process and represents all the equipment to be associated with an individual process hood. Briefly, the four hoods contain: (1) the equipment for preparing slag-and-crucible solution, (2) a revision of the present equipment for treating the Wet-Chemistry supernatants, (3) the equipment for receiving and blending the column feed streams, and (4) the equipment for the solvent-extraction, solvent-treatment, column-waste-treatment, and product-storage operations. Duplication of key equipment, spare pumps, overflow tanks, and vacuum traps were incorporated into the system to insure safe and continuous operation.

A prime factor in the feasibility of the Recuplex Process is the containment of the processing facilities within the 234-5 Building. The construction of a new and separate building, or an addition to the Purification and Fabrication Building, would seriously limit the potential economic advantages of the Recuplex installation. There is presently available within the 234-5 Building unoccupied floor space which is suitable for additional chemical make-up facilities (Room 337) and a limited amount of room suitable for process operations (Room 221). Equipment arrangement studies based upon the engineering flow sketches have indicated that this space can satisfactorily accommodate the Recuplex assembly. Sketches have been prepared to indicate the plan and elevation views of the most suitable arrangements of the process vessels and the accompanying instrumentation. Room 337 in the 234-5 Building can accommodate the reagent-preparation vessels required in the Recuplex Process and still permit future expansion of the present make-up room (adjacent to Room 337) in the event of other plant needs. Room 221 presently houses the supernatant-recovery hoods and the skull-recovery hood, each of which follows the Barrier-type construction. Two arrangement patterns have been developed to contain the Recuplex processing equipment within the room. One method involves the replacement of the two Barrier-type supernatant hoods with one revised and consolidated Greenhouse-type hood; the other would employ the present supernatant-recovery hoods in the Recuplex Process. In both plans the reception-and-blending hood and the hood for the solvent-treatment operations are of the Greenhouse-type construction, the latter having a special shroud extending through the ceiling to house the pulse columns. The installation of the shroud can be accomplished without undue construction difficulties and without interfering with present processing operations. The location of the barrier for the skull-recovery hood, and the contamination problems associated with handling the slag-and-crucible material and the periodic replacement of the dissolver vessel, suggested the extension of the barrier for the skull hood and the construction of the slag-and-crucible hood according to the Barrier pattern.

Employing the present supernatant-recovery hoods in the Recuplex assembly requires either the expansion of Room 221 to include a portion of the adjacent locker rooms or the acceptance of an appreciable reduction in the working space between the process hoods. It is believed that replacement of these hoods with one, consolidated Greenhouse hood permits a more efficient and more advantageous use of the available floor space and will result in greater operating facility and efficiency. The equipment-arrangement plan involving a revised supernatant-recovery hood is therefore recommended as being the more suitable and advantageous for the Recuplex Process. Very rough preliminary estimates indicate that the cost of installing the Recuplex Process would be approximately $1,300,000. The value of the increased plant production, from the recovery of the slag-and-crucible material alone, would equal the installation cost in approximately 13 months operation at present plant capacity (assuming the value of metallic plutonium is $100 per gram). It is estimated that a detailed project proposal will require 8 to 12 man-months of engineering effort. In the event the project is approved, the completion date for equipment fabrication and installation will be primarily dependent upon material procurement.

III. CONCLUSIONS

The Recuplex Process has been demonstrated through the cooperative efforts of the Chemical Research, Chemical Development, and Process Assistance branches of the Separations Technology Unit to be a feasible method for the simultaneous and efficient recovery of waste materials in the 234-5 Building and direct coupling of the product streams from the Redox and Bismuth Phosphate Processes to the 234-5 Building operations. The design goals of greater than 90 per cent recovery of the plutonium in the slag-and-crucible waste stream and the purification and concentration of the five plutonium-bearing feed streams to a degree permitting direct shipment to Wet-Chemistry operations (with a solvent-extraction waste-loss less than 0.2 per cent) can be attained. Methods have been established, for the satisfactory disposition of the three waste streams. A further refinement of the treatment for the caustic wash of the organic solvent is in the process of development. The corrosion rates of presently available stainless steels are fully satisfactory in all thermally cold processing vessels. The higher corrosion rates obtained in the slag-and-crucible dissolver, with the HNO_3-HF solution, can be adequately met by one of the following measures: adjustment of the chemistry to provide satisfactory dissolution in a non-corrosive environment, development of special materials of construction with increased chemical resistivity, or periodic replacement of the dissolver. The proposed process recognized future expansion of plant -

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capacities and is sufficiently flexible to process varying feed-stream quantities and compositions over the full range of production rates up to 120 Kg/mo. The processing equipment can be contained within the 234-5 Building. It will be necessary, however, to maintain close coordination between design layout and the proposed supplementary development program.

IV. RECOMMENDATIONS

In view of the favorable results of the Recuplex feasibility investigation, it is recommended that the following steps be taken leading to the installation of a Recuplex-Process facility in Building 234-5: (1) the procurement of further detailed chemical and engineering information proceeding concurrently with (2) the preparation of a detailed Project Proposal, and following authorization, (3) the preparation of a detailed engineering design.

V. DISCUSSION

A. Design Basis.

In order to allow for the future expansion of plant capacities, the basis chosen for the development and design of the Recuplex Process was a production rate of eighty kilograms of plutonium metal per month with an allowance of 20 per cent for down-time, or an instantaneous rate of 100 Kg/mo.(2) The process, however, is sufficiently flexible to permit operation at an instantaneous production rate of 120 Kg/mo. with only a few minor equipment changes. To facilitate the laboratory and engineering design studies, an arrangement(16) was made with the Los Alamos Scientific Laboratory to ship to the LA recovery facilities all the slag-and-crucible material accumulated at Hanford Works prior to start-up of the Recuplex equipment. This agreement made it possible to base the process design upon steady-state conditions and eliminate the short-term operating period necessary to work-off the large back-log of waste material.

Process vessels were designed to have at least a 12-hour hold-up capacity and 20 per cent freeboard. To maintain continuity of operation and minimize the occurrence of "bottlenecks", some of the equipment pieces were duplicated and the hold-up capacity of others were extended to 24 hours. The processing equipment was designed to accommodate: (1) and (2), 330 liters/day of combined 2BP and F-10-P solution (the proportion of each being dependent upon the relative production rates of the Redox and Bismuth Phosphate Processes); (3), 10 liters/day of skulls-and-metal fabrication waste materials; (4), 10.7 crucibles and their associated slag per day, or 294 liters/day of S-&-C solution; and (5), 64 liters/day of prepared Wet-Chemistry supernatants.

B. Development of the Recuplex Process

The Recuplex feasibility investigation can best be considered according to the five major phases of the program: (1) dissolution of the slag-and-crucible material and the preparation of a feed stream suitable for introduction to the extraction column; (2) development of an efficient solvent-extraction process having suitable operating characteristics; (3) treatment of

[redacted]

of the three process waste streams; (4) corrosion studies; and (5) adequacy of the 234-5 Building facilities to accommodate the processing equipment.

(1) Slag-and-Crucible Dissolution and Solution Preparation

Of the five plutonium-bearing streams treated in the Recuplex Process only one required special development work for the preparation of a suitable feed solution: viz the crucibles and their associated slag from the Purification and Fabrication Building. The principal source of the material is the 234-Building Reduction step in which two combined plutonium-tetrafluoride batches are reduced to plutonium metal in a magnesium-oxide crucible, termed the S-1 crucible. A second source of crucibles is the 235-Building Pouring-and-Casting operation, in which two buttons are melted in an H-1 pouring crucible and transferred to an H-2 casting crucible. The H-1 and H-2 crucibles have the same composition as the S-1 crucibles, but are smaller and have a longer life. At present there are twice as many S-1 crucibles sent to recovery storage as H-2 crucibles and approximately twenty S-1 crucibles are discarded for each H-1 crucible. The compositions of typical S-1, H-1, and H-2 crucibles, and the slag associated with a reduction crucible are presented in Table I.

TABLE I

COMPOSITIONS OF TYPICAL REDUCTION, POURING, AND CASTING CRUCIBLES

Operation	Reduction			Pouring	Casting	Recovery
Type Crucible	S-1	Slag	Sand	H-1	H-2	Composite
Quantity per S-1 crucible	1	1	1	0.05	0.5	1
Components (grams/crucible)						
MgO	1519	---	106.5	426	270	1781.8
SiO_2	19.4	---	2.2	5.5	3.4	23.6
Fe_2O_3	3.9	---	---	1.1	0.7	4.4
CaO	8.1	---	---	2.3	1.4	8.9
R_2O_3	---	---	0.6	---	---	0.6
Ca	---	30.8	---	---	---	30.8
CaI_2	---	115.2	---	---	---	115.2
CaF_2	---	203.3	---	---	---	203.3
Pu	8.066			(a)	3.140(a)	9.636
Total weight	2017.07			434.9	278.64	2178.24

(a) A small portion of the Pu shown to be associated with the H-2 crucibles is actually associated with the H-1 crucibles.

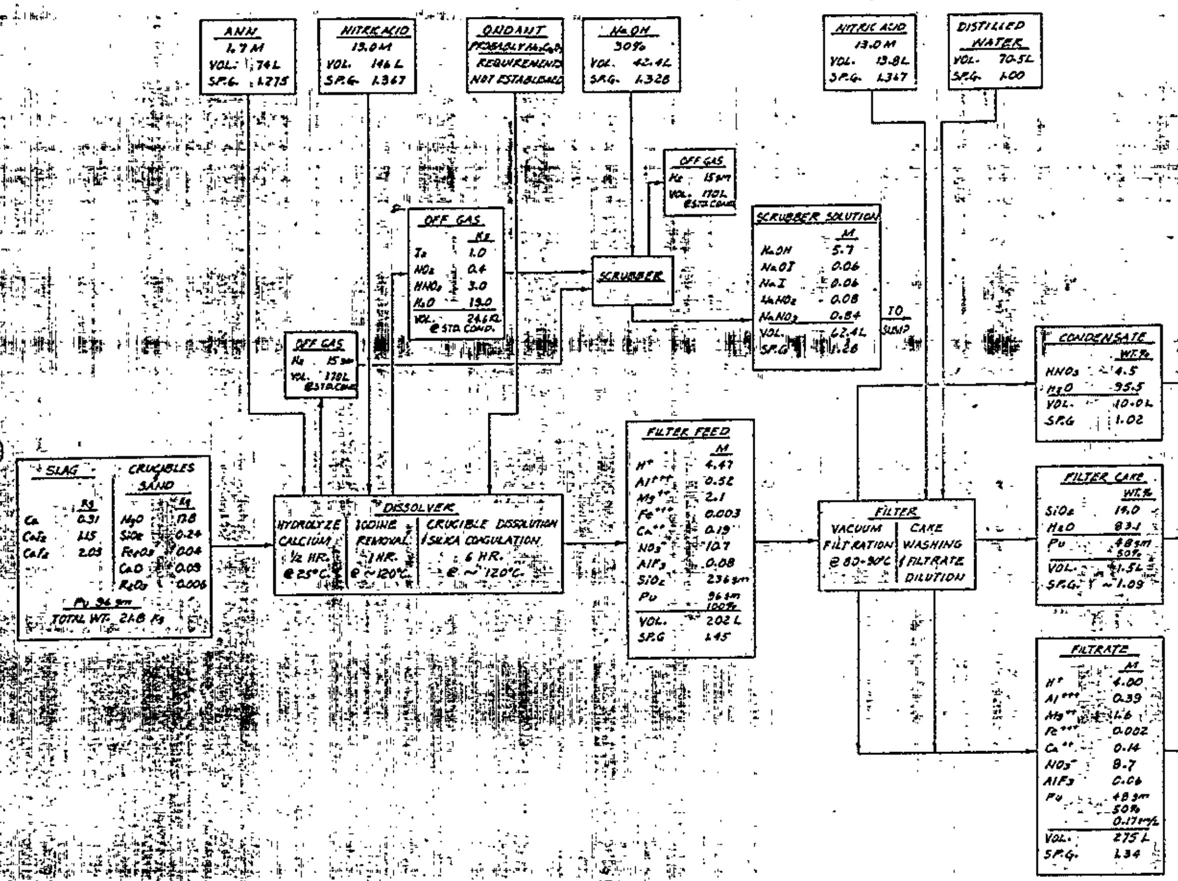
The design goal for the recovery of plutonium from the slag-and-crucibles has been established at 90 per cent of the plutonium present in the material. Accurate determination of the plutonium content of original material, however, necessitates a material balance of the streams entering and leaving the dissolution-and-filtration process. Since this is a time-consuming operation involving the processing of the entire silica residue, the evaluation of a dissolving procedure suitable for the Recuplex Process was facilitated by employing the Accountability values for the plutonium content of the material. In two of the experimental tests discussed below, however, complete plutonium balances were performed. The Accountability and analytically determined values agreed within 5 per cent in one case and within 11 per cent in the other. The amount of plutonium ascribed to an individual crucible and its associated slag by the Accountability Records is the difference between the weight of Pu button obtained in the 234-5 Reduction step and the weight of the plutonium in the PuF_4 batch prior to reduction (with periodic corrections for loss to pickling solution). It is recognized that the values can be in error in individual cases.

(a) Chemical flowsheets.

The chemical flowsheets of Figures H-2-15213 and H-2-15084 present two alternate procedures for the preparation of a slag-and-crucible feed solution, both of which include a dissolution step and a filtration step. The filtration step is necessitated by the silica content of the material, since the presence of SiO_2 in the final feed in concentrations greater than 10 mg/l seriously effects column operation, causing emulsification and poor disengaging times. The difference in the dissolving procedures of Figures H-2-15213 and H-2-15084 is that in the former the fluoride ion present in the slag is immediately complexed by aluminum nitrate-nonahydrate, while in the latter free HF is employed in the dissolution. By avoiding the immediate use of ANN to complex F^-, the plutonium loss to the silica cake is reduced to a minimum. Since a recovery yield greater than 90 per cent can be attained by the flowsheet in Figure H-2-15084 without re-working the silica residue, the method is recommended as being more suitable for the Recuplex Process. The procedure, however, presents a corrosion problem which has not as yet been fully investigated. There are presently three satisfactory approaches to the problem: 1) adjustment of the chemistry to provide satisfactory dissolution in a non-corrosive environment, 2) development of special stainless plastic lining (as teflon or fluorothene) for increased resistivity to chemical attack under the conditions of HW #1 Chemical Flowsheet, and 3) periodic replacement of the dissolver. Preliminary data indicate that the corrosion rates with presently available stainless steels (ca 0.0008 to 0.004 in/mo.) would make dissolver replacement economically feasible.

The chemical flowsheet presented in Figure H-2-15213 is based on the slag-and-crucible recovery process presently employed at the Los Alamos Scientific Laboratory. Data on recovery yields and optimum dissolving procedures were obtained from Los Alamos and from laboratory studies carried out by the Chemical Research, Chemical Development, and Process Assistance groups at Hanford. The Hanford-Works procedure (Figure H-2-15084) was developed by the Process Assistance (234-5 Development) group. The optimum conditions indicated by these studies are based on the dissolution of one reduction crucible and its associated slag. Development of the optimum operating

BASIS
TEN S-1 CRUCIBLES AND ASSOCIATED MATERIAL



PRELIMINARY
Not Approved for Construction

HW-22596

REVISIONS

SCALE NONE		APPROVALS		
DRAWN HJB	DATE 1-15-51	TJK	R.L.	1-12-51
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ENGINEER REQUEST NO. 234-5-4788				
PROJECT NO. [redacted]				

REFERENCE DRAWINGS

NEXT USED ON [redacted]

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

SLAG & CRUCIBLE
SOLUTION PREPARATION
PRELIMINARY
CHEMICAL FLOWSHEET

PROJECT TITLE RECUPLEX FEASIBILITY SURVEY
SEPARATIONS TECHNOLOGY-PROCESS

BLDG. NO. 234-5 DWG. NO. H-2-15213

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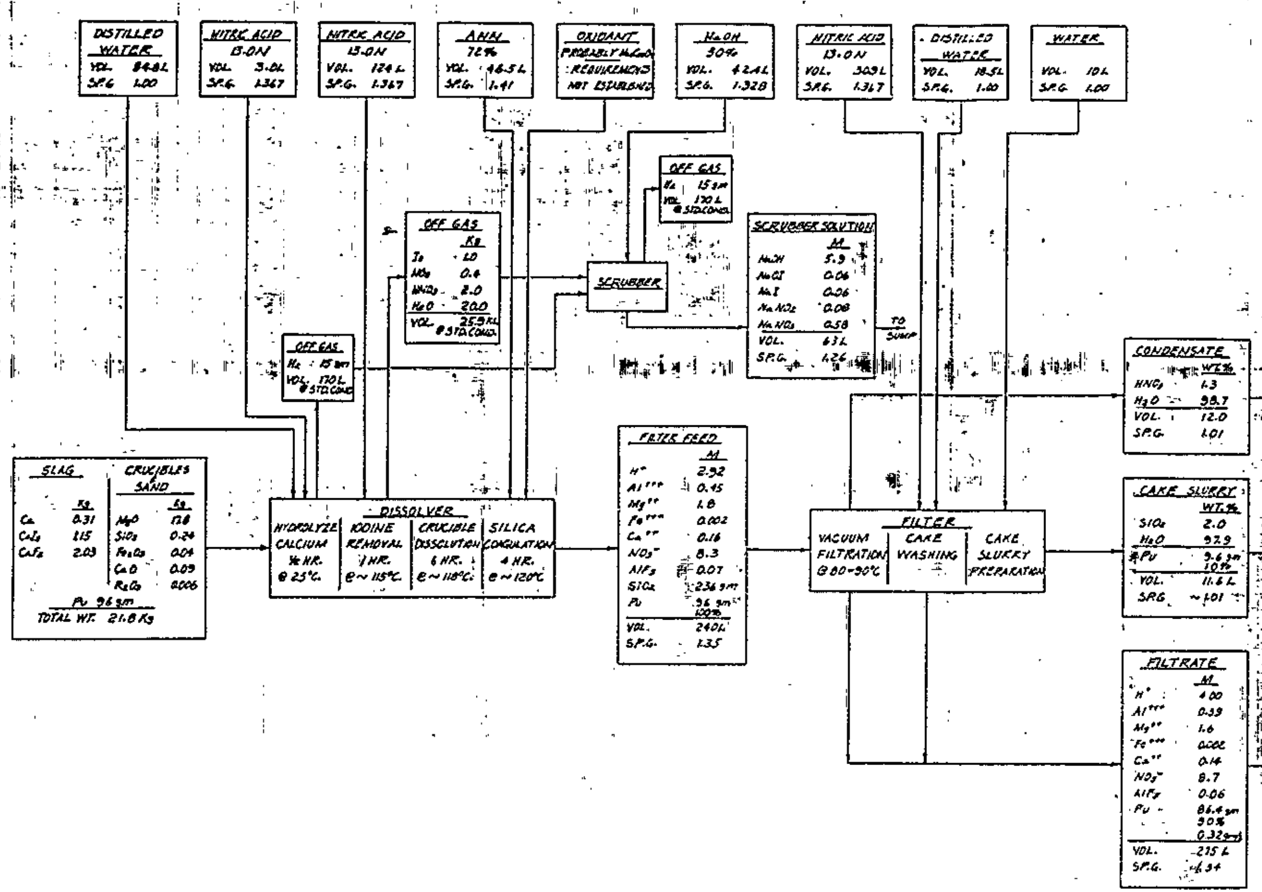
HW-22596



BASIS
TEN S-1 CRUCIBLES AND ASSOCIATED MATERIAL

HW-22596

NO.	DESCRIPTION	DRN. BY DATE	APP'D BY	DPT.	DATE
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SLAG & CRUCIBLE
SOLUTION PREPARATION

CHEMICAL FLOWSHEET HW#1

PROJECT TITLE RECUPLEX FEASIBILITY SURVEY
SEPARATION TECHNOLOGY - PROCESS

BLDG. NO. 234-5 DWG. NO. H-2-15084 SHEET NO. 1 SHEETS

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conditions for multi-crucible dissolvings can best be performed in the final process equipment since the construction of the necessary semi-works facilities would entail an installation essentially the same size as the final one with the associated problem of space location.

(i) Slag-and-crucible solution preparation: preliminary chemical flowsheet.

The Los Alamos procedure involves charging the solid materials to the dissolver kettle and adding 72 per cent aluminum nitrate-nonahydrate. The mixture is air-sparged at room temperature for one-half hour in order to hydrolyze the calcium present, complex the fluoride ion, and drive off the hydrogen formed. The iodine is then removed by boiling with 15 M nitric acid at 120°C. A six-hour period of refluxing at 120°C concludes the dissolution procedure, serving to dissolve the crucible and coagulate the silica. The resultant solution is diluted 25 per cent by volume with prepared oxalate supernatants and filtered at a temperature of 80 - 90°C to decrease the viscosity of the solution and prevent the formation of solid magnesium nitrate. The treated supernatant solution is employed as a diluent rather than distilled water in order to maintain the plutonium concentration at a maximum value.

Data on the laboratory dissolution of slag-and-crucible material, according to the procedure described above, were obtained from the Los Alamos Scientific Laboratory. In seven experimental runs the plutonium recovered in the filtrate and cake-wash solutions varied from 35 to 69 per cent of the Accountability value for the plutonium in the original material.(15) The dissolution procedure and the test data are summarized in Table II. To increase the slag-and-crucible yield, it was necessary to extend the Los Alamos facilities to include equipment for hydrofluorinating the silica cake and treating the dissolver residues with hydriodic acid. The space and equipment requirements necessarily involved in the expanded assembly made it highly desirable to develop a one-step, total dissolution procedure for incorporation into the proposed Recuplex Process.

The application of the Los-Alamos dissolution procedure to uranium and plutonium reduction crucibles and their associated slag was tested at the Hanford Works. The tests conducted with uranium as a stand-in for plutonium (which were primarily for the filtration studies discussed below) resulted in essentially complete uranium dissolution. When the procedure was applied to slag-and-crucible material containing plutonium, however, total plutonium dissolution was not obtained. In a series of seven dissolvings, each involving one small-scale reduction crucible and its associated slag, 10 - 40 per cent of the plutonium present in the material was recovered in the filtrate solution. The amount of plutonium originally present was determined from the Accountability Records. The data, obtained by the Process Assistance (234-5 Development) group, are presented in Table III. Similarly, dissolution of five plant-scale crucibles and their associated slag in the Chemical Research laboratories resulted in 58 per cent recovery of the plutonium indicated by the Accountability Records to be present in the material. The undissolved heel remaining in the kettle after three crucible dissolutions contained 11 per cent of the accountable plutonium and required treatment with hydriodic acid for its removal. Since dissolution of the slag-and-crucible material by the Los Alamos procedure would not permit 90 per cent recovery of the plutonium without further treatment of the silica cake,

TABLE II

SLAG-AND-CRUCIBLE DISSOLUTION LABORATORY TESTS PERFORMED AT LOS ALAMOS SCIENTIFIC LABORATORY*

Basis: 1 reduction crucible and associated slag.

Procedure: 3.095 liters of .81M $Al(NO_3)_3 \cdot 9\ H_2O$ solution added to slag, crucible and sand in 12 liter reaction vessel. Solution was air sparged for 30 minutes to allow the H_2 gas to be evolved safely. Then, 4.8 liters of 15.7M HNO_3 was added slowly. The I_2 evolved during this reaction was absorbed in a KOH scrubber. After the I_2 liberation reaction had subsided, HNO_3 sufficient to render the slag-and-crucible solution 5M in free acid was added. The solution was refluxed for 6 hours at 100°C. The solution was then cooled to 80°C and filtered through a 500 ml sintered glass funnel. The silica cake was washed with 6N HNO_3. The slag-and-crucible solution was diluted to 9.0 liters with 6N HNO_3.

Run Number	Total Pu[a] in S-&-C (grams)	Pu Recovered in Filtrate and Cake Washes (%)
A-669[b]	6.31	35.3
A-670[b]	13.2	39.7
A-X-7[b]	4.77	53.5
A-X-8[b]	3.75	68.5
KR-1130-L-A[c]	2.08	68.7
XT8L-A+B[d]	18.82	41.7
XT9L-A[c]	7.6	62.3

Notes:
* Data obtained at the Los Alamos Scientific Laboratory.(15)
a. Based upon Accountability Records.
b. Laboratory reduction crucible and associated slag.
c. One-half plant reduction crucible and associated slag.
d. Total plant reduction crucible and associated slag.

TABLE III

SLAG-AND-CRUCIBLE SOLUTION PREPARATION PRELIMINARY LABORATORY TESTS*

Basis: 1 small-scale crucible and associated slag.

Procedure: Material charged to dissolver.
350 ml 1 M ANN added.
325 ml conc. HNO_3 added.
Solution air-sparged at 25°C for 15 minutes.
Mixture heated without reflux for 45 - 60 minutes.
Mixture heated with reflux during indicated reflux period.
Solution filtered at 80 - 90°C.

Run Number	Reflux Period (hours)	Total Pu[a] in S-&-C (gm)	Pu Recovered in filtrate (%)
1	6	0.436	12.8
2	6	0.252	39.6
3	5	0.638	15.2
4	5	1.150	9.5
5	4.5	1.06	29.2
6	8	0.806	34.8
85	6	0.564	36.4

Notes: * Data obtained by the Process Assistance (234-5 Development) group.
a. Based on Accountability Records.

various modifications were tested on a laboratory scale.

(ii) Slag-and-crucible solution preparation: HW chemical flowsheet No. 1.

Three modified procedures were tested by the Process Assistance (234-5 Development) group with small-scale reduction crucibles. The first involved the use of nitric acid without any fluoride-ion complexing agent and resulted in 93.9 per cent recovery of the accountable plutonium in the material. In the second modified procedure, nitric acid was added in incremental amounts and the mixture heated until visual observation indicated that all the iodine had been driven off (10-15 minutes). The solution was then refluxed for approximately four hours to digest the crucible and associated plutonium. The fluoride was subsequently complexed with ANN and an additional four-hour refluxing period followed, serving primarily to coagulate the silica into an easily filterable form. In four test runs, 87 to 94 per cent of the accountable plutonium was recovered in the filtrate. A series of samples was taken throughout the test indicated that plutonium dissolution was essentially completed within the first hour of the crucible-digestion period.

TABLE IV

SLAG-AND-CRUCIBLE SOLUTION PREPARATION
HW LABORATORY TESTS - PROCEDURE I*

Basis: 1 small-scale crucible and associated slag.

Procedure:
Material charged to dissolver.
750 ml of 6N HNO_3 added.
Mixture heated without refluxing for one hour.
Mixture heated with refluxing for 5 hours.
Solution filtered at 20°C.

Run Number	Reflux Period (hours)	Total Pu[a] in S-&-C (grams)	Pu Recovered in filtrate (%)
86	5	1.165	93.9

Notes:
* Data obtained by the Process Assistance (234-5 Development) group.
[a] Based on Accountability Records.

TABLE V

SLAG-AND-CRUCIBLE SOLUTION PREPARATION
HW LABORATORY TESTS - PROCEDURE II*

Basis: 1 small-scale crucible and associated slag.

Procedure:
Material charged to dissolver.
Approximately 50 per cent of indicated HNO_3 added.
Mixture heated without reflux approximately 10 - 15 minutes until all I_2 driven off (determined by visual observation).
Remainder of HNO_3 added.
Mixture refluxed for four hours.
Indicated quantity of 2.3M ANN added.
Mixture refluxed for an additional four hours.
Solution filtered at 80 - 90°C.

Run Number	Total HNO_3 added	ANN added (ml)	Total Pu in S-&-C (grams)	Pu Recovered in Filtrate: Before ANN added. (%)	Pu Recovered in Filtrate: At End Of Run (%)
138	500 ml-8M	123	0.650	94	91.4
143	500 ml-8M	123	0.955	88.2[d]	84.6
144[b]	400 ml-10M	100	0.256	93.4	80.1
145[b]	400 ml-10M	100	0.548	86.8	84.6

Notes:
* Data obtained by the Process Assistance (234-5 Development) group.
a. Based on Accountability Reports.
b. Crystallization occurred on cooling solution due to high salt concentration.
c. Of the total Pu charged to slag-and-crucible #138 0.8 per cent was found in the silica cake.
d. Of the total Pu charged to slag-and-crucible #143 0.8 per cent was found in the silica cake.

TABLE VI

SLAG-AND-CRUCIBLE SOLUTION PREPARATION
HW LABORATORY TESTS - PROCEDURE III*

Basis: 1 small-scale crucible and associated slag.

Procedure: Material charged to dissolver.
Approximately 250 ml 8M HNO_3 added.
7.73 ml 2.3 M ANN added (equivalent to the fluoride ion present in slag).
Mixture heated without reflux approximately 10 - 15 minutes until all I_2 driven off (determined by visual observation).
Remainder of 8M HNO_3 added (500 ml total).
Mixture refluxed for five hours.
115 ml 2.3 M ANN added.
Mixture refluxed for an additional four hours.
Solution filtered at 80 - 90°C.

Run Number	Total Pu[a] in S-&-C (grams)	Pu Recovered in Filtrate: Before final addition of ANN (%)	Pu Recovered in Filtrate: At End of Run (%)
140	0.437	95	93.7

Notes: * Data obtained by the Process Assistance (234-5 Development) group.
a. Based on Accountability Records.

In the third experimental procedure, an amount of ANN equivalent to the calculated F⁻ content of the material was added to the dissolver with a portion of the nitric acid. After the evolution of the iodine, the remaining nitric acid was added and the mixture refluxed for five hours. An excess of ANN was then added and the silica coagulated by a four-hour refluxing period. The recovery yield was 95 per cent. The data, presented in Tables IV, V, and VI clearly indicate that the presence of uncomplexed fluroride ion in the acid solution is necessary for the complete dissolution of plutonium.

(b) Filtration procedures.

Data were obtained by the Chemical Development group on the optimum conditions for filtration of dissolved slag-and-crucible solution, employing uranium as a stand-in for plutonium.(18) Tests were conducted on a laboratory-scale with a 3-inch diameter (0.05 sq. ft.) sintered stainless-steel filter plate and on a semi-works scale with a sintered stainless-steel star filter having approximately 1.7 sq. ft. initial effective area. Precoats of thicknesses varying from 1/8" to 3/4" were placed on the plates by filtering a water slurry of either Celite 521 or Hy-Flo Super Cel filter aid. In each case the precoat was washed with water. Filter aid was added to the dissolver solution approximately ten minutes before the start of filtration. The temperature of the filter feed was maintained in the range of 80 to 95°C and the filtration was accomplished by applying 16 inches of mercury vacuum to the downstream side of the plate. The data, presented in Table VII, indicate that with a precoat thickness of 1/8 inch and 2.5 grams/liter of Hy-Flo Super Cel filter aid in the dissolver solution, the overall filtration rates vary from 10 to 40 (gal)/(hr)(sq. ft.). The lower rates were obtained under sub-standard digestion conditions in the dissolver, that is, a digestion and silica coagulation temperature of 100°C rather than 120°C. Employing filter aid in the above manner reduces the SiO_2 content of the filtrate to 0.01 g/l, which does not appreciably effect disengaging time and is acceptable for satisfactory column operation.

Slag-and-crucible solutions were filtered in the laboratory through National Filter Media glass cloth G-201 and Fiberglas 183 without employing filter aid. Data from these tests are included in Table VII. Filtration rates during the initial filtration cycle are generally higher than the rates obtained with filter aid on sintered stainless steel. As the cake depth on the glass cloth increases, however, the filtration rate decreases rapidly, resulting in overall filtration rates of approximately 10 (gal)/(hr)(sq. ft.). The reduction of the silica content of the dissolver solution obtained by the use of glass-cloth filters was sufficient to permit satisfactory disengaging times.

The results of the experimental studies demonstrate that both of the filtering techniques described above provide adequate filtration rates and silica removal efficiencies under Recuplex operating conditions.

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TABLE VII

SLAG-AND-CRUCIBLE SOLUTION PREPARATION

FILTRATION STUDIES*

Dissolution: Basis: One crucible and associated slag. (U as stand-in for Pu), dissolved according to the Los Alamos procedure.

Filter Equipment:

Lab. Steel Plate Horizontal, 3-inch diameter (0.05 sq.ft.), filter plate of Micrometallic, porosity "D", sintered stainless steel was clamped between flanges in 3-inch i.p.s. pipe, filter body having open, upper body and bottom outlet to vacuum, filter flask. Feed solution entered the upper, filter body through a 1/4-inch, glass tube emptying directly onto the filter plate.

Lab. Glass Cloth Filter plate of above equipment was replaced by glass cloth supported on stainless steel, wire mesh, 4 mesh per inch. Gaskets restricted filter area to 0.0412 sq.ft. Glass cloth was either National Filter Media Co. 0-201 or Fiberglas 183 as noted. No precoat or filter aid used for glass-cloth filtrations.

321 Building Semi-works scale equipment employed a standard, Micrometallic, porosity "F", sintered stainless steel, star filter having approx. 1.7 sq.ft. initial effective area. Filter element was enclosed in filter case which was filled with feed solution as filtrate was drawn by vacuum from the dissolver tank, through the filter, and into the filtrate receiver.

Filter Procedure Precoat of desired thickness was laid on filter by filtering a water-slurry of the filter aid. Precoat was washed with water until wash came through clear. Filter aid was added to dissolver solution approx. 10 minutes before start of filtration. Feed rate to filter adjusted to keep filter covered with liquid. Filtration accomplished by 16 inches of mercury vacuum. Temperature of filter feed 95° to 80° C.

Disengaging Time Disengaging time determined by agitating selected volumes of filtrate and CAX (15% TBP in CCl_4) for one minute in a 50-ml., graduated cylinder and measuring the time for clean separation of the liquid phases after agitation has stopped. Pretreatment of filtrate and volume ratios are noted below.

Filtrate Clarity Per cent transmission of light of 657-millimicron wavelength was determined for all filtrates from the lab runs. The results are not listed because "clarity", beyond the point of being visibly clear, is not considered significant. All filtrations listed produced filtrates that were sparkling clear.

Run No.	Filter Medium, Precoat or type of glass cloth	Filter Aid Conc., g./l.	Total Time, min.	Total(1) Filtrate, Gal./sq.ft.	Overall Rate, Gal./(hr.)(sq.ft.)(1)		Instantaneous Rate After 10 gal./sq.ft., Gal./(hr.)(sq.ft.)	Disengaging(2) Time, seconds	SiO_2 in(2) Filtrate, g./l.
					For 7 gal./sq.ft.	For 12 gal./sq.ft.			
Lab. I	1/8 inch of Celite 521	2.56	76	12	—	—	5.7	69(6)	0.012
Lab. II	3/4 inch of Celite 521	2.56	258	30	—	—	9.5	253(6)	0.02
Lab. III	1/4 inch of Hy-Flo Super Cel	5.0	101	24	—	—	18	55(6)	0.02
Lab. IV	1/4 inch of Hy-Flo Super Cel	10.0	105	18	—	—	9.5	149(6)	0.016
Lab. V	1/4 inch of Hy-Flo Super Cel	0	83	18	—	—	11.5	98(6)	0.03
Lab. VI	1/4 inch of Hy-Flo Super Cel	2.5	86	24	52	36	17	300(6)	0.05
Lab. I'	National 0-201	0	72	15(4)	39	9	11	37(7)	—(10)
Lab. II'	National 0-201	0	70	16(4)	38	11	14	33(7)	—(10)
Lab. IV'	Fiberglas 183	0	24	7(4)	18		(10)	35(7)	—(10)
321-I(3)	1/8 inch of Hy-Flo Super Cel	2.5	40	12	35	18	13	35(8)	0.01
321-II(3)	1/8 inch of Hy-Flo Super Cel	2.5	76	12	18	10	7	33(8)	—
321-III	1/8 inch of Hy-Flo Super Cel	2.5	30	17	53	40	34	86(9)	0.01

*Data obtained by Chemical Development, Separations Technology Unit(18).

Notes:

(1) Due to geometry of filter used in 321 Building, the filter area decreased from 1.7 sq.ft. to approx. 1.2 sq.ft. as the run progressed. The decrease has been considered.

(2) If more than one sample was taken during the run, only the maximum value is shown.

(3) During runs 321-I and 321-II, the silica-coagulation step was performed at 100°C. for 16 to 20 hours.

(4) None of the filtrate was recycled.

(5) Estimated rate after 7 gal./sq.ft. filtrate volume.

(6) 1:1 volume ratio aqueous to organic; aqueous was undiluted filtrate, Sp.Gr. 1.40.

(7) 1:1 volume ratio aqueous to organic; aqueous was filtrate after 50% volume dilution.

(8) 1:1 volume ratio aqueous to organic; aqueous was filtrate diluted to Sp.Gr. 1.32.

(9) 1:1 volume ratio aqueous to organic; aqueous was filtrate at Sp.Gr. 1.35.

(10) Analysis not yet known.

[redacted]

(2) Solvent Extraction

(a) General principles.

The use of liquid-liquid extraction as a means of separating small concentrations of a desired component from a solution has grown rapidly in the last few years, especially within the field of nucleonic engineering. Separation of the desired material is accomplished by its transfer from one liquid phase to a second, immiscible, liquid phase according to its respective solubility in the two liquids. Thus, plutonium in the aqueous stream, containing various associated impurities, may be purified and concentrated by contacting it with an organic solvent in which the plutonium is preferentially soluble. The plutonium may subsequently be further concentrated and returned to an aqueous solution by a similar extraction process in which the solubility distribution favors the aqueous phase. Through the efficient performance of these two operations, the Recuplex Process provides the necessary purification and concentration for the five-component feed stream and yields a product solution suitable for Wet Chemistry processing.

The solvent-extraction steps in the Recuplex Process take place in columns having a suitable number of stages and operating continuously and countercurrently in a manner similar to a fractionating column. In fact, a direct analogy may be drawn between solvent extraction and fractional distillation. Thus, the volatility term found in distillation problems has a direct counterpart in solvent extraction in the form of the $E{:}\frac{o}{a}$ value which is defined as the ratio of the solubility of one component in the organic phase to its solubility in the aqueous phase under conditions of phase equilibrium. By employing $E\,\frac{o}{a}$ values and calculating the concentrations in the two phases, an equilibrium curve may be constructed which has the same shape as the normal Y *vs.* X equilibrium curve. Similarly, the concept of a theoretrical plate and the use of HETS or HTU values have their counterparts in extraction and distillation design calculations.

Operation of an extraction unit involves the introduction of the two phases at opposite ends of the tower, intimate mixing during their countercurrent passage through the column, and their final separation and discharge. The aqueous feed stream may be introduced at the bottom and the organic extractant at the top, or *vice versa*, depending upon the relative densities of the two liquids. Either phase may be the continuous medium. The total flow rates are limited by the flooding capacity of the column; *id est*, the ability of the disengaging section to permit the formation of an interface and prevent one phase from being entrained in the discharge stream of the other. Phase separation in the disengaging section is influenced by the density difference and immiscibility of the two liquids, and the presence of emulsifying contaminants. A disengaging time of two minutes is normally considered the maximum period for satisfactory column operation.

(b) Selection of the organic diluent.

Early experimental studies of the Recuplex extraction operations were performed with tributyl phosphate as the selective solvent and the hydrocarbon AMSCO 125-90W as the organic diluent. It was believed, however, that the use of an alternate, non-flammable diluent would be preferable in the 234-5 Building operations. A series of tests (cf Table VIII)(10) was conducted with slag-and-crucible solution to determine a suitable TBP-diluent mixture that would disengage within the acceptable time limit. The density difference between the two phases could be increased by increasing the molecular weight of the diluent, varying the TBP content of the organic phase, or by diluting the aqueous phase. Adequate disengaging times and satisfactory column operation were obtained by diluting the slag-and-crucible solution 25 per cent (by volume) and employing as a solvent a mixture of 15 per cent (by volume) TBP in carbon tetrachloride.

TABLE VIII

PROPERTIES OF DILUENT MIXTURES*

Diluent		Solvent		Disengaging Time[a]
Composition (% by weight)	Density (gm/ml)	TBP (% by volume)	Density (gm/ml)	(minutes)
100% C_2HCl_5	1.503	30	1.449	2
100% C_2HCl_5	1.503	15	1.552	1
13.1% C_2Cl_6 86.9% CCl_4	1.591	30	1.406	>4
44.6% C_2Cl_6 55.4% CCl_4	1.629	30	1.433	>3
44.6% C_2Cl_6 55.4% CCl_4	1.629	15	1.528	1
100% CCl_4	1.595	15	1.492	ca 2

Notes: * Data obtained by the Chemical Research group.(10)
[a] Tests were conducted with undiluted slag-and-crucible solution having a density of approximately 1.40 gm/ml.

(c) Chemical flowsheets.

Schematic representations of two feasible methods of performing the solvent-extraction steps in the Recuplex Process are presented in Figures H-2-15206 and H-2-15207.(9) The chemical flowsheet of Figure H-2-15206 presents an adequate and satisfactory processing scheme. The latter flowsheet, however, is considered the more advantageous on the basis of its greater flexibility in handling possible variances in stream composition. The two flowsheets are based on laboratory and pilot-plant investigations of approximately six preliminary flowsheets. The data necessary for their preparation were obtained through the cooperative efforts of the Chemical Research and Chemican Development groups.

(i) Chemical flowsheet No. 1.

The chemical flowsheet presented in Figure H-2-15206 contains two solvent-treatment towers, the extraction or CA column and the stripping or CC column, S[redacted]

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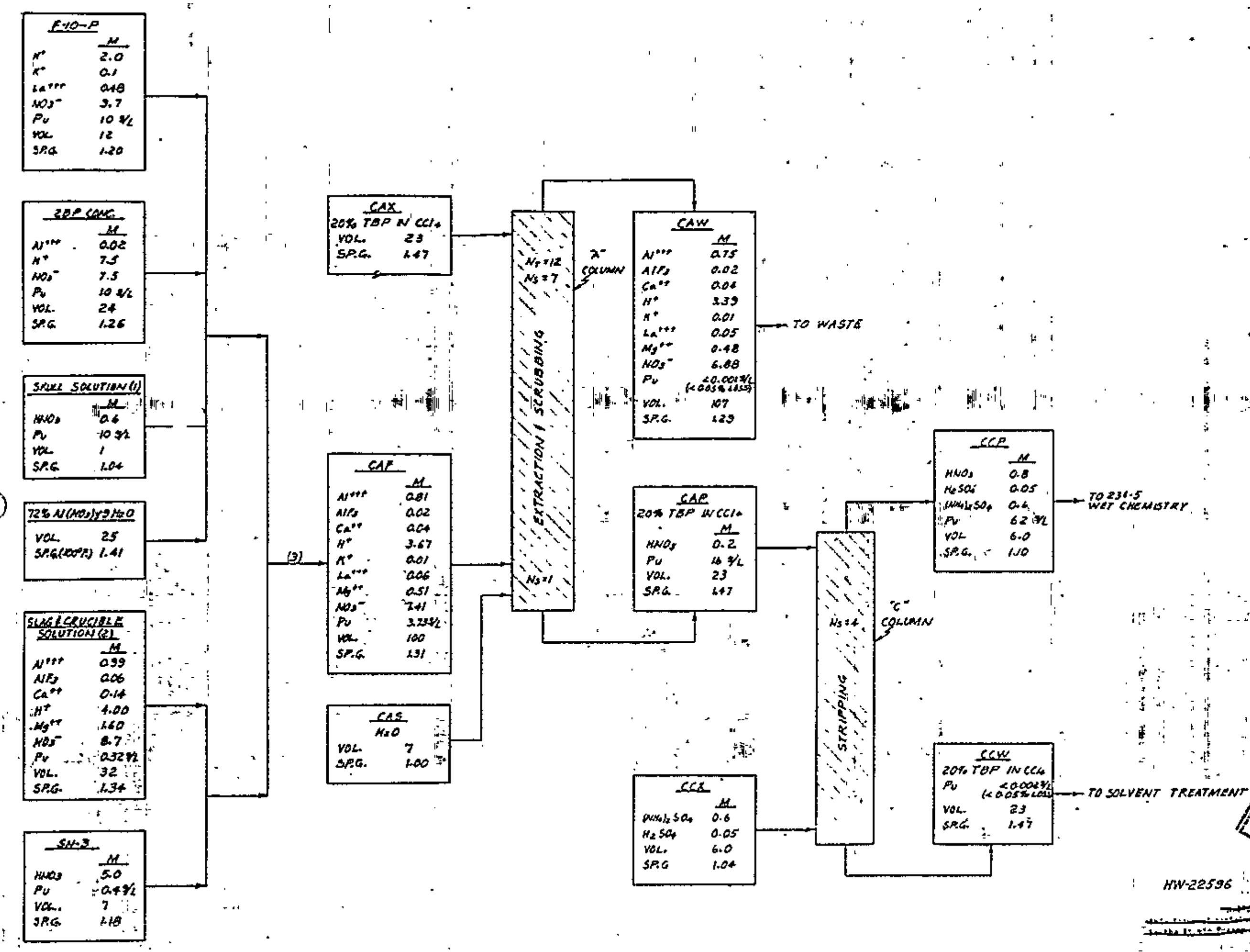

BASIS

100 VOLUMES OF CAF.

NOTES

1. THE SKULL SOLUTION HAS NOT BEEN INCLUDED IN THE CAF STREAM. THE SOLUTION COMPOSITION IS VARIABLE DUE TO THE DIFFERING PROPORTIONS OF METAL WASTE STREAMS THAT ORIGINATE AFTER THE 234-5 CASTING OPERATION. THE SKULL SOLUTION VOLUME IS SMALL AND WILL NOT AFFECT COLUMN OPERATION.
2. THE SLAG & CRUCIBLE SOLUTION COMPOSITION AS GIVEN IS ONLY APPROXIMATE AND WILL DEPEND UPON FINAL DISSOLUTION PROCEDURE.
3. THE RATIO OF VARIOUS FEEDS IS BASED ON POSTULATED OPERATING RATES, AND WILL VARY ACCORDING TO PLANT REQUIREMENTS.
4. NUMBERS INSIDE COLUMNS REFER TO APPROXIMATE NUMBER OF TRANSFER UNITS OR STAGES REQUIRED FOR SPECIFIED CONDITIONS.

N_T = NO. OF THEORETICAL TRANSFER UNITS.*
N_S = NO. OF THEORETICAL STAGES.
*OVERALL AQUEOUS FILM BASIS FOR THE "A" COLUMN EXTRACTION SECTION.

PRELIMINARY
Not Approved for Construction

HW-22596

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NO.	DESCRIPTION	DRN. BY DATE	APP'D BY	DIV.	DATE
REVISIONS				

SCALE: NONE	APPROVALS		
	BY	DIV.	DATE
DRAWN WZB DATE 1-14-51	CZK	Ach	1-20-51
CHECKED RZA DATE 1-14-51			
ISSUED DATE			
SEC. ENG. DIV.			
ENGINEER REQUEST NO. 234-5-4788			
PROJECT NO. [redacted]			

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

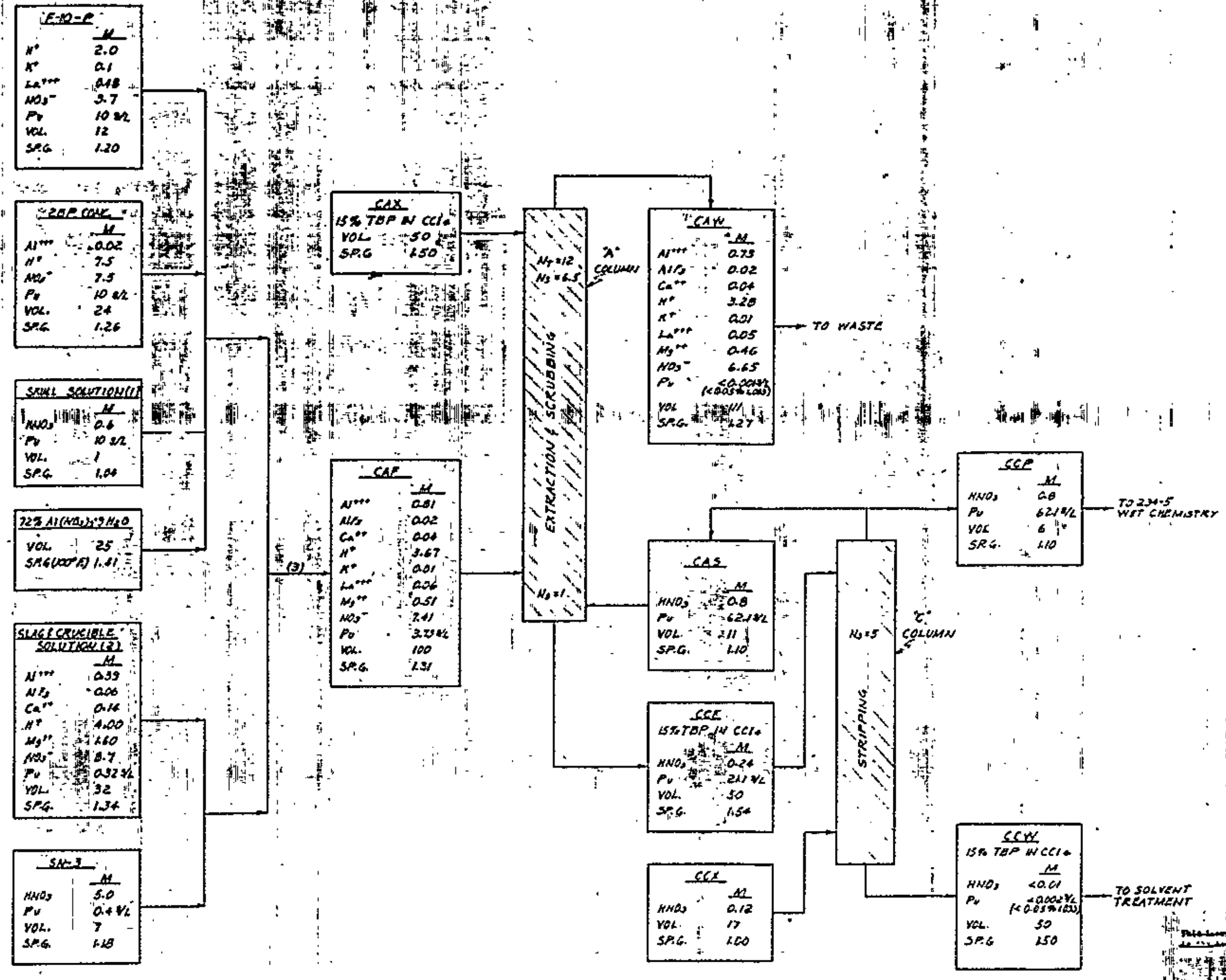
SOLVENT EXTRACTION
CHEMICAL FLOWSHEET HW#1

PROJECT TITLE RECUPLEX FEASIBILITY SURVEY
SEPARATIONS TECHNOLOGY-PROCESS

BLDG. NO. 234-5 DWG. NO. H-2-15206 SHEET NO. 1

DWG. NO.	DRAWING TITLE
HW-22604	RECUPLEX PROCESS CHEM. FLOWSHEET HW#1
REFERENCE DRAWINGS	
NEXT USED ON	

[redacted]



HW-22596

BASIS

100 VOLUMES OF CAF.

NOTES

1. THE SKULL SOLUTION HAS NOT BEEN INCLUDED IN THE CAF STREAM. THE SOLUTION COMPOSITION IS VARIABLE DUE TO THE DIFFERING PROPORTIONS OF METAL WASTE STREAM THAT ORIGINATE AFTER THE 234-5 CASTING OPERATION. THE SKULL SOLUTION VOLUME IS SMALL AND WILL NOT AFFECT COLUMN OPERATION.
2. THE SLAG & CRUCIBLE SOLUTION COMPOSITION AS GIVEN IS ONLY APPROXIMATE AND WILL DEPEND UPON FINAL DISSOLUTION PROCEDURE.
3. THE RATIO OF VARIOUS FEEDS IS BASED ON POSTULATED OPERATING RATES, AND WILL VARY ACCORDING TO PLANT REQUIREMENTS.
4. NUMBERS INSIDE COLUMNS REFER TO APPROXIMATE NUMBER OF TRANSFER UNITS OR STAGES REQUIRED FOR SPECIFIED CONDITIONS.
   - N_T = NO. OF THEORETICAL TRANSFER UNITS.*
   - N_S = NO. OF THEORETICAL STAGES.
   - *OVERALL AQUEOUS FILM BASIS FOR THE "A" COLUMN EXTRACTION SECTION.

NO.	DESCRIPTION	APP'D BY DATE	APP'D BY	DIV.	DATE
	REVISIONS				

SCALE: NONE
DRAWN HZB DATE 11-19-51
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PROJECT NO. [redacted]

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U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

SOLVENT EXTRACTION
CHEMICAL FLOWSHEET HW#

PROJECT TITLE RECUPLEX FEASIBILITY SURVEY
SEPARATIONS TECHNOLOGY - PROCESS

BLDG. NO. 234-5 DWG. NO. H-2-15207 SHEET NO.

DWG. NO.	DRAWING TITLE
HW-22604	RECUPLEX PROCESS CHEM. FLOWSHEET HW#2

REFERENCE DRAWINGS

NEXT USED ON

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by means of which a plutonium solution can be effectively prepared for treatment in the Wet-Chemistry step in the 234-5 Building. The purpose of the CA column is to effect the transfer of plutonium from the combined aqueous feed stream to the organic phase with minimum loss of plutonium to the aqueous waste and minimum entrainment of the original contaminants to the organic product stream. To accomplish this efficiently, the CA column contains an extraction section and a scrubbing section, the former serving to transfer the plutonium to the organic phase and the latter serving to scrub entrained contaminants from the organic stream prior to its passage to the stripping column. Since the solvent, or CAX stream, is the heavier solution, the feed or CAF stream enters the column above the scrubbing section and rises as the continuous medium to the aqueous-waste discharge port of the top of the column. Distilled water is introduced at the bottom to serve as the scrub or CAS stream. The plutonium-bearing organic stream leaves the bottom of the scrubbing section as the product of the CA column and is pumped to the top of the stripping column.

The state equilibrium data for the CA column, which are presented in Table IX, are representative of the series of laboratory tests upon which the chemical flowsheet of Figure H-2-15206 is based. The data were obtained by countercurrent, batch equilibration and separation of the two phases in a series of separatory funnels, each of which represented an extraction stage. Thus, analysis of the phases at each stage led to the distribution of plutonium and nitric acid throughout the theoretical "column". The run (number 18, conducted by the Chemical Research group) differed slightly from the procedure presented in Figure H-2-15206. The plutonium solutions were not introduced into the "column" as a combined feed. but were divided into two streams, the CAFa and the CAFb feeds. The former contained plutonium at a concentration of 19.1 g/l, (approximately twice the flowsheet value) and was introduced into the "column" immediately above the scrubbing section. The CAFb stream, diluted 1:1 with distilled water, entered the "column" two stages above the CAFa entry point. The feed streams did not contain a salting agent. The extraction section comprised six stages and the scrubbing section two stages. The plutonium distribution between the two phases throughout the "column" is represented by the equilibrium curve in Figure 1.

The data indicate that the performance of the column was good; the measured plutonium loss to the aqueous waste stream was 0.11 per cent. Factors influencing the extraction efficiency were the valence state of the plutonium ion and the absence of the salting agent, aluminum nitrate nonahydrate. In systems involving TBP as a selective solvent, the extraction coefficient is slightly greater for Pu IV than for Pu VI and has been demonstrated to be dependent upon the concentration of ANN present in the feed.(11) The feed stream employed in the experimental run, however, had approximately 25 per cent of the plutonium in the plus VI valence state and did not contain a salting agent. It is estimated that the loss of the CAW would have been approximately 0.025 per cent if ANN had been added to the CAF stream. Compensation for these factors has been made in the preparation of the chemical flowsheet. Spectrographic analysis of the scrub solutions obtained in an earlier test indicated that scrubbing with water removes considerable quantities of calcium, magnesium, aluminum, and manganese from the organic

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Figure No. I

PLUTONIUM EQUILIBRIUM CURVE

Run No. 18

Countercurrent Batch Equilibration and Separation

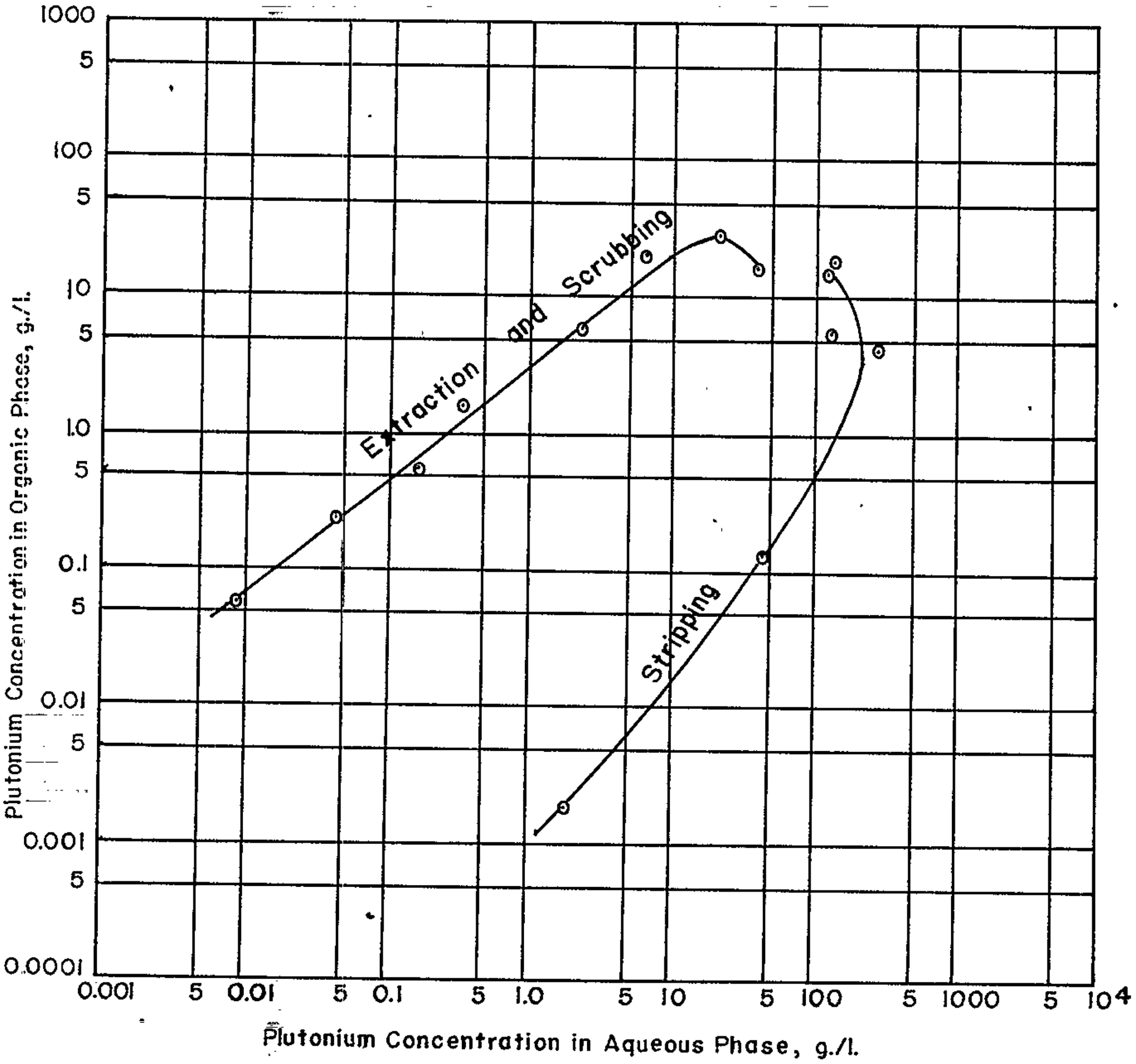

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TABLE IX

CA COLUMN STAGE EQUILIBRIUM DATA*

Run Number 18
Countercurrent batch equilibration and separation.

Run Characteristics:

Extraction - 6 stages
Scrubbing - 2 stages
No ANN employed in CAF.
S-&-C diluted 100%

Stream	Volumes	Composition
CAS	6	H_2O
CAFa[a]	30	19.1 g/1 Pu, 4.5 M HNO_3
CAFb	40	0.077 g/1 Pu
CAX	46.5	15% TBP in CCl_4
CAW	76	0.0081 g/1 Pu
CAP	46.5	12.6 g/1 Pu, 5.4 g/1 HNO_3

Pu loss to aqueous waste - 0.11%

Stream Operation		Stage No.	Organic Phase		Aqueous Phase		Extraction Coefficients	
In	Out		Pu g/1	HNO_3 g/1	Pu g/1	HNO_3 g/1	EQ Pu	EQ HNO_3
CAX	CAW	1	0.061	-----	0.0081	-----	7.5	-----
		2	0.205	-----	0.045	-----	4.5	-----
		3	0.546	27.2	0.133	214	4.1	0.13
CAFb		4	1.60	27.2	0.34	214	4.7	0.13
		5	5.95	27.2	2.15	243	2.8	0.11
CAFa		6	15.6	21.9	5.65	242	2.8	0.09
		7	18.8	12.0	17.5	125	1.1	0.10
CAS	CAP	8	12.6	5.4	43.0	50	0.3	0.11

Note: * Data obtained by the Chemical Research group.
[a] The plutonium content of the CAFa stream was twice the flowsheet concentration.

phase.(12) Following the scrubbing operation, the plutonium stream contains only a few tenths of one per cent total impurities (based on the plutonium present) and is adequately purified by an oxalate or peroxide precipitation step in 234-5 Wet Chemistry.

The purpose of the CC column is to transfer or strip the isolated plutonium from the organic phase to the aqueous solution and effect simultaneously a great enough volume reduction to permit direct shipment of the solution

[redacted]

[redacted]

to Wet Chemistry. The organic product stream from the CA column is pumped to the top of the CC column and is contacted with an aqueous stripping agent rising from the bottom. To be effective, the stripping solution must contain either a reducing agent or a plutonium-complexing agent. Rapid reduction of the Pu IV and Pu III by a compound such as hydroxylamine sulfate should result in an efficient stripping operation since the extraction coefficient for Pu III is lower than for Pu IV. It has been demonstrated, however, that the sulfate ion complexes the plutonium and can strip the organic phase satisfactorily.

Stage equilibrium data and the plutonium equilibrium curve for an experimental CC column similar to that in Figure H-2-15206 are presented in Table X and Figure 1 respectively. These data were obtained in the same manner and during the same test run as those described above. The stripping operation was efficient, with a plutonium loss to the organic waste stream of only 0.012 per cent.

TABLE X

CC COLUMN STAGE EQUILIBRIUM DATA*

Run Number 18
Countercurrent batch equilibration and seperation.

Run Characteristics:

Stripping - 6 stages

Stream	Volume	Composition
CCF[a]	46.5	12.6 g/l Pu, 5.4 g/l HNO_3
CCX	5	0.6 M $(NH_4)_2SO_4$, 0.05 M H_2SO_4
CCW	46.5	0.0014 g/l Pu, < 0.16 g/l HNO_3
CCP	5	108 g/l Pu, 36 g/l HNO_3

Pu loss to organic waste - 0.012%

Stream Operation		Stage No.	Organic Phase		Aqueous Phase		Extraction Coefficients	
In	Out		Pu g/l	HNO_3 g/l	Pu g/l	HNO_3 g/l	E^o_a Pu	E^o_a HNO_3
CCF	CCP	9	12.9	2.45	108	36	0.12	0.068
		10	14.8	1.57	114	27.1	0.13	0.058
		11	4.81	0.98	309	17.1	0.016	0.057
		12	5.13	<0.65	109	11.8	0.047	<0.055
		13	0.125	<0.33	48.3	9.1	0.003	<0.036
CCX	CCW	14	0.0014	<0.16	1.31	7.2	0.001	<0.022

Notes: * Data obtained by the Chemical Research group.
[a] The plutonium content of the CAFa stream was twice the flowsheet concentration.

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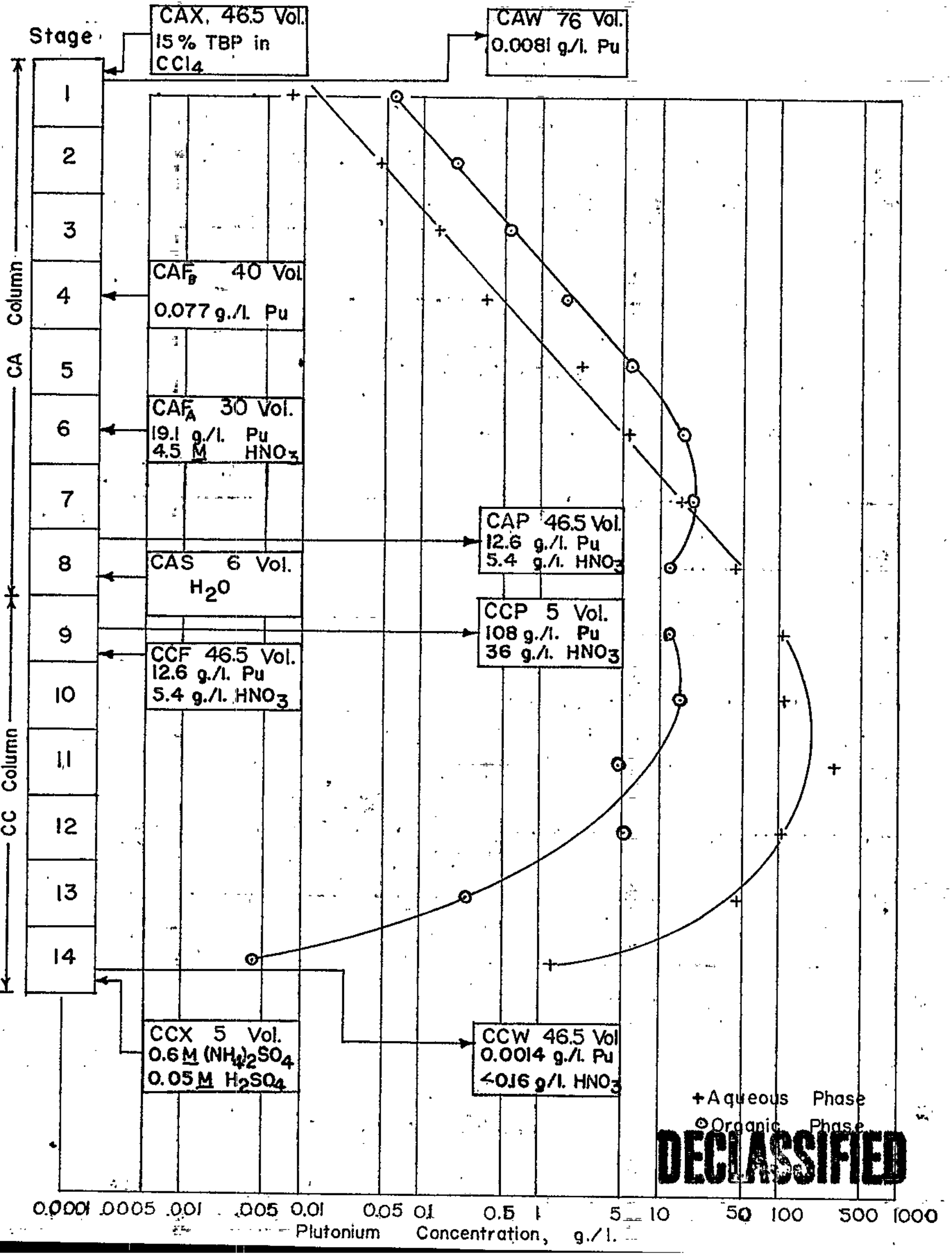
Figure No. 2

PLUTONIUM CONCENTRATION PROFILE

Run No. 18

Countercurrent Batch Equilibration and Separation



A pictorial representation of the mass-transfer and concentration operations that take place within the CA and CC columns is presented in Figure 2. Termed a plutonium concentration profile, the graph is based upon the countercurrent batch equilibrations described above and consists of a plot of stage number vs. the plutonium concentrations in the aqueous and organic phases. Since the abscissa is plotted on a logarithmic scale, the distance between the curves is a measure of the extraction coefficient, E_a^o.

The aqueous stream discharged from the top of the CC column is the product of the Recuplex Process and is sufficiently concentrated to be treated in 234-5 Building Wet Chemistry. The sulfate ion present in the CCP stream remains in the supernatant solution that results from the subsequent oxalate-precipitation step, and is recycled to the Recuplex facilities. The possible effects that the sulfate ion might have on the extraction efficiency of plutonium in the CA column were investigated by batch extractions of an aqueous feed which simulated the aqueous phase at an intermediate stage within the extracting section.(13) The simulated solution comprised 0.17 M La^{3}, 0.60 M Al$(NO_3)_3$, 0.65 M Mg^{+2}, 0.1 M CA^{+2}, 0.05 M AlF$_3$, 3.7 M HNO$_3$, and 0.048 M $(HN_4)_2 SO_4$. The solvent was 30% TBP in AMSCO 125-90W (1 M HNO$_3$), while the volume of organic to aqueous was 1:4. A similar solution containing no sulfate ion was extracted in the same manner for purposes of comparison. The results, presented in Table XI, demonstrated that plutonium extraction is not appreciably affected by the sulfate ion.

TABLE XI

EFFECT OF SULFATE ON CA COLUMN EXTRACTION*

Organic: 30% TBP in AMSCO 125-90W

Aqueous: Simulated F-10-P, 2BP, and slag-and-crucible solution at intermediate stage.

Volume Ratio: organic: aqueous: 1:4

Consecutive Batch Extraction	Sulfate Present		Sulfate Absent	
	Pu organic g/l	E_a^o a.	Pu organic g/l	E_a^o a.
1	0.169	88	0.168	83
2	0.032	48	0.032	27
3	0.0056	19	0.0061	16
4	0.0017	3.8	0.0021	15

Notes: * Data obtained by the Chemical Research group.
a. The constant amount of less extractable plutonium evidently present was possibly due to the fluoride ion in the solution.

(ii) Chemical flowsheet No. 2.

The chemical flowsheet presented in Figure H-2-15207 differs from the procedure of Figure H-2-15206 in that a product solution containing 60 g/l of plutonium is produced without the use of a complexing sulfate salt in the stripping stream. Dilute nitric acid is introduced into the bottom of the CC column and strips the plutonium from the organic phase during its rise to the aqueous discharge port at the top. Only one-third of the concentrated aqueous stream, however, is removed from the system as the product; the major portion of the stream is returned to the bottom of the scrubbing section in the CA column, thereby eliminating the need for a separate scrub feed. Within the CA column, the plutonium in the concentrated stream is transferred to the organic phase, thus increasing the plutonium concentration in the organic product stream to a point where stripping can be accomplished satisfactorily without a reducing agent or a complexing ion. Such a procedure, termed internal refluxing, permits greater flexibility in column operation and is especially desirable in view of the fact that the feed streams to the Recuplex Process will vary in their quantities and compositions.

Stage equilibrium data and plutonium equilibrium curves for experimental CA and CC columns similar to those in Figure H-2-15207 are presented in Tables XII and XIII and Figure 3. The data were obtained by countercurrent batch equilibrations and separations of the solutions in separatory funnels. The laboratory test procedure (Run No. 19, performed by the Chemical Research group) differed slightly from the procedure offered in the recommended flowsheet. The feed to the theoretical "column" was divided into two streams of different concentrations. The more dilute stream contained slag-and-crucible solution diluted 100 per cent rather than 25 per cent and was introduced into the "column" two stages above the more concentrated feed. The degree of extraction and stripping obtained in the test was excellent. The plutonium loss to the aqueous waste stream was 0.19 per cent, while loss to the organic waste stream was 0.008 per cent. The plutonium concentrations profile, constructed from these data and presented in Figure 4, indicates the manner in which the concentration varies throughout the columns and demonstrates the clearly defined concentration peak obtained in the aqueous phase by means of internal refluxing.

(d) Product purity

The degree of product purity attained by the solvent-extraction step is an important measure of the practicability of the Recuplex Process. The CCP streams from the two batch equilibration tests described above (runs 18 and 19, performed by the Chemical Research group) were submitted for spectrochemical analyses and the relative quantities of elemental impurities present were determined. The analytical results, presented in Table XIV, demonstrate that the Recuplex product stream contains fewer impurities than the product solution from the 231 Building and, consequently, the solution can be transferred directly to 234-5 Building Wet Chemistry. The plutonium in three product solutions from previous batch-equilibration tests (Runs 8, 12, and 13,) was converted to the metallic state by the 234-5 Wet Chemistry process in the laboratories of the Process Assistance (234-5 Development) group. The plutonium in two of the test solutions (Runs 7 and 12) which had been stripped from the organic phase by dilute ammonium sulfate was precipitated as the oxalate, and the plutonium in the third solution which had been stripped by dilute nitric acid

[redacted]

TABLE XII

CA COLUMN STAGE EQUILIBRIUM DATA*

Run Number 19
Countercurrent batch equilibration and separation.

Run Characteristics:

Extraction – 7 stages
Scrubbing – 1 stage
Reflux ratio – 2:1
S-&-C diluted 100%

Stream	Volumes	Composition
CAS	10	49.6 g/1 Pu, 36 g/1 HNO_3
CAFa	35	8.3 g/1 Pu, 230 g/1 HNO_3, 0.8 M ANN
CAFb	40	0.007 g/1 Pu, 180 g/1 HNO_3
CAX	45	15% TBP in CCl_4
CAW	85	0.00056 g/1 Pu
CAP	45	18.6 g/1 Pu, 5.4 g/1 HNO_3

Pu loss to aqueous waste 0.019%.

Stream Operation		Stage No.	Organic Phase		Aqueous Phase		Extraction Coefficient	
In	Out		Pu g/1	HNO_3 g/1	Pu g/1	HNO_3 g/1	E_a^o Pu	E_a^o HNO_3
CAX	CAW	1	0.0038	27.8	0.00056	184	6.8	0.15
		2	0.020	——	0.00137	——	14.6	——
		3	0.055	——	0.0081	——	6.8	——
		4	0.134	——	0.0252	——	5.3	——
CAFb		5	0.410	——	0.075	——	5.5	——
		6	1.78	——	0.323	——	5.5	——
CAFa		7	12.6	25.5	1.69	210	7.4	0.12
CAS	CAP	8	18.6	10.4	23.5	110	0.8	0.09

Note: * Data obtained by the Chemical Research group.

[redacted]

TABLE XIII

CC COLUMN STAGE EQUILIBRIUM DATA*

Run Number 19
Countercurrent batch equilibration and separation.

Run Characteristics:

Stripping - 6 stages
Reflux ratio - 2:1

Stream	Volumes	Composition
CCF	45	18.6 g/l Pu, 5.4 g/l HNO_3
CCX	15	0.1 M HNO_3
CCW	45	0.00044 g/l Pu, 0.16 g/l HNO_3
CCP	5	49.6 g/l Pu, 36 g/l HNO_3
CCR	10	49.6 g/l Pu, 36 g/l HNO_3

Pu loss to organic waste 0.008%.

Stream Operation		Stage No.	Organic Phase		Aqueous Phase		Extraction Coefficient	
In	Out		Pu g/l	HNO_3 g/l	Pu g/l	HNO_3 g/l	E_a^o Pu	E_a^o HNO_3
CCF	CCP CCR	9	12.9	5.42	49.6	35.6	0.26	0.15
		10	3.89	0.98	39.5	19.2	0.099	0.051
		11	0.415	0.65	12.6	12.2	0.033	0.053
		12	0.069	<0.16	0.91	7.46	0.076	<0.021
		13	0.00126	<0.33	0.0044	7.31	0.29	<0.045
CCX	CCW	14	0.00044	<0.16	0.00097	6.56	0.45	<0.024

Note: * Data obtained by the Chemical Research group

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Figure No. 3

PLUTONIUM EQUILIBRIUM CURVE

Run No. 19

Countercurrent Batch Equilibration and Separation

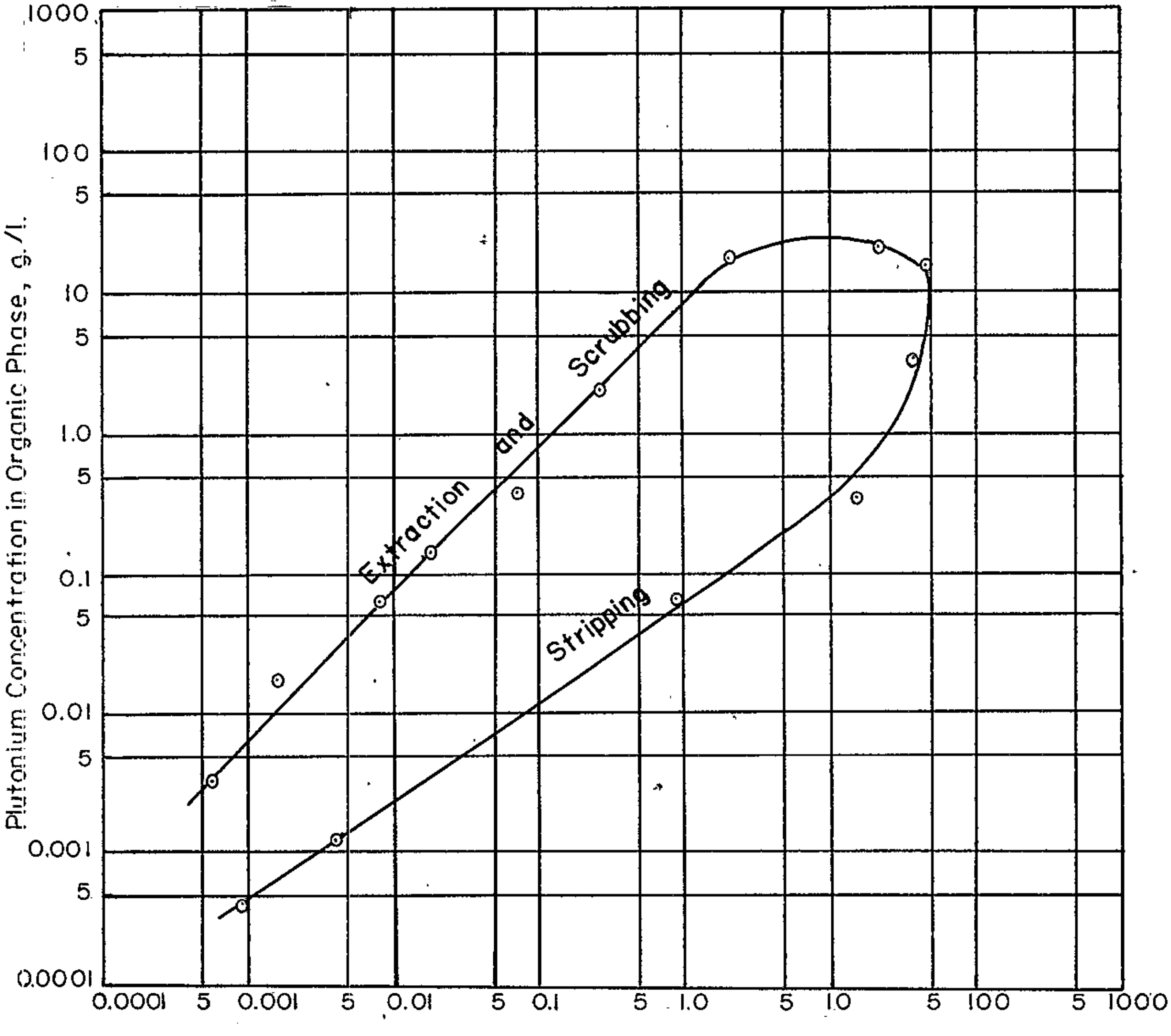

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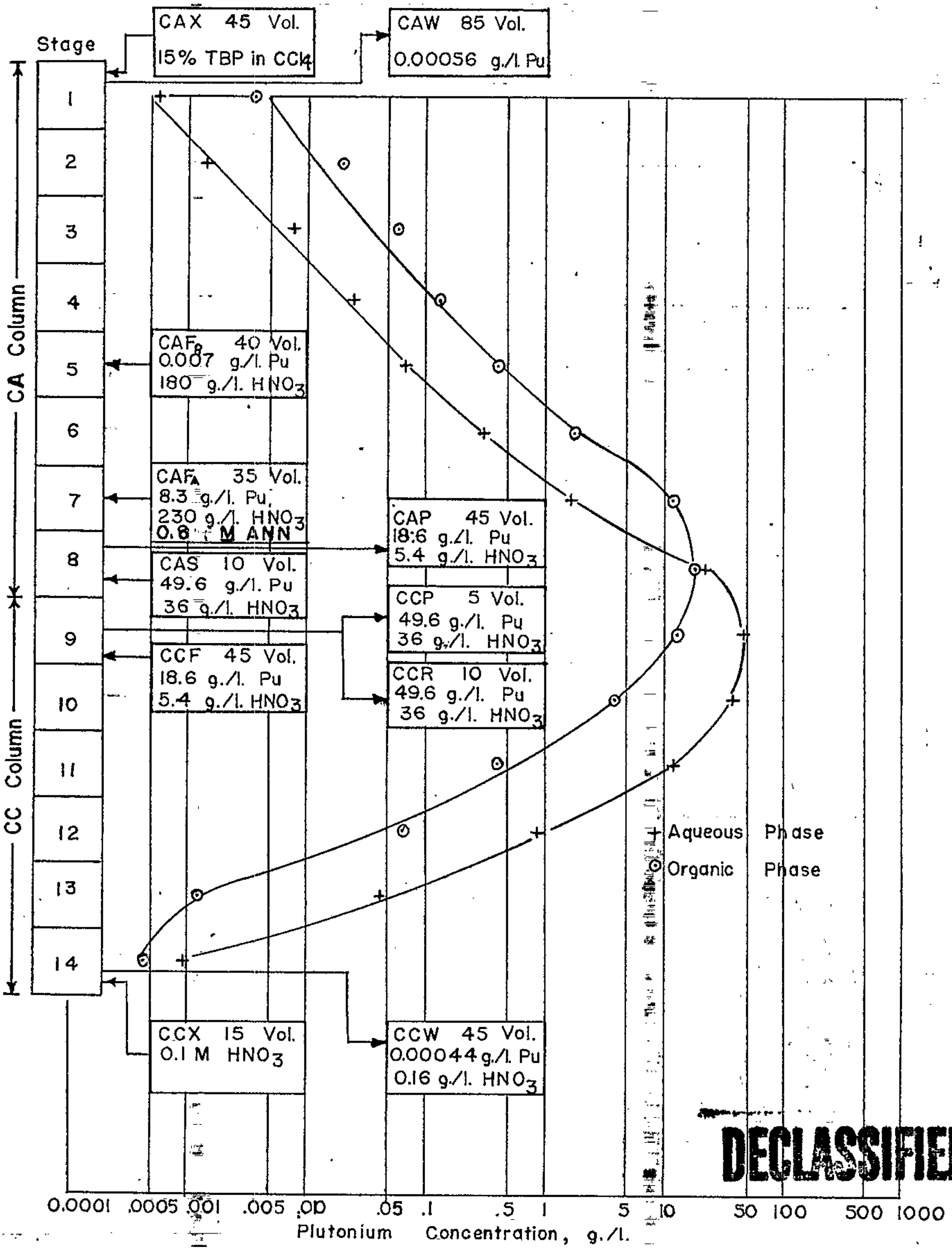
Figure No. 4

PLUTONIUM CONCENTRATION PROFILE

Run No. 19

Countercurrent Batch Equilibration and Separation



as the peroxide. Following conversion to plutonium tetrafluoride and reduction to the metal, the plutonium "button" was analyzed for comparison to standard buttons. The results (Table XIV) demonstrate that the degree of metal purity attained by the Recuplex Process compares favorably with that obtained by the standard operating procedure.

TABLE XIV

PURITY OF THE RECUPLEX PRODUCT STREAM
SPECTRO CHEMICAL ANALYSIS RESULTS

Product	CCP Solution Run 18	CCP Solution Run 19	AT Solution (a.)	Pu Metal Run 8	Pu Metal Run 12	Pu Metal Run 13	Pu Metal (a.)
Run Conditions	Sulfate strip	Reflux-HNO_3 strip	Standard Plant operation	Sulfate strip oxalate pptn.	Sulfate strip oxalate pptn.	HNO_3 strip H_2O_2 pptn.	Standard Plant operation
Elements	ppm[b]	ppm	ppm	ppm	ppm	ppm	ppm
Al	20	< 20	33	< 20	< 20	< 20	14
Ca	200	80	--	50	80	100	21
Cr	50	50	3250	5	< 2	5	25
Fe	1000	1000	8000	200	50	500	372
K	200	2	--	< 2	< 2	< 2	--
La	80	300	> 2000	130	50	30	342
Mg	50	100	80	50	200	200	252
Ni	50	200	1135	50	200	500	71

Notes: (a) Average plant values obtained from Document No. HW-22252, "Flow Diagram - Purification and Fabrication Building - Model 110," (9-26-51) by P. E. Collins.

(b) Ppm = parts per million based upon plutonium.

(e) Selection of the contactor.

The laboratory studies conducted by the Chemical Research group to determine the most feasible extraction and stripping procedures were coordinated with a pilot-plant investigation undertaken by the Chemical Development group to select a contactor having suitable operating and physical characteristics for the Recuplex Process. Among the prime factors considered in the selection of a Recuplex contactor were the ability to process the design throughputs with minimum plutonium loss, operating ease and flexibility in in handling streams varying in quantity and composition, minimum maintenance or replacement difficulties, and a total column height sufficient to permit its containment within the space available in the 234-5 Building. Pilot-plant tests were conducted with two types of contactors, the Fenske stacked extractor and a pulse column. Preliminary studies indicated that the required height of the former unit would not exceed the height of Room 221, 234-5 Building, while the height of the latter was such that it could be encased in a special shroud extending above Room 221.

(1) Fenske stacked extractor.

The Fenske stacked extractor employed by the Chemical Development group in a series of exploratory studies was a 20-stage, 600 ml./stage unit originally designed at the Pennsylvania State College, Petroleum Refining Laboratory. Mixing of the two phases was achieved by means of reciprocating perforated plates, which were supported by a shaft and driven by a hydraulic motor. The flow of liquids was concurrent through the mixing and setting zones, and countercurrent between the stages. The extractor was equipped with facilities which permitted sampling of both phases on each stage, resulting in the direct determination of stage efficiencies. The plate frequency could be varied from 0 to 2000 cycles per minute and the length of the stroke from 0 to 0.37 inch. Under certain conditions, sufficient agitation could be provided to make each stage 100 per cent efficient, that is, the two phases reach equilibrium. The overall dimensions of twenty assembled stages were about 36" high by 15" long and 8" wide.

Throughout all the test runs of the Fenske stacked extractor, uranium was employed as a stand-in for plutonium. Stream conditions were based on three preliminary Recuplex flowsheets, numbers 8A(4), 8B(5), and 8C(6), none of which specified internal refluxing in the CA column as in Figure H-2-15207. The principal differences between the three flowsheets and the alternate flowsheet in Figure H-2-15206 were the stream flow ratios and ANN content, and the division of the CA column feed into a concentrated CAFa stream and a dilute CAFb stream. In all cases the CAFa stream comprised simulated F-10-P and 2BP solutions, while the CAFb stream in some runs was filtered S-&-C solution, prepared from crucibles in which uranium reductions had been performed. The selective solvent was 15 per cent by volume of TBP in a CCl_4 diluent. Water was employed as the CA column scrubbing agent in each test run. The stripping solution specified in flowsheets 8B and 8C was ammonium sulfate, and in flowsheet 8A, dilute nitric acid.

The results of the stage-efficiency tests of the CA and CC columns, summarized(17) in Tables XV and XVI, demonstrate that low uranium losses and adequate extraction efficiencies are obtainable under the conditions of preliminary flowsheets 8B and 8C. The uranium losses in 17 to 19 stages of the CA column ranged from 0.01 to 0.9 per cent under various operating conditions, while estimated Murphree aqueous-phase stage efficiencies ranged from 40 to 60 to about 100 per cent. In the CC column uranium losses in twenty stages ranged from 0.03 to 0.5 per cent, while estimated Murphree solvent stage efficiencies ranged from approximately 30 to 80 per cent.

The flooding performance of the Fenske stacked extractor, however, was highly erratic, not only under stream conditions based upon the preliminary flowsheets, but also in experiments in which pure CCl_4 was contacted countercurrently with distilled water. The data, presented in Tables XVII and XVIII, indicate that under the same operating conditions the flooding capacity of the extractor varied over a four-to five-fold range.

[redacted]

TABLE XV

CA COLUMN STAGE EFFICIENCY

FENSKE STACKED EXTRACTOR*

Flow-sheet	CAF' (a)	Through-put, Ml./Min., Sum of Both Phases	Inter-face Posi-tion	Ampli-tude, In.	Fre-quency, Cyc./Min.	U Loss %		Over-all Stage Efficiency, (c) %			Murphree Aqueous-Phase Stage Efficiency, (c) %		
						1st 3 Stages	19 Stages (e)	1st 3 Stages	1st 3 Stages After CAF'	19 Stages (e)	1st 3 Stages	1st 3 Stages After CAF'	19 Stages (e)
8A	None	260	Bottom	0.31	400	0.2	0.1	80		15	85		20
8A	None	270	Bottom	0.31	400	0.04	0.9	91		9	98		24
8A	None	620	Bottom	0.31	500	1.0	0.01	67		20	75		25
8B	Simulated	770	Bottom	0.31	500	0.01	28	100		7	100		10
8B	Simulated	780	Bottom	0.31	400	0.03	17	100		10	100		15
8C	Actual(b) S & C	220	Bottom	0.31	400	0.09	0.05(d)	71	47	17(d)	94	81	42(d)
8C	Actual(b) S & C	390	Top	0.31	400	1.0	0.04(d)	74	31	17(d)	94	64	40(d)

Notes: * Data obtained by the Chemical Development group.(17)

(a) Stream Flow Ratios:

	CAS:	CAFa:	CAFb:	CAX:	CCX
Flowsheet 8A:	12:	50:	50:	30:	20
Flowsheet 8B:	15:	50:	100:	50:	20
Flowsheet 8C:	15:	50:	75:	50:	20

(b) SiO_2 content about 50 p.p.m.

(c) Based on estimated approximate phase equilibrium data. The stated stage efficiencies are for uranium transfer.

(d) 17 stages

(e) The relatively high apparent 19-stage (and 17-stage) uranium losses and corresponding low apparent stage efficiencies may be due to the presence of small concentrations of "inextractable" uranium. In the case of all except the last two of the runs tabulated above there was also some uranium contamination of the aqueous extractor effluent (CAW) by solution from the concentrated end bypassing the extractor via the standpipe used for interface control, through a faulty valve.

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TABLE XVI

CC COLUMN STAGE EFFICIENCY

FENSKE STACKED EXTRACTOR*

Flowsheet$^{(a)}$	Throughput, Ml./Min., Sum of Both Phases	Interface Position	Amplitude, In.	Frequency, Cyc./Min.	U Loss, %		Overall Stage Efficiency,$^{(c)}$%		Murphree Solvent Stage Efficiency,$^{(c)}$%	
					3 Stages	20 Stages	1st 3 Stages	20 Stages	1st 3 Stages	20 Stages
8A	400	Bottom	0.31	600	20	0.03	55	16	65	35
8A	1060	Bottom	0.31	500	50	0.5	30	17	30	30
8C	390	Bottom	0.31	600	5	0.15	70	12	80	30
8C	400	Bottom	0.31	400	40	0.03	35	14	35	35
8C	510	Bottom	0.31	400	18$^{(b)}$	0.03	13$^{(b)}$	9	30$^{(b)}$	30
8C	490	Top	0.31	400	15$^{(b)}$	0.08	35$^{(b)}$	9	60$^{(b)}$	30

Notes: * Data obtained by the Chemical Development group.$^{(17)}$

(a) Stream Flow Ratios:

	CAS:	CAFa:	CAFb:	CAX:	CCX:
Flowsheet 8A:	12:	50:	50:	30:	20:
Flowsheet 8B:	15:	50:	100:	50:	20:
Flowsheet 8C:	15:	50:	75:	50:	20:

(b) 5 stages

(c) Based on estimated approximate phase equilibrium data. The stated stage efficiencies are for uranium transfer.

[redacted]

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TABLE XVII

CA COLUMN FLOODING CAPACITY

FENSKE STACKED EXTRACTOR*

Flowsheet	CAF'	Interface Position	Amplitude, In.	Frequency, Cycles/Min.	Flooding Capacity Ml./Min., Sum of Both Phases
ANN vs. CCl_4(a)	(a)	Bottom	0.19	600	<130 to >1120
ANN vs. CCl_4(a)	(a)	Top	0.19	600	< 90 to >1740
8A	Simulated	Bottom	0.19	600	<200 to 500
8A	None	Bottom	0.31	400	<200 to >620
8B	Actual S.& C.(b)(c)	Bottom	0.31	400	<220 to >1600
8A	None	Bottom	0.31	600	100
8A	None	Bottom	0	0	600
8B	Actual S.& C.(b)(c)	Bottom	0.31	500	1100
8C	Actual S.& C.(b)(d)	Bottom	0.31	400	330
8C	Actual S.& C.(b)(d)	Bottom	0.31	0 to 900	> 320
8C	Actual S.& C.(b)(d)	Bottom	0	0	> 750
8A	Simulated	Top	0.19	600	<200
8C	Actual S.& C.(b)(d)	Top	0.31	400	<380 to > 550

Notes: * Data obtained by the Chemical Development group.(17)

(a) Composition of CAS, CAF, CAF': 390 g./l. $Al(NO_3)_3 \cdot 9H_2O$
Composition of CAS: 100% CCl_4
CAS:CAF:CAF':CAX vol. flow ratio = 12:50:50:30

(b) Filtered actual slag-and-crucible solution, prepared from 234-5 crucibles in which uranium reduction had been carried out.

(c) SiO_2 content about 15 p.p.m.

(d) SiO_2 content about 50 p.p.m.

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TABLE XVIII

CC COLUMN FLOODING CAPACITY

FENSKE STACKED EXTRACTOR*

Flowsheet	Interface Position	Amplitude, In.	Frequency, Cyc./Min.	Flooding Capacity, Ml./Min., Sum of Both Phases
H_2O vs. CCl_4 (a)	Bottom	0.19	600	3200
H_2O vs. CCl_4 (b)	Bottom	0.31	400	700 to 2800
H_2O vs. CCl_4 (b)	Bottom	0	0	2000
H_2O vs. CCl_4 (a)	Top	0.19	600	1600
8A	Bottom	0.31	600	700
8C	Bottom	0.31	400	400 to 850
8C	Bottom	0.31	600	> 390
8C	Top	0.31	400	> 1000

Notes: * Data obtained by Chemical Development group.(17)

(a) $H_2O:CCl_4$ vol. flow ratio = 2:3.

(b) $H_2O:CCl_4$ vol. flow ratio = 1:3.

[redacted]

(ii) Pulse columns.

In view of the difficulties experienced in the operation of the Fenske unit, an investigation of pulse columns as suitable contactors for the Recuplex Process was initiated by the Chemical Research and Chemical Development groups. A pulse column is a countercurrent flow, vertical extractor having three principal components: (1) a pulse generator, which is a single or multiple piston-cylinder system with an adjustable-length stroke driven by a constant speed driver; (2) a transmitting system, which consists of a constant diameter pipeline joining the generator to the base of the column; and (3) the pulse column proper, which consists of a constant-diameter, vertical cylinder containing fixed, horizontal, close-fitting, perforated plates, surmounted by a larger-diameter settling chamber. Intimate mixing of the two phases is achieved by means of the pulse generator which repeats the passage of an incremental volume of liquid through each plate a certain number of times. The number of passages is determined by the amplitude and frequency of the pulse.

The extraction operation within a pulse column is more related to the operation within a packed column than to the mechanism in the Fenske extractor since the two phases are not mixed and separated at each plate. Consequently, the contacting efficiency of the pulse column must be expressed in terms of the HETS concept rather than the individual plate efficiency. The Height-Equivalent-to-a-Theoretical Stage is defined as that height of packing material (in this case, close-fitting perforated plates) which is equivalent to a theoretical plate on which the two phases reach equilibrium conditions. Thus, if the number of theoretical stages necessary to achieve a desired degree of separation are known, the necessary column height can be calculated from the HETS value.

Preliminary tests conducted by the Chemical Research group with a once-inch diameter pulse column indicated good operation at throughputs up to 600 gal/(hr.)(sq. ft.) and a flooding capacity of approximately 800 gal/(hr.)(sq. ft.). A more extensive investigation was initiated by the Chemical Development group with a 3-inch i.d. glass pulse column. The plate section of the column was 9.1 feet high and contained stainless-steel plates spaced two inches apart and perforated with 1/8 inch holes to provide a 23 per cent free area.

Uranium was employed as a stand-in for plutonium in all the experimental runs. Stream conditions approximated those in chemical flowsheet HW No. 2 (Figure H-2-15207).

The test results, summarized in Tables XX through XXIII, demonstrate that a throughput of 320 gal/(hr.)(sq. ft.), sum of both phases, corresponding to flow conditions in Figure H-2-15207 is approximately 50 to 70 per cent of the flooding capacity of a three-inch CA column. The throughput corresponding to flow conditions in Figure H-2-15206 is 260 gal/(hr.)(sq. ft.). Since the extraction efficiency is relatively insensitive to volume velocity variations in this range, a three-inch pulse column is applicable to either flowsheet for the extraction and scrubbing operations. The stripping efficiency is not significantly effected by variations in throughput over

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the range of 120 to 590 gal./(hr.) (sq. ft.), sum of both phases. A two-inch i.d. pulse column is consequently suitable for the stripping operation under the conditions in both chemical flowsheets, the corresponding volume velocities being 130 and 300 gal/(hr.)(sq. ft.). If future studies can establish the stripping operation to be satisfactory at throughputs in the order of 50 gal/(hr.)(sq. ft.), it will be possible to specify the CC column diameter as three inches and thus permit the use of identical pulse generators.

Extraction and flooding characteristics were correlated with the arithmetic product of pulse amplitude and frequency (inches/min.) under the particular flowsheet conditions investigated, optimum performance was obtained between 30 and 100 inches/min. More intimate mixing occurred throughout the columns when the aqueous phase was the continuous medium.

Tentative specifications for the Recuplex pulse columns as recommended by the Chemical Development group are presented in Table XIX. The number of theoretical stages in the extraction, scrubbing and stripping sections required to meet the conditions of chemical flowsheet No. 2 are 6.5, 1 and 5 respectively. The maximum HETS values (Height-Equivalent-to-a-Theoretical Stage) for the corresponding sections are approximately 3, 3, and 4 feet. Consequently, minimum plate-section heights of 19, 3, and 20 feet are required to meet the Recuplex design goals. A safety margin was incorporated into the tentative column specifications by increasing the extraction and stripping plate-section heights to 22 and 26 feet respectively. The height of the scrubbing section was not increased in order to avoid excessive internal refluxing of the plutonium within the CA column. The resultant overall column height, twenty-nine feet including end sections, is sufficient to permit containment of the columns within the 234-5 Building.

In view of the more reliable performance characteristics of the pulse columns, this type of contactor is felt to be more suited for use in the Recuplex Process. A second factor influencing the selection of the pulse column is the fact that replacement of the Fenske unit would be costly and time-consuming due to the complex castings of the assembly. Although the use of the Fenske contactor would not require extension of the unit through the ceiling of Room 221 in the 234-5 Building, the necessary space for the special shroud can be provided without undue construction difficulties and without interferring with present processing operations. On the basis of these considerations, the pulse column is recommended as the more satisfactory and reliable unit.

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TABLE XIX

TENTATIVE SPECIFICATIONS
RECUPLEX PULSE COLUMNS*

	CA Column	CC Column
Overall height, feet	29	29
Plate-section height, feet		
Minimum, for extraction	19	
Specified, for extraction	22	
Minimum, for scrubbing	3	
Specified, for scrubbing	3	
Minimum, for stripping		20
Specified, for stripping		26
End-section height, feet		
Top end-section	2	1.5
Feed point	0.5	--
Bottom End-section	1.5	1.5
Plate-section diameter, in.	3	2
Perforated plates	Stainless steel, spaced 2" apart, 0.125 in. holes, 23% free area.	
Pulse amplitude (fixed), in.	1.0	1.0
Pulse frequency (varied), cyc/min.	30-70	40-100
Continuous phase	Aqueous	Aqueous

Note: * Data obtained by the Chemical Development group.

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TABLE XX
CA COLUMN CONTACTING EFFICIENCY
PULSE COLUMN*

	Operating Time				Pulse Characteristics						
	Approach To Steady State		At Steady State								
Run No.	Minutes	Displacements(a)	Minutes	Displacements(a)	Amplitude, (Inches)	Frequency, (Cycles/Min.)	Ampl. x. Freq. (Inches/Min.)	Throughput, Sum of Both Phases Gal./(Hr.)(Sq.Ft.)	Aqueous To Organic Volume(d) Flow Ratio	U Loss, (%)	HETS (b)(c) (Ft.)
EXTRACTION SECTION											
3"-2-AR	60	2.9	60	2.9	ca. 0.2	60	12	472	2.15	21	19.7
3"-3-AR	60	2.8	60	2.8	ca. 0.1	85	9	453	2.15	16	16.8
3"-4-AR	60	2.8	60	2.8	ca. 0.05	100	5	459	2.06	14	15.6
3"-6-AR	60	2.4	60	2.4	1.25	40	50	401	2.17	0.018	3.5
3"-7-AR	90	2.6	90	2.6	1.25	40	50	273	2.49	0.021	3.4
3"-9-AR	60	2.2	60	2.2	0.4	50	20	367	1.86	9.7	13.6
3"-10-AR	60	2.5	60	2.5	0.25	60	15	411	1.96	19	18.9
3"-11-AR	80	2.2	40	1.1	0.41	60	25	246	2.37	17	16.6
3"-14-AR	80	2.2	80	2.2	0.40	80	32	251	2.44	2.2	7.7
3"-15-AR	80	2.2	80	2.2	0.50	100	50	254	2.35	0.088	4.1
3"-16-AR	80	2.2	80	2.2	0.85	60	51	261	2.12	0.051	4.0
3"-18-AR	60	2.6	60	2.6	0.4	75	30	408	2.17	2.3	8.1
3"-19-AR	80	2.3	80	2.3	0.85	40	34	249	2.60	4.1	9.8
3"-20-AR	60	2.4	60	2.4	0.85	40	34	375	2.03	3.0	9.2
3"-22-AR	60	2.6	60	2.6	0.85	60	51	381	2.71	0.53	2.2
3"-24-AR	60	2.5	60	2.5	0.85	40	34	387	2.11	0.74	6.5
3"-26-AR	120	2.8	120	2.8	0.85	60	51	207	2.58	0.048	3.9
3"-28-AR	120	2.7	120	2.7	0.85	60	51	198	2.60	0.039	3.9
3"-29-AR	120	2.7	120	2.7	1.25	40	50	196	2.55	0.083	4.3
SCRUBBING SECTION											
3"-2-ARS	120	1.1	60	0.6	0.85	40	34	316	0.210	0.25	1.9

Notes:

* Data obtained by the Chemical Development group.(19)
a. Continuous (aqueous) phase flow rate (in ml./min.) multiplied by the operating time (in minutes) and divided by the column volume (in ml.).
b. Height equivalent to a theoretical stage.
c. Calculated by use of approximate equilibrium data.
d. Flowsheet conditions approximated those in Figure H -2-15207.

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TABLE XXI
CC COLUMN CONTACTING EFFICIENCY
PULSE COLUMN*

	Operating Time				Pulse Characteristics			Throughput,	Aqueous To		
	Approach To Steady State		At Steady State						Organic		HETS
Run No.	Minutes	Displacements (a)	Minutes	Displacements (a)	Amplitude, (Inches)	Frequency, (Cycles/Min.)	Ampl. x. Freq. (Inches/Min.)	Sum of Both Phases Gal./(Hr.)(Sq Ft.)	Volume(d) Flow Ratio	U Loss, (%)	(b)(c) (Ft)
3"-2-CR	60	2.1	60	2.1	1.25	40	50	592	0.598	6.8	2.9
3"-3-CR	90	2.1	90	2.1	1.25	40	50	398	0.622	4.6	2.8
3"-6-CR	240	2.0	180	1.5	0.85	80	68	134	0.673	0.25	3.5
3"-8-CR	70	1.9	80	2.1	0.81	80	65	419	0.660	0.081	3.0
3"-9-CR	60	1.4	60	1.4	0.85	40	34	391	0.651	9.2	6.1
3"-10-CR	90	2.4	90	2.4	0.85	80	68	433	0.696	0.24	3.5
3"-11-CR	240	2.2	240	2.2	0.85	80	68	129	0.834	0.58	4.5
3"-12-CR	80	2.2	80	2.2	1.25	40	50	427	0.734	3.8	4.8

Notes:

* Data obtained by the Chemical Development group.(19)

a. Continuous (aqueous) phase flow rate (in ml./min) multiplied by the operating time (in minutes) and divided by the column volume (in ml.).

b. Height equivalent to a theoretical stage.

c. Calculated by use of approximate equilibrium data.

d. Flowsheet conditions approximated those in Figure H -2-15207.

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TABLE XXII
CA COLUMN FLOODING CAPACITY
PULSE COLUMN

Run No.	Pulse Characteristics Amplitude, (Inches)	Pulse Characteristics Frequency, (Cycles/Min.)	Pulse Characteristics Ampl. x. Freq., (Inches/Min.)	Aqueous To Organic Volume(d) Flow Ratio	Flooding Capacity Sum of Both Phases Gal./(Hr.)(Sq.Ft.)
EXTRACTION SECTION					
3"-1-AR(A)	ca. 0.1	80	8	1.98	650 ± 50
3"-1-AR(B)	ca. 0.05	115 ± 5	6	2.14	450
3"-5-AR	1.25	40	50	2.13	500 ± 100
3"-12-AR	0.85	75 ± 5	64	1.89	243
3"-13-AR	0.48	125 ± 5	60	2.19	244
3"-17-AR	0.48	105	50	2.21	401
3"-21-AR	0.85	75 ± 5	64	2.07	376
3"-23-AR	1.0	55 ± 5	55	2.13	376
3"-25-AR	0.85	60	51	---	340 ± 50
3"-27-AR	1.0	55 ± 5	55	2.34	212
3"-30-AR	1.25	40	50	2.70	400
SCRUBBING SECTION					
3"-3-ARS	0.85	40	34	0.180	740
3"-4-ARS	0 85	40	34	0 174	735

Notes:

* Data obtained by the Chemical Development group.(19)

a. Flowsheet conditions approximated those in Figure H-2-15207

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[redacted]

TABLE XXIII
CC COLUMN FLOODING CAPACITY
PULSE COLUMN

Run No.	Pulse Characteristics Amplitude, (Inches)	Frequency, (Cycles/Min.)	Ampl. x. Freq., (Inches/Min.)	Aqueous To Organic Volume (d) Flow Ratio	Flooding Capacity Sum of Both Phases Gal./(Hr.)(Sq.Ft.)
3"-1-CR	1.25	40	50	0.587	985
3"-4-CR	1.25	40	50	0.548	980
3"-5-CR	0.85	> 110	94	0.695	119
3"-7-CR	0 85	> 90	77	0.705	419

Notes:

* Data obtained by the Chemical Development group.(19)

a. Flowsheet conditions approximated those in Figure H-2-15207.

[redacted]

(3) Waste Treatment.

The proposed Recuplex Process discharges three waste streams: (a) the silica cake from the filtration step in the slag-and-crucible solution preparation, (b) the aqueous waste stream from the extraction-and-scrubbing column, and (c) the dilute caustic stream associated with treatment of the organic solvent. Laboratory studies have been made of the plutonium loss to these streams and the most suitable manner in which they may be either discarded or recycled.

(a) Silica cake from slag-and-crucible solution preparation.

The design goal for recovery of the slag-and-crucible material is 90 per cent of the plutonium present in the material. It is believed that a dissolution procedure using a nitric acid-hydrofluoric acid mixture (as represented in the Slag-and-Crucible Solution Preparation-Chemical Flowsheet HW No. 1) will provide a recovery yield of 90 per cent, or greater, and eliminate the need for further treatment of the silica cake. In two of the runs described in Table V, the entire silica cake was hydrofluorinated and the residue was metathasized with KOH and filtered. The plutonium-bearing solids were dissolved in nitric acid and the solutions analysed for plutonium. The results established that in both instances approximately 0.8 per cent of the total plutonium content was associated with the silica cake. If the plutonium loss to the SiO_2 cake is 10 per cent or less in plant-scale processing of slag-and-crucible material, the cake will be slurried and transferred to underground storage. Semi-works tests performed with the sintered stainless steel filter described in Table VII indicated that the cake can be readily removed from the plate by backwashing with water and air at 10 psig. Agitation is necessary to provide a uniform suspension with water and insure ease of transfer. Properties of the solid and suspended cake are presented in Tables XXIV and XXV. It is estimated that with a 1:2 volume ratio of cake to liquid, approximately 100 gal/mo. of silica slurry will be transferred to underground storage.

In the event that the flowsheet in Figure H-2-15213 would be employed, there are three alternate procedures available for processing the silica cake: (1) storing the cake separately for later recovery by modifications to the Recuplex equipment; (2) processing the stored silica in the Hot Semi-works Plant; and (3) treating the silica cake with hydrofluoric acid on a semi-routine basis in the platinum-lined skull dissolver and returning the dissolved plutonium to the Recuplex Process. Proposed changes in the 234-5 Building operations would alleviate the silica processing problem by reducing the quantity of silica requiring treatment, i.e., the use of thin-walled crucibles and a more highly purified MgO in crucible fabrication.

The preliminary design of the filter for separating the silica from the dissolved slag-and-crucible solution has included facilities for either suspending the cake and jetting to waste storage or removing the solid for further treatment.

(b) Aqueous waste stream from the CA column.

The process design goal for the plutonium loss to the aqueous waste stream discharged from the CA column is 0.01 per cent with an upper limit of 0.2 per cent. With a waste loss of this low order of magnitude, further processing of the CAW would be economically unsound. If the plutonium content of an individual CAW batch should exceed the specified maximum, provisions have been included in the design for plutonium recovery by recycling the material to the solvent-extraction columns. Such a procedure would require a temporary shutdown of the virgin throughput.

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TABLE XXIV

FILTRATION OF SLAG AND CRUCIBLE SOLUTIONS

FILTER CAKE PROPERTIES*

Dissolution: Dissolution according to Los Alamos procedure.

Solution: Slag and crucible dissolver solution (U as stand-in for Pu).

$Mg(NO_3)_2$	2.0 M	SiO_2 (from crucible) 1.5 g./l.
$Ca(NO_3)_2$	0.29 M	Filter Aid (Hy-Flo Super Cel), as noted.
$Al(NO_3)_3 \cdot 9H_2O$	0.56 M	
HNO_3	5.0 M	Volumes, 20 liters/crucible
UNH	0.0005 M	Sp. Gr. 1.40

Filtration: Filter medium was either micrometallic, sintered stainless steel plate with precoat or glass filter cloth without precoat. Total pressure difference was 16" of mercury (vacuum on filtrate receiver). At the end of filtration, cake was sucked free of excess liquid.

Notes and Observations: The cake has been easily removed from the Micrometallic filter plate by backwashing with mixed water and compressed air at 10 lb./sq. in. ga.

Agitation is necessary for slurrying the cake with water.

Cake containing filter aid is more easily slurried than cake without filter aid.

Run	Filter Aid, g./l. in Dissolver Solution	Sp. Gr. of Cake	Cake Volume cu.in./Crucible	Cake Analysis, Weight %	
				SiO_2	UNH
A	0	---	31	---	---
B	0	1.49	50	---	---
C	2.5	---	22	---	---
D	2.5	1.34	28	3.5(a)	0.016

Note: * Data obtained by the Chemical Development group.(18)

(a) Material balance around dissolver and filter indicates 15% SiO_2 in cake.

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TABLE XXV

FILTRATION OF SLAG-AND-CRUCIBLE SOLUTIONS

VISCOSITY OF FILTER CAKE SUSPENSIONS*

The cake from run D (TableXXIV) was slurried with water to various volume ratios of water to settled solids. Viscosity of the slurry was measured with a Brookfield viscosimeter (No. 1 spindle): (a)

Slurry Dilution, Vol. ratio of Supernate to Settled Solids	Spindle Speed, rev./min.	Viscosity, Centipoise
1:1	100	21.2
	50	17.2
	20	15.0
	10	15.0
2:1	100	16.6
	50	13.0
	20	13.0
	10	10.0
6:1	100	11.9
	50	8.0
	20	9.0
	10	7.0

Notes: * Data obtained by the Chemical Development group.(18)

(a) Accuracy of the instrument is doubtful below 15 centipoise.

[redacted]

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Representative waste-loss data obtained by the countercurrent, batch-equilibration tests described in the preceding section (Tables IX and XII) indicated a plutonium loss to the CAW stream of 0.11 per cent and 0.019 per cent for Runs 18 and 19 respectively. The CAW loss in Run 18 was within the acceptable limit. It is estimated that this loss would have been approximately 0.025 per cent if ANN had been added to the CAF stream.

Neutralization of the aqueous waste from the CA column produces a large quantity of gelatinous, slow-settling solids, the volume of which, even after centrifugation, is more than half that of the acid waste. Removal of the solids from the alkaline slurry results in a total alpha decontamination of approximately 1000. The supernatant, however, contains approximately ten times the alpha activity which is present in solutions customarily cribbed at Hanford. The process design, therefore, has provided that the CAW stream be neutralized and transferred directly to underground black iron storage tanks. The storage facilities will be adequate to receive approximately 10,000 gals/mo. of neutralized CAW slurry.

(c) <u>Aqueous waste from the solvent-treatment tower.</u>

Studies conducted by the Chemical Research group indicate that under prolonged exposure to alpha irradiation tributyl phosphate is decomposed, forming dibutyl phosphate. The presence of DBP in the organic solvent results in the fixation of plutonium in a stable compound which is preferentially soluble in the organic phase and cannot be stripped into an aqueous solution. The data, presented in Table XXVI, demonstrate that after 100 hours contact time the concentration of fixed plutonium is approximately 1 per cent of the maximum plutonium concentration in the organic phase. Under the conditions of the chemical flowsheet in Figure H-2-15207, 455 liters of organic solvent would be exposed to alpha irradiation approximately one hour per day and at the end of a 3-month period would contain approximately 0.2 g/l of fixed plutonium. This represents 0.03 per cent of the processed plutonium.

TABLE XXVI

DECOMPOSITION OF TBP DUE TO ALPHA IRRADIATION*

Procedure: TBP was exposed to alpha irradiation by preparing a solution containing 22 g/l Pu in 15% TBP-85% CCl_4. At various intervals of time, aliquots were removed and stripped with five equal volumes of 0.1 M HNO_3. The Pu remaining in the organic phase was then analyzed.

Contact Period (hours)	Pu Remaining in Organic Phase After Stripping (mg/l)	(%)
0 - 0.1	3.7	0.02
2	2.0	0.01
2.5	2.6	0.01
24	35	0[illegible]
96	215	1.0

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Note: * Data obtained by the Chemical Development group.

[redacted]

It is recommended that the DBP and fixed plutonium be removed from the organic solvent following operating periods of 100 contact hours. Preliminary studies indicate that efficient treatment of the solvent can be accomplished by contacting the stream with an aqueous solution of 5 per cent sodium hydroxide in a packed tower. Since the suggested flow ratio of organic to aqueous phases is 20:1, the resultant aqueous solution, under conditions of the chemical flowsheet in Figure H-2-15207, would have a plutonium concentration of approximately 4 g/l. Although the stream represents a waste loss of only 0.03 per cent, it is currently believed that a procedure can be developed whereby the plutonium can be recovered.

(4) Corrosion Studies.

An investigation of the possible corrosion effects that might be exhibited by the solutions in various phases of the Recuplex Process was initiated by the Chemical Development and Process Assistance (234-5 Development) groups. Various materials were exposed to boiling slag-and-crucible dissolver solutions prepared according to the Preliminary Chemical Flowsheet in Figure H-2-15213 and the HW Chemical Flowsheet No. 1 in Figure H-2-15084. Similar tests were conducted with the aqueous waste solution from the CA column and the two-phase solution mixture corresponding to the feed point in the CA column. The corrosion rates exhibited by the stainless steels in the thermally cold process solutions were less than 0.0005 inches per month. All materials exposed to boiling dissolver solutions simulating conditions in the preliminary flowsheet for slag-and-crucible solution preparation (Figure H-2-15213) corroded at average rates less than 0.0015 in./mo. Samples exposed to the corresponding vapors indicated a maximum corrosion rate of 0.0027 in./mo. These values are not excessive and fall within the generally accepted corrosion rate for stainless steels (0.002 in./mo.). The experimental procedures and the corrosion data obtained in these tests are summarized in Tables XXVII through XXX.

A preliminary investigation of the resistance of types 304 ELC, 309 SCB, and 347 stainless steels to the dissolver, solution in HW No. 1 chemical flowsheet for slag-and-crucible solution preparation (Figure H-2-15084) indicate a corrosion rate of 0.0008 - 0.004 in./mo. It is believed that under these conditions periodic replacement of the dissolver vessel would be economically feasible. The experimental results are presented in Table XXXI.

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TABLE XXVII

CORROSION STUDIES CAW STREAM FROM CA COLUMN

AT ROOM TEMPERATURE*

Test Specimens: Type 304 ELC and Carpenter 20 stainless steel. Specimen immersed in liquid at 70-80°F. Only sensitized 304 ELC and parent metal Carpenter 20 are to be tested.

Procedure: Specimens immersed in synthetic CAW prepared according to 8A Recuplex flowsheet. Specimens exposed to CAW equilibrated with CCl_4 + 15% TBP, and to CAW equilibrated with hydrocarbon + 15% TBP. Solution changed once per week.

Results:

Test Specimen		Solution, Aqueous CAW Equilibrated as Indicated	Average Overall Corrosion Rate After 504 Hours (21 Days) Exposure inches/month
Material	Type		
Type 304 ELC	Sensitized	CCl_4 + 15% TBP	Nil
		HC + 15% TBP	Nil
Carpenter 20	Parent metal	CCl_4 + 15% TBP	Nil
		HC + 15% TBP	Nil

TABLE XXVIII

CORROSION STUDIES AGITATED TWO PHASE SYSTEM

AT FEED POINT OF CA COLUMN*

Test Specimens: Type 304 ELC and 316 stainless steel. Specimens immersed in the agitated phases at room temperature. Only sensitized 304 ELC and parent 316 were tested.

Procedure: CAS, CAF, CAF', and CAX (Recuplex 8A Flowsheet) mixed in ratio of 12:50:50:30 and agitated in beaker with glass stirrer. Metal samples totally immersed in Agitated system. Solution changed weekly.

Results:

Test Specimens		Average Overall Corrosion Rate After 442 Hours (18 days) Exposure inches/month
Material	Type	
Type 304 ELC	Sensitized	Nil
Type 316	Parent Metal	Nil

Note: * Data obtained by the Chemical Development group.(18)

[redacted]

[redacted]

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TABLE XXIX

CORROSION STUDIES-CAW STREAM FROM CA COLUMN AT BOILING POINT*

Test Specimens: Type 309 SCb, 304 ELC, and 347 stainless steel. Each specimen exposed to both boiling liquid and condensing vapors. Specimens are both parent metal and sensitized metal.

Procedure: CAW prepared synthetically based on Recuplex 8A Flowsheet. One-half of the synthetic aqueous solution equilibrated with CCl_4 + 15% TBP and the other half with hydrocarbon + 15% TBP. Specimens contained in glass flask equipped with reflux condenser. Solution maintained at boiling point. Solution changed once per week.

Results:

Test Specimen		Solution, Aqueous CAW Equilibrated as Indicated (1)	Average Overall Corrosion Rate After 240 Hours (10 Days) Exposure inches/month	
Material	Type		Liquid Exposure	Vapor Exposure
Type 309 SCb	Parent Metal	CCl_4 + TBP	(2)	(2)
		HC + TBP	(2)	(2)
	Sensitized	CCl_4 + TBP	0.0001	0.0004
		HC + TBP	0.0001	0.0004
Type 304 ELC	Parent Metal	CCl_4 + TBP	(2)	(2)
		HC + TBP	(2)	(2)
	Sensitized	CCl_4 + TBP	0.0003	0.0004
		HC + TBP	0.0003	0.0004
Type 347	Parent Metal	CCl_4 + TBP	(2)	(2)
		HC + TBP	(2)	(2)
	Sensitized	CCl_4 + TBP	0.0002	0.0004
		HC + TBP	0.0002	0.0004

Note: * Data obtained by the Chemical Development group.(18)

(1) HC represents hydrocarbon. Both HC and CCl_4 contain 15% TBP

(2) Tests not initiated because of low corrosion rates obtained with sensitized metal.

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(3) A sensitized metal sample is one that has been heated to 1250°F for 1 hour and water quenched.

[redacted]

TABLE XXX

CORROSION STUDIES SLAG AND CRUCIBLE DISSOLUTION
CONDITIONS IN PRELIMINARY CHEMICAL FLOWSHEET*

Test Specimens: Types 309, 304 ELC, and 347 stainless steel, Pyrex and cobalt glass.

Each specimen exposed to both boiling liquid and condensing vapors. Metal samples both parent metal and sensitized metal.

Procedure: Each 24 hours, fresh slag and crucible from cold uranium reduction was placed in glass reaction flask with the test specimens. A standard dissolution (Los Alamos flowsheet) was carried out using ANN to complex the fluoride. After iodine removal and dissolution, the solution was refluxed at 100-120°C for 24 hours. After 24 hours the solution was removed, the samples weighed, measured and replaced in the flask with fresh slag and crucible. This cycle was repeated 18 times.

Results:

Test Specimens		Average Overall Corrosion Rate Based on 18 Batch Dissolutions inches/month	
Material	Type	Liquid Exposure	Vapor Exposure
Type 309 S.S.(1)	Parent metal	0.0009	0.0027
	sensitized (2)	0.0067	0.0207
Type 304 ELC S.S.	Parent metal	0.0009	0.0017
	sensitized	0.0009	0.0022
Type 347	Parent metal	0.0008	0.0018
	sensitized	0.0013	0.0024
Cobalt glass	----	0.0002	0.0001
Pyrex glass	----	0.0004	0.0003

Note: * Data obtained by the Chemical Development group.(18)

(1) This material was thought to be Type 309 SCB stainless steel when the tests were initiated. It was later discovered that the metal was non-stabilized.

(2) A sensitized metal sample is one that has been heated to 1250°F. for 1 hour and water quenched.

[redacted]

TABLE XXXI

CORROSION STUDIES SLAG AND CRUCIBLE DISSOLUTION

CONDITIONS IN HW NO. 1 CHEMICAL FLOWSHEET*

Test Specimens: Types 309 SCB, 304 ELC and 347 stainless steel. Each specimen exposed to both boiling liquid and condensing vapors.

Procedure: Every 24 hours fresh slag and crucible from cold uranium reduction was placed in glass reaction flask with test specimens. Dissolution (HW No. 1 flowsheet) performed with 2 hours dissolution in HNO_3 and 18-20 hours digestion with ANN added to complex fluoride ion. Solution then removed, the samples weighed, measured and replaced in flask. Cycle repeated 4 times.

Results:

Test Specimen		Average Overall Corrosion Rate Based on 4 Batch Dissolutions inches/month	
Material	Type	Liquid Exposure	Vapor Exposure
Type 309 SCB stainless steel	Parent Metal	0.0008	0.0012
	sensitized (1)	0.0008	0.0013
Type 304 ELC stainless steel	Parent Metal	0.0016	0.0037
	sensitized	0.0017	0.0035
Type 347 stainless steel	Parent Metal	0.0009	0.0027
	sensitized	0.0014	0.0022

Note: * Data obtained by the Chemical Development group.(18)

(1) A sensitized metal sample is one that has been heated to 1250°F for 1 hour and water quenched.

[redacted]

(5) Equipment Requirements and Adequacy of 234-5 Building Facilities.

The development data and recommended chemical flowsheets obtained through the combined efforts of the Chemical Research, Chemical Development, and Process Assistance (234-5 Development) groups were employed by the Process Assistance (Engineering) group to prepare the engineering flow diagrams and design the equipment required in the proposed Recuplex Process.

(a) Engineering design bases.

In order to allow for the future expansion of plant capacities, an instantaneous production rate of 100 Kg/mo. was chosen as the design basis for equipment sizing. Sufficient flexibility was incorporated, however, to permit operation at an instantaneous rate of 120 Kg/mo. with few minor equipment changes. The magnitude of the various feed, product, and waste streams which the Recuplex Process was designed to handle is presented in Figure H-2-15208 by means of a material balance of the process based upon twenty-four hours operation at an instantaneous rate of 100 Kg/mo.

(i) Tank capacity.

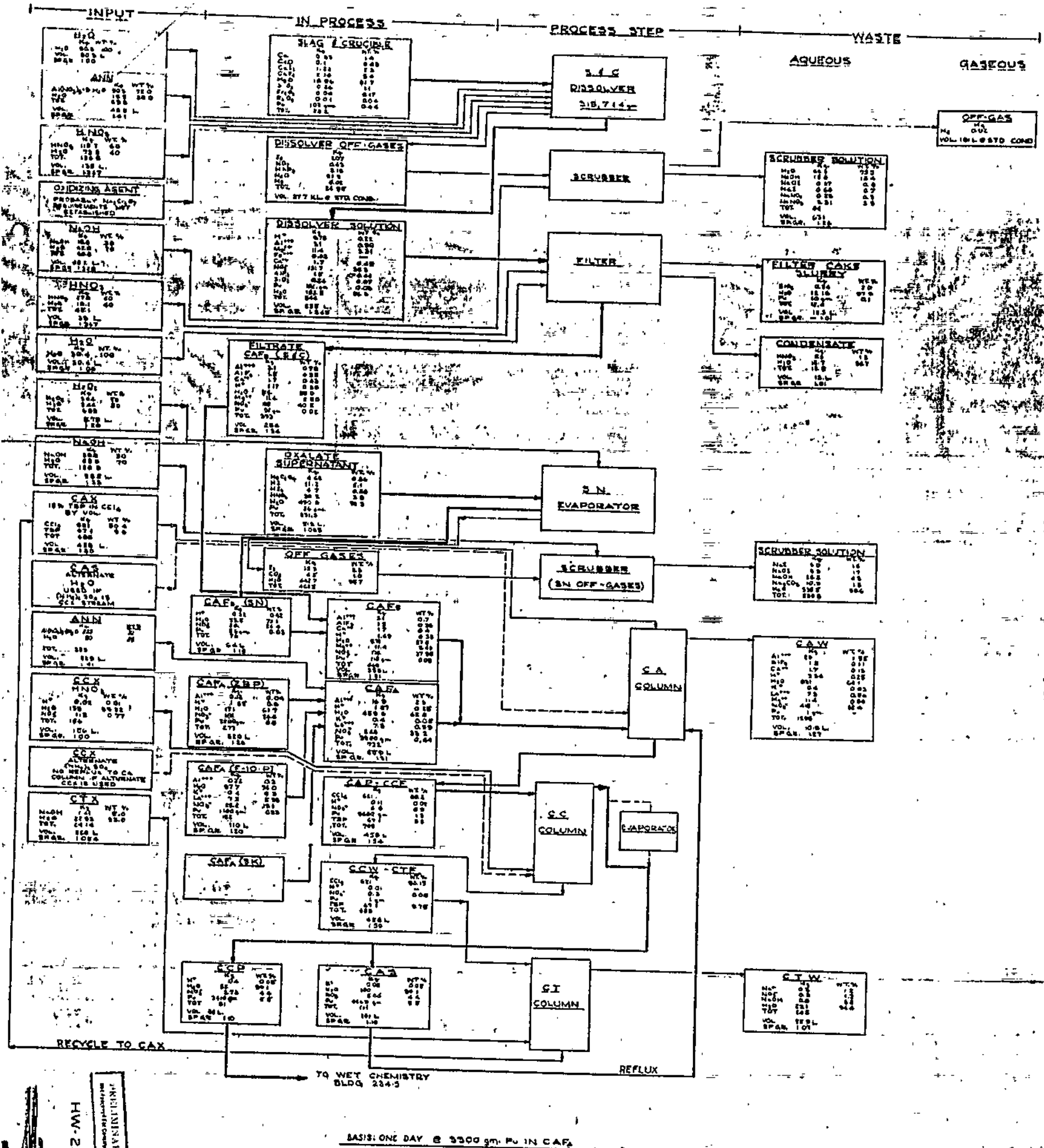
Tank sizing was based on a minimum of twelve hours hold-up and an allowance of at least a 20 per cent freeboard. Greater hold-up capacities were incorporated at potential "bottlenecks" within the system and some of the required equipment was duplicated in order to insure continuity of operation.

(ii) Critical-mass considerations.

The storage and handling of concentrated plutonium solutions necessitated special design of certain tanks to prevent the possibility of exceeding critical-mass conditions. The receiver, make-up, and feed tanks of the concentrated feed stream to the extraction tower were designed to be safe with respect to criticality by (1) employing geometries permitting an unlimited range of concentrations or plutonium content, i.e. eight inches or less in diameter and twelve inches edge-to-edge spacing, and/or (2) employing batch-size control which permits, over the plutonium concentration and average composition range expected, a total of 1000 grams of plutonium per batch.(20) The product receiver and storage tanks were made infinitely safe from the criticality viewpoint by the geometrical configuration employed. The tanks were designed to be rectangular in shape with one dimension a maximum of eight inches and the other two dimensions unlimited.(20) The tanks can be arranged within the hood with no restrictions as to their edge-to-edge distance if they are contained within two parallel planes separated by the eight-inch dimension. Plutonium-bearing vessels located outside these planes must be protected from neutron absorption by 24 inches of air or its equivalent.

(iii) Hood design.

One of the problems peculiar to the design of a process for the purification and concentration of plutonium is the need for housing the equipment in such a manner as to protect personnel from the hazards of plutonium contamination. At present, there are two processes in the Separations Area which are concerned with stream volumes and equipment sizes on approximately the same scale as that proposed in the Recuplex process. The equipment in the older



BASIS: ONE DAY @ 3500 gm. Pu IN CAF

PRELIMINARY

HW-22596

GENERAL ELECTRIC CO.
HANFORD WORKS
MATERIAL BALANCE
FLOWSHEET-RECUPLEX

PROJECT TITLE
RECUPLEX FEASIBILITY SURVEY
SEPARATIONS TECHNOLOGY-PROCESS

BLDG 234-5
DWG NO H-2-15208

of the two buildings, the 231 Concentration Building, is housed according to the so-called "Greenhouse" pattern, while that in the newer 234-5 Purification and Fabrication Building is enclosed in the Barrier-type hoods. The purpose of the Barrier-type construction is to provide an unpierced physical separation between the operating and servicing areas of the equipment. To accomplish this, three areas are constructed, each separated from the other two by solid barriers and each ventilated by a separate exhaust system. As illustrated in Figure 5, the middle area is designated as the process hood and contains a single row of processing vessels. Operation of the vessels is performed from one side of the hood through rubber-glove ports, while such services as sampling and reagent-addition are accomplished from the other side through rubber-glove ports and air-locks. Theoretically, maintenance work requiring direct access to the hood and consequent removal of hood panels is done from the servicing area, thereby, permitting the long-term contamination of the servicing-room air without affecting the operating area. The Concentration-Building Greenhouse pattern, on the other hand, differs from the Barrier-type in that servicing and operation of the equipment are not performed in isolated areas, and therefore only two ventilation systems are required. The contaminated process vessels are confined within a transparent hood (cf Figure 5), which is directly accessible from all sides for both equipment maintenance and operation. Experience in the 231 and 234-5 Buildings has demonstrated that the Greenhouse-type construction is preferable. Use of a Barrier-type hood rather than a Greenhouse results in increased costs for installation, maintenance, and operation. The fabrication of Barrier-type hoods not only involves extra materials and manpower, but also requires that the process vessels and the enclosing hoods be place in a single row. Greenhouse hoods, on the other hand, permit better utilization of floor space since there are less restrictions on the arrangement of the vessels. Practice in the 234-5 Building has shown that maintenance of some of the less accessible equipment must be performed by piercing the Barrier from the operating side of the hood. Such action requires the construction of a portable air-lock and shutdown of hood operations. In the 231 Building, however, it has been found that the sweepage of air into a hood through an opened panel is sufficient to restrict the spread of contamination and obviates the need for portable air-locks. There is a considerable amount of inconvenience involved in operating equipment housed within a Barrier-type hood, especially the walking distance between the two sides of the hood. To perform certain steps in the 234-5 operating procedure it is necessary to have two operators, one in the operating area and another in the servicing room; in some instances, this imposes severe restrictions upon communication and operating efficiency. The ready accessibility of the equipment in a Greenhouse hood, however, results in more convenient and more efficient operating procedures, requiring a fewer number of operating personnel. In view of these considerations, preliminary design of the housing for the proposed Recuplex process was based primarily upon the Greenhouse pattern. It is felt, however, that strict adherance to either the 231 or the 234-5 hood plans is not to be recommended, but that the desirable features of both should be incorporated in a new and improved hood design.

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(b) Engineering flow sketches.

Four engineering flow sketches have been prepared, each of which describes one or more phases of the Recuplex Process and represents all the equipment to be associated with an individual process hood. Briefly, the four process hoods contain: (i) the equipment for preparing slag-and-crucible solution; (ii) a revision of the present equipment for treating Wet-Chemistry supernatants; (iii) the equipment for receiving and blending of the column feed streams; and (iv) the equipment for the solvent-extraction, solvent-treatment, column-waste-treatment, and product-storage operations.

[redacted]

[redacted]

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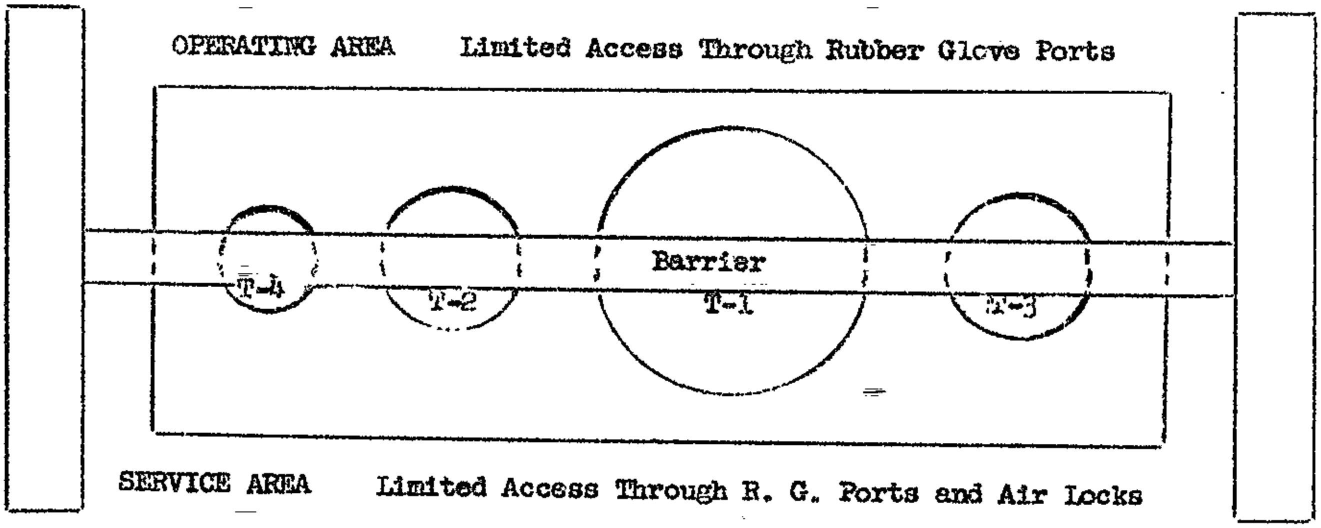
Figure No. 5

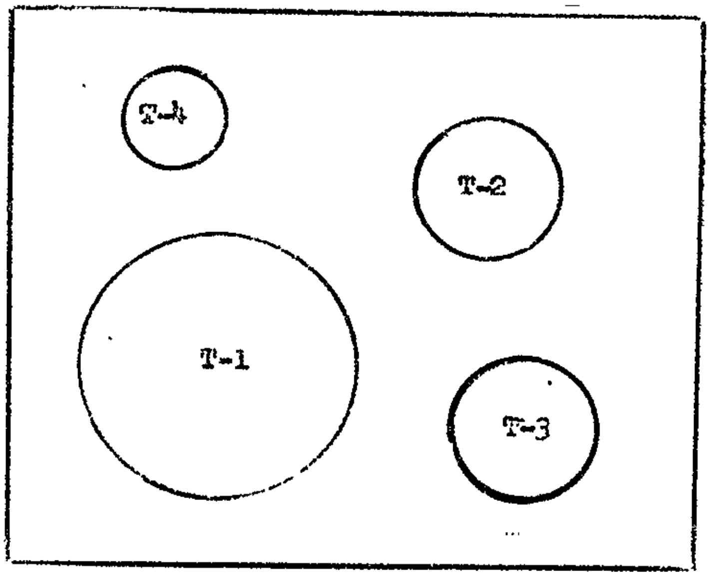
SCHEMATIC REPRESENTATION OF BARRIER AND GREENHOUSE CONSTRUCTION

BARRIER-TYPE PROCESS HOOD



Direct Access by Panel Removal

GREENHOUSE-TYPE PROCESS HOOD



OPERATING AND SERVICING AREA

Limited Access Through Rubber Glove Ports

Direct Access by Panel Removal

DECLASSIFIED

[redacted]

(i) Slag-and-crucible solution preparation.

As Figure H-2-15082 indicates, the dissolution and filtration assemblies within the hood have been duplicated to insure continuity of the batch operation. The design of the dissolution assembly is based upon similar equipment employed at the Los Alamos Scientific Laboratory with certain modifications. Corrosion studies of the dissolving procedures recommended in the Chemical Flowsheets indicate that the dissolver should be constructed of stainless steel. An intermediate heat transfer fluid will be used in the dissolver jacket to eliminate the feasibility of a jacket failure resulting in the mixing of the dissolver solution with the process cooling water or steam and being discharged to the crib. The 10-foot reflux column and the caustic scrubber arrangement are similar to the Los Alamos design except that a cooling coil has been incorporated in the scrubber pot to remove the heats of reaction and prevent excessive vapor entrainment.

The filtration assembly differs from the Los Alamos equipment and is adaptable either to direct filtration or to the use of a precoat on the filter and filter aid in the feed solution. Means have been provided for diluting the filter feed and filtering the solution at elevated temperatures in order to decrease the viscosity of the solution and prevent the formation of solid magnesium nitrate. The silica cake may be either slurried for jetting to waste storage or removed as a solid for future recovery processing. Approximately 100 gal/mo. of silica slurry would be sent to underground storage. The filtrate vacuum line is designed to contain a cold trap and deentrainment trap as a preventive measure against possible carry-over into the vacuum equipment. It is proposed that the chemical addition tanks be located immediately above, but outside the hood proper. This would reduce the need for direct entry into the hood to repair leaks, valves, etc. in the chemical feed lines.

Preliminary specifications for the equipment associated with the slag-and-crucible hood are presented in Figure H-2-15209.

(ii) Supernatant solution preparation.

A proposed revision and condensation of the present supernatant-recovery hoods is presented in Figure H-2-15079. The principal differentation from the present operation is the consolidation of the equipment presently contained in two Barrier-type hoods into one Greenhouse hood. Duplicate evaporating assemblies have been preserved; the supernatant storage capacity, however, has been enlarged and hydrogen-peroxide feed tanks and an additional hold tank for the final solution have been incorporated. The chemical tanks have been placed outside the hood proper. Preliminary specifications for the equipment associated with the supernatant-treatment hood are presented in Figure H-2-15210.

(iii) Solution reception and blending.

The reception and blending operations of the five plutonium-bearing streams to be fed to the extraction columns are incorporated into one process hood. The flow sketch in Figure H-2-15080 includes the RR Can unloading facilities; it is proposed, however, that these be enclosed within a separate hood. It is also felt that the reagent-addition tanks should be placed outside the hood and vented to room air. Although complete duplication of the processing

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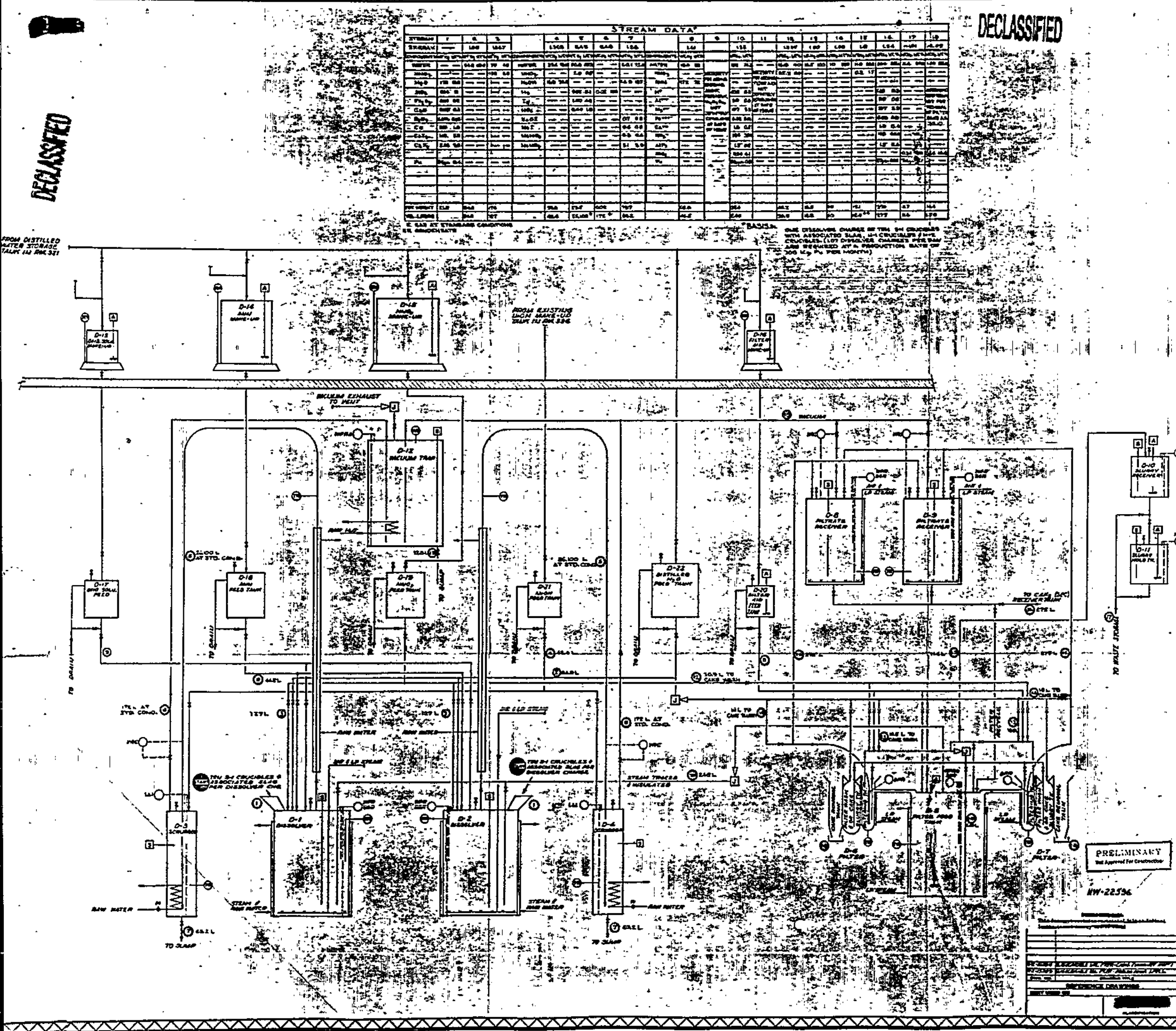

* GAS AT STANDARD CONDITIONS
** CONDENSATE

*BASIS: ONE DISSOLVER CHARGE OF TEN 34 CRUCIBLES WITH ASSOCIATED SLAG, [illegible] CRUCIBLES [illegible] CRUCIBLES. (107 DISSOLVER CHARGES PER DAY ARE REQUIRED AT A PRODUCTION RATE OF 300 Kg Pu PER MONTH)

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LEGEND

- A AGITATOR
- J JET
- S SAMPLER
- DP DIFFERENTIAL PRESS
- F FLOW
- LL LIQUID LEVEL
- SG SPECIFIC GRAVITY
- T TEMPERATURE
- V VACUUM
- W WEIGHT
- WF WEIGHT FACTOR

FOLLOWED BY I, R, RC OR RA DENOTES INDICATOR, RECORDER, RECORDING CONTROLLER, AND RECORDER ALARM, RESPECTIVELY

PRELIMINARY
Not Approved For Construction

HW-22596

REFERENCE DRAWINGS

REVISIONS

SCALE: NO SCALE

APPROVALS

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

SLAG AND CRUCIBLE
SOLUTION PREPARATION
ENGINEERING FLOW SKETCH

[illegible] FEASIBILITY [illegible]

BLDG. NO. 234-5 DWG. NO. 4-2-15032

[redacted] DECLASSIFIED

H-2-15209

SLAG AND CRUCIBLE SOLUTION PREPARATION - PRELIMINARY EQUIPMENT SPECIFICATIONS

Equipment Number	Process Vessel	Capacity	Solution Volume (Liters)	Tank Capacity (Liters)	Nominal Tank Capacity (Gallons)	Approximate Overall Dimensions (Inches)		Material Of Construction	Instrumentation										Auxiliary Equipment							Utilities					
						Diameter	Height		DPR	FI	LLI	SGR	TR	VR	VRC	WFR	WFRA	WI	Agitator	Coil	Jacket	Jet	Sampler	Sparger	Vent	Inst. Air	Bldg. Air	Raw Water	Power	Steam	Distilled Water
D-1 & D-2	Dissolver	1 Run	240	413	100	34	48	Stainless Steel				x	x			x					x	x	x	x	x	x	x	x		x	
D-3 & D-4	Scrubber	1 Run	63	120	30	14	48	Carbon Steel		x	x		x		x					x			x			x		x			
D-5	Filter Feed Tank	1 Run	240	300	80	28	32	Stainless Steel				x	x				x		x		x	x			x	x	x	x	x	x	
D-6 & D-7	Filter	1 Run	—	—	—	20 x 25	36	Stainless Steel	x				x						x		x					x	x	x	x	x	
D-8 & D-9	Filtrate Receiver Tank	1 Run	275	341	90	30	32	Stainless Steel				x	x		x	x							x	x	x	x	x			x	
D-10	Slurry Receiver Tank	1 Run	11.6	16	5	13	12	Stainless Steel				x				x			x				x		x	x			x		
D-11	Slurry Hold Tank	1 Run	11.6	16	5	13	12	Stainless Steel								x			x				x		x	x			x		
D-12	Vacuum Trap	—	—	790	200	36	48	Stainless Steel						x			x			x		x	x		x	x		x			
D-13	Oxidant Make-Up Tank	2 Runs	(Not Determined)	38	10	15	15	Stainless Steel										x	x										x		x
D-14	ANN Solution Make-Up Tank	2 Runs	93	116	30	20	25	Stainless Steel										x	x										x		x
D-15	HNO_3 Make-Up Tank	2 Runs	316	380	100	31	30	Stainless Steel										x	x										x		x
D-16	Filter Aid Make-Up Tank	2 Runs	25	38	10	15	15	Carbon Steel										x	x										x		x
D-17	Oxidant Feed Tank	1 Run	(Not Determined)	16	5	13	12	Saran																	x						
D-18	ANN Solution Feed Tank	1 Run	46.5	57	15	16	20	Lucite																	x						
D-19	HNO_3 Feed Tank	1/3 Run	42	57	15	16	20	Saran																	x						
D-20	Filter Aid Feed Tank	1 Run	13	16	5	13	12	Lucite											x						x						
D-21	NaOH Feed Tank	1 Run	42.4	57	15	16	20	Lucite																	x						
D-22	Distilled Water Feed Tank	1 Run	103	125	35	23	24	Lucite														x			x						x

LEGEND

DPR	Differential Pressure Recorder	VR	Vacuum Recorder
FI	Flow Indicator	VRC	Vacuum Recording Controller
LLI	Liquid Level Indicator	WFR	Weight Factor Recorder
SGR	Specific Gravity Recorder	WFRA	Weight Factor Recorder Alarm
TR	Temperature Recorder	WI	Weight Indicator

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[redacted]

[redacted]

-66-
HW-22596

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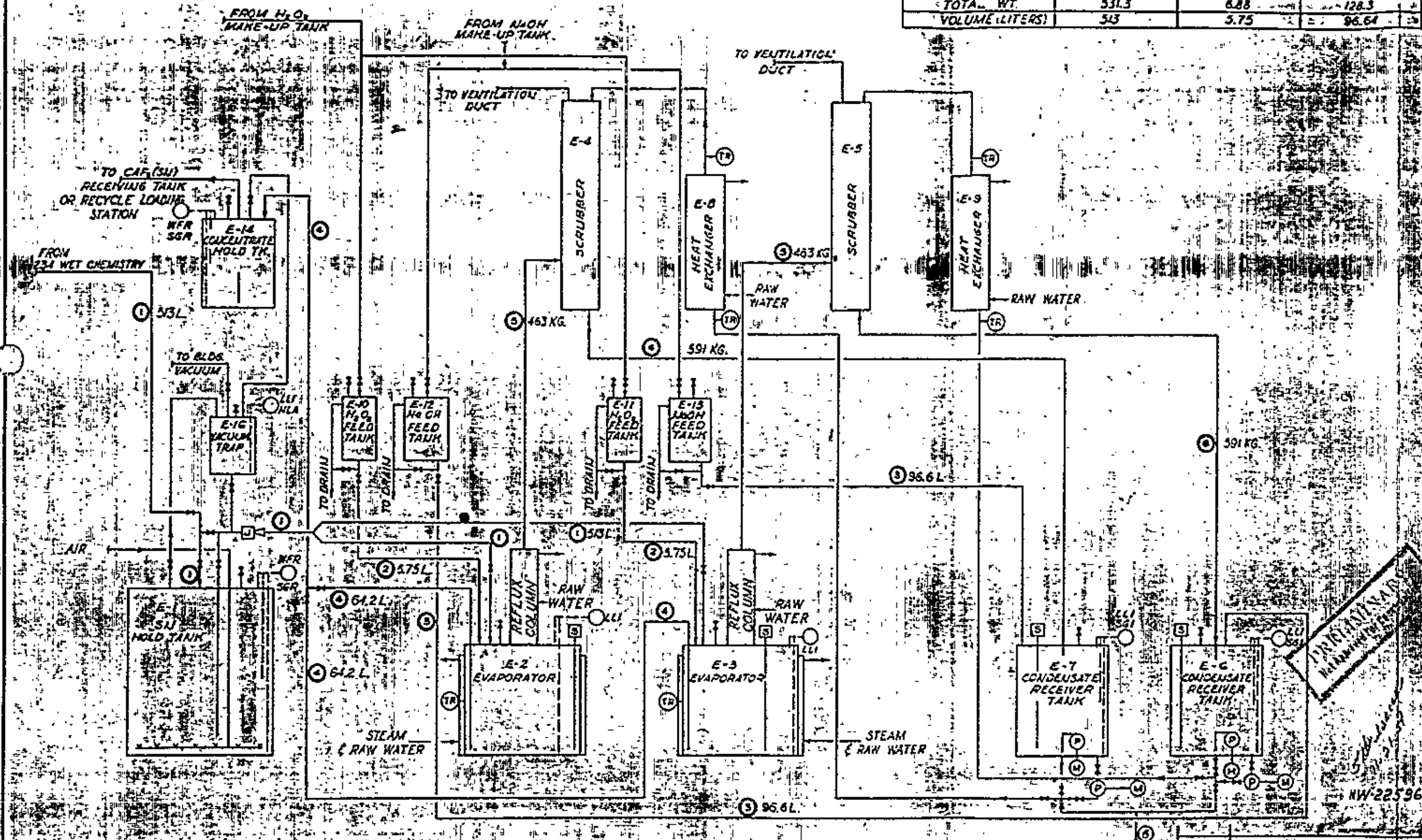
STREAM	①		②		③		④		⑤		⑥	
SP GRAVITY	1.04		1.20		1.33		1.18		—		—	
COMPONENTS	KG.	WT.%	KG.	WT.%	KG.	WT.%	KG.	WT.%	KG.	WT.%	KG.	WT.%
CO_2	—	—	—	—	—	—	—	—	4.52	1.02	—	—
$H_2C_2O_4$	4.62	0.87	—	—	—	—	—	—	—	—	—	—
HI	11.16	2.10	—	—	—	—	—	—	—	—	—	—
HI_3	4.70	0.86	—	—	—	—	—	—	—	—	—	—
HNO_3	20.21	3.80	—	—	—	—	20.2	26.7	—	—	—	—
H_2O_2	—	—	3.44	50	—	—	—	—	—	—	—	—
I_2	—	—	—	—	—	—	—	—	15.26	3.45	—	—
Na_2CO_3	—	—	—	—	—	—	—	—	—	—	10.88	1.84
NaI	—	—	—	—	—	—	—	—	—	—	9.01	1.52
NaOH	—	—	—	—	38.5	30	—	—	—	—	25.48	4.31
NaOI	—	—	—	—	—	—	—	—	—	—	9.97	1.69
Pu (GRAMS)	(26)	—	—	—	—	—	(26)	—	—	—	—	—
WATER	490.6	92.3	3.44	50	89.8	70	53.48	73.3	442.7	95.7	535.6	90.6
TOTAL WT.	531.3		6.88		128.3		73.72		462.5		590.9	
VOLUME (LITERS)	513		5.75		96.64		64.17		—		—	

*BASIS — ONE DAY



LEGEND

A - AGITATOR
J - JET
M - MOTOR
P - PUMP
S - SAMPLER
HLA - HIGH LEVEL ALARM
LLI - LIQUID LEVEL INDICATOR
SGI - SPECIFIC GRAVITY INDICATOR
SGR - SPECIFIC GRAVITY RECORDER
TR - TEMPERATURE RECORDER
WFR - WEIGHT FACTOR RECORDER

NOTE

THIS DRAWING SUPERSEDES DRAWING OF THE SAME NUMBER DATED 10-19

PRELIMINARY

HW-22596

DECLASSIFIED

REVISIONS

SCALE: NO SCALE
DRAWN J. H. DATE 11-1-51
CHECKED DATE 11-1-51
ENGINEER REQUEST NO. 234-5-4768
PROJECT NO. [redacted]

APPROVALS

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

OXALATE SUPERNATANT TREATMENT
ENGINEERING FLOW SKETCH

PROJECT TITLE RECUPLEX FEASIBILITY SURVEY SEPARATIONS TECHNOLOGY - PROCESS

BLDG. NO. 234-5 DWG. NO. H-2-15079 SHEET NO. 1 1

DWG. NO.	DRAWING TITLE
H-2-15210	OXALATE SUPERNATANT TREATMENT - PRELIMINARY EQUIP. SPECS.

REFERENCE DRAWINGS

NEXT USED ON

[redacted] DECLASSIFIED

H-2-15210

OXALATE SUPERNATANT TREATMENT - PRELIMINARY EQUIPMENT SPECIFICATIONS

Equipment Number	Process Vessel	Capacity (Lots)	Solution Volume (Liters)	Tank Capacity (Liters)	Nominal Tank Capacity (Gallons)	Approximate Overall Dimensions (Inches) Diameter	Height	Materials Of Construction	Instrumentation HLA	LLI	SGI	SGR	TR	WFR	Auxiliary Equipment Agitator	Coil	Jacket	Jet	Pump	Sampler	Sparger	Vent	Utilities Inst. Air	Raw H2O	Power	Steam
E-1	SAST Storage Tank	3	200	757.1	200	50	66	Stainless Steel				x		x				x			x	x	x			
E-2 & E-3	Supernate Evaporator	1	200	283.9	75	40	44	Carbon Steel Glass Lined		x			x			x	x			x		x		x		x
E-4 & E-5	Scrubber	1	—	—	—	11	107	Carbon Steel																		
E-6 & E-7	Condensate Receiver Tank	1	200	355.8	94	30	42	Stainless Steel		x	x								x	x		x			x	
E-8 & E-9	Heat Exchanger	1	—	—	—	10	47	Carbon Steel					xx											x		
E-10 & E-11	H_2O_2 Addition Tank	1	2.4	3.0	0.8	5.5	18	Lucite														x				
E-12 & E-13	NaOH Addition Tank	1	37.7	50	13	12	27	Lucite														x				
E-14	Concentrate Hold Tank	7	25	190	50	24	27	Stainless Steel				x		x								x				
E-15	Recycle Hold Tank	2	25	50	13	15	18	Stainless Steel				x		x								x				
E-16	Vacuum Trap	—	—	22.7	6	12	16	Stainless Steel	x													x				

LEGEND

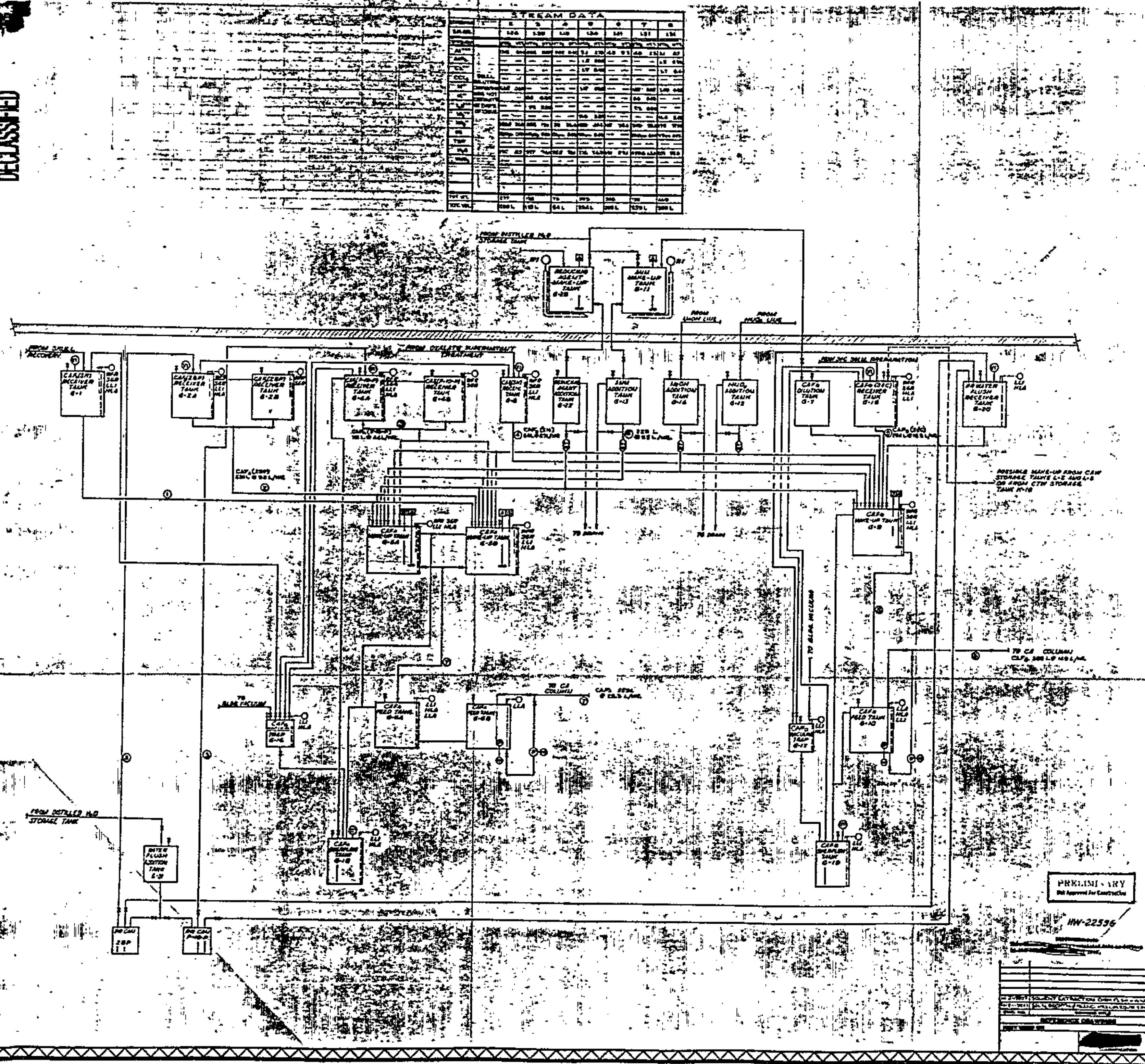
HLA	High Level Alarm
LLI	Liquid Level Indicator
SGI	Specific Gravity Indicator
SGR	Specific Gravity Recorder
TR	Temperature Recorder
WFR	Weight Factor Recorder

[redacted]

DECLASSIFIED

11-29-61

DECLASSIFIED



LEGEND

HLA	HIGH LEVEL ALARM
LLA	LOW LEVEL ALARM
LLI	LIQUID LEVEL INDICATOR
SGR	SPECIFIC GRAVITY RECORDER
VI	VACUUM INDICATOR
WFR	WEIGHT FACTOR RECORDER
WI	WEIGHT INDICATOR
Ⓐ	AGITATOR
Ⓜ	MOTOR
Ⓟ	PUMP
Ⓢ	SAMPLER
⟨ST⟩	STRAINER

DECLASSIFIED

PRELIMINARY
Not Approved for Construction

HW-22596

REFERENCE DRAWINGS

[redacted]

SCALE: 1/10 SCALE

APPROVALS

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

SOLUTION RECEPTION
& BLENDING
ENGINEERING FLOW SKETCH

RECUPLEX FEASIBILITY SURVEY

234 5 H-2-15080

[redacted]

DECLASSIFIED

H-2-15211

SOLUTION RECEPTION AND BLENDING - PRELIMINARY EQUIPMENT SPECIFICATIONS

Equipment Number	Process Vessel	Capacity (Hours)	Solution Volume (Liters)	Tank Capacity (Liters)	Nominal Tank Capacity (Gallons)	Approximate Overall Dimensions (Inches) Diameter	Height	Material Of Construction	Instrumentation HLA	LLA	LLI	SGR	VI	WI	WFR	Auxiliary Equipment Agitator	Motor	Pump	Sampler	Strainer	Vent	Utilities Inst. Air	Power	Distilled Water
G-1	CAF$_A$ (SX) Receiver Tank	48	18.4	23	6.1	8	27	Stainless Steel	x		x	x	x		x						x	x		
G-2A	CAF$_A$ (2EP) Receiver Tank	9	82.5	103	27.2	19	25	Stainless Steel	x		x	x	x		x						x	x		
G-2B	CAF$_A$ (2EP) Receiver Tank	9	82.5	103	27.2	19	25	Stainless Steel				x			x						x	x		
G-4A	CAF$_A$ (F-10-P) Receiver Tank	18	82.5	103	27.2	19	25	Stainless Steel	x		x	x	x		x						x	x		
G-4B	CAF$_A$ (F-10-P) Receiver Tank	18	82.5	103	27.2	19	25	Stainless Steel				x			x						x	x		
G-5A	CAF$_A$ Make-Up Tank	6	139.8	175	46.3	22	30	Stainless Steel	x		x	x			x	x			x		x	x	x	
G-5B	CAF$_A$ Make-Up Tank	6	139.8	175	46.3	22	30	Stainless Steel	x		x	x			x	x			x		x	x	x	
G-6A	CAF$_A$ Feed Tank	6	139.8	175	46.3	22	30	Stainless Steel	x	x	x										x	x		
G-6B	CAF$_A$ Feed Tank	6	139.8	175	46.3	22	30	Stainless Steel		x							xx	xx			x	x	x	
G-7	CAF$_B$ Dilution Tank	—	—	63	16.6	16	20	Lucite													x			x
G-8	CAF$_B$ (SW) Receiver Tank	31	82.5	103	27.2	19	25	Stainless Steel	x		x	x	x		x						x	x		
G-9	CAF$_B$ Make-Up Tank	12	179.0	224	59.2	24	32	Stainless Steel	x		x	x			x	x			x		x	x	x	
G-10	CAF$_B$ Feed Tank	18	267.0	333	88	29	36	Stainless Steel	x	x	x						xx	xx			x	x	x	
G-11	ANN Make-Up Tank	12	114.5	143.1	37.9	22	26	Stainless Steel						x		x							x	x
G-12	HNO$_3$ Addition Tank	—	—	7.6	2	9	10	Saran												x	x			
G-13	ANN Addition Tank	2	76.3	103	27.2	19	25	Lucite												x	x			
G-14	NaOH Addition Tank	—	—	7.6	2	9	10	Lucite												x	x			
G-15	CAF$_B$ (S. & C.) Receiver Tank	12	147.0	188	50	23	30	Stainless Steel	x		x	x	x		x						x	x		
G-16	CAF$_A$ Vacuum Trap	—	—	125	33	21	27	Stainless Steel	x		x										x	x		
G-17	CAF$_B$ Vacuum Trap	—	—	125	33	21	27	Stainless Steel	x		x										x	x		
G-18	CAF$_A$ Overflow Tank	—	—	125	33	21	27	Stainless Steel	x		x		x								x	x		
G-19	CAF$_B$ Overflow Tank	—	—	125	33	21	27	Stainless Steel	x		x		x								x	x		
G-20	PR Water Flush Receiver Tank	—	—	103	27.2	19	25	Stainless Steel	x		x		x								x	x		
G-21	Water Flush Addition Tank	—	—	103	27.2	19	25	Lucite													x			x
G-22	Reducing Agent Addition Tank	—	—	76	2	9	10	Lucite												x	x			
G-23	Reducing Agent Make-Up Tank	—	—	48.8	13	15	17	Stainless Steel						x		x							x	x

LEGEND

HLA	High Level Alarm
LLA	Low Level Alarm
LLI	Liquid Level Indicator
SGR	Specific Gravity Recorder
VI	Vacuum Indicator
WFR	Weight Factor Recorder
WI	Weight Indicator

DECLASSIFIED

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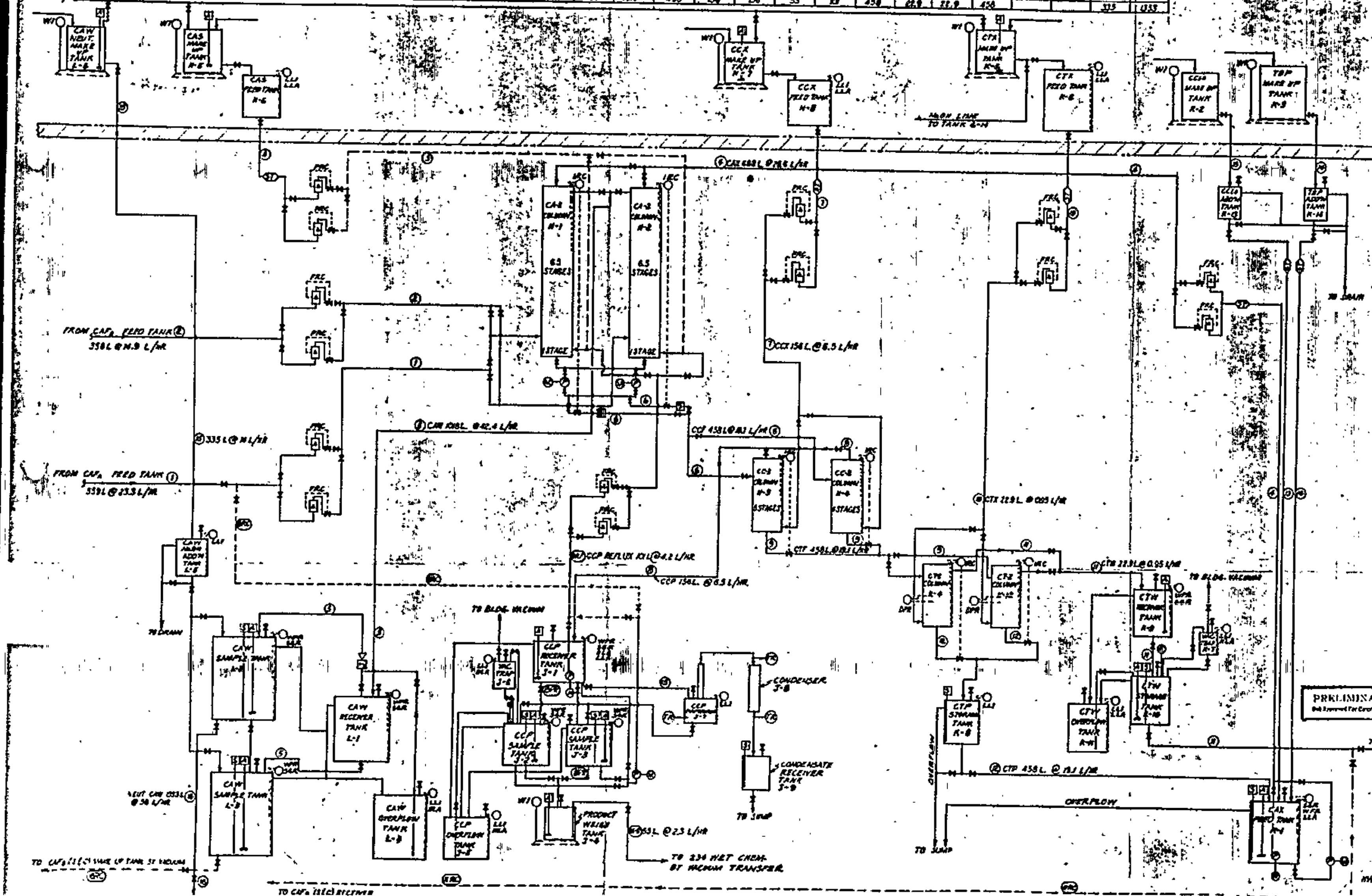
HW-225.

STREAM DATA *

STREAM NO.	①	②	③	④	⑤	⑥	⑦	⑧	⑧a	⑧b	⑨	⑩	⑪	⑫	⑬	⑭	⑮	⑯
SP. GR.	1.31	1.31		1.50	1.27	1.54	1.00	1.10	1.10	1.10	1.50	1.05	1.07	1.50			1.33	1.29
COMPONENT	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %	WT. M WT. %
Al+++	48.9 2.3	3.1 0.7			20 1.55													20 1.1
HF		1.5 0.33			1.5 0.11													1.5 0.13
Ca++		1.7 0.4	ALTERNATE STREAM TO BE USED IF H₂O IS USED AS THE STREAM		1.7 0.13													1.7 0.15
CCl4				688 90.6		[illegible] 66.1					[illegible] 90.5			621 90.4	WEIGHTS OF ⑬ & ⑭ STREAMS INDEFINITE			
H+	2.87 0.25	1.49 0.33			5.34 0.75	0.8 0.02	0.08 0.01	0.8 0.05	0.04 0.05	0.08 0.05	0.07 —							
K+	0.4 0.05				0.4 0.03													0.4 0.02
La+++	7.3 0.99				7.3 0.56													7.3 0.4
Mg++		10.8 2.43			10.8 0.88													10.8 0.8
NO3-	243 33.2	178 31.85			[illegible] 32.40	4.8 0.9	12 0.77	17.3 4.4	2.73 4.4	3.00 4.4	0.3 0.05		0.3 1.2					[illegible] 24.0
Pu	[illegible] 0.44	[illegible] 0.03			[illegible] —	range 1.3		range 5.5	range 5.5	range 5.5	[illegible] —			[illegible] —				[illegible] —
TBP				[illegible] 9.4		41.7 9.6					41.7 9.5			41.7 9.6				
H2O	608.6 82.0	275 39.8			[illegible] 64.0		138 99.2	155 90.1	51 90.1	100 90.1		22.9 95.00	23.1 94.4				312 70.0	[illegible] 69.2
HNO3																		
NaOH												1.2 3.00	0.8 3.2				133.8 30.0	
Na+													0.3 1.3					76.8 4.4
WT.	732 KG.	448 KG.		688 KG.	[illegible] KG.	705 KG.	156 KG.	172 KG.	61 KG.	111 KG.	689 KG.	24.1 KG.	24.3 KG.	688 KG.			446 KG.	1741 KG.
VOL. (LITERS)	559	358		458	1018	458	156	156	53	101	458	22.9	22.9	458			335	1335

*BASIS: ONE DAY AT 3300 g Pu IN CAF STREAM.



LEGEND
A-AGITATOR
J-JET
M-MOTOR
P-PUMP
S-SAMPLER
ST-STRAINER
DPR-DIFFERENTIAL PRESSURE RECORDER
FRC-FLOW RECORDING CONTROLLER
IRC-INTERFACE RECORDING CONTROLLER
LLI-LIQUID LEVEL INDICATOR
SGR-SPECIFIC GRAVITY RECORDER
VI-VACUUM INDICATOR
WI-WEIGHT INDICATOR
WFR-WEIGHT FACTOR RECORDER
HLA-HIGH LEVEL ALARM
LLA-LOW LEVEL ALARM
TR-TEMPERATURE RECORDER

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PRELIMINARY
Not Approved For Construction

REVISIONS

SCALE NONE

APPROVALS

234-5-4788

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

SOLVENT EXTRACTION, SOLVENT TREATMENT, WASTE TREATMENT & PRODUCT STORAGE ENGR. FLOW SKETCH

RECUPLEX FEASIBILITY SURVEY
SEPARATIONS TECHNOLOGY - PROCESS

BLDG. NO. 234-5 DWG. NO. H-2-15081

REFERENCE DRAWINGS
SOLVENT EXTRACTION, SOLVENT TREATMENT UNIT
PRODUCT STORAGE PRELIMINARY EQUIP. LIST
SOLVENT EXTRACTION CHEMICAL FLOWSHEET

[redacted]

DECLASSIFIED

H-2-15212

SOLVENT EXTRACTION, SOLVENT TREATMENT, WASTE TREATMENT, AND PRODUCT STORAGE - PRELIMINARY EQUIPMENT SPECIFICATIONS

Equipment Number	Process Vessel	Capacity (Hours)	Solution Volume (Liters)	Tank Capacity (Liters)	Nominal Tank Capacity (Gallons)	Approximate Overall Dimensions		Materials Of Construction	Instrumentation											Auxiliary Equipment									Utilities		
						Diameter	Height		DPR	FRC	HLA	IRC	LLA	LLI	SGR	TR	VI	WFR	WI	Agitator	Coil	Jacket	Jet	Motor	Pump	Sampler	Strainer	Vent	Inst. Air	Power	Distilled Water
H-1	CA1 Column	—				6"	29'	Stainless Steel		xxxx		x																	x		
H-2	CA2 Column	—				6"	29'	Stainless Steel		xxxx		x																	x		
H-3	CC1 Column	—				5"	29'	Stainless Steel		x		x																	x		
H-4	CC2 Column	—				5"	29'	Stainless Steel		x		x																	x		
H-5	CAS Make-Up Tank	12			17.2	16"	20"	Stainless Steel											x	x										x	x
H-6	CAS Feed Tank	24			34.4	20"	25"	Stainless Steel					x	x													x		x		
H-7	CCX Make-Up Tank	12	78	97.5	26.	17"	20"	Stainless Steel											x	x										x	
H-8	CCX Feed Tank	24	156	195.	51.5	20"	34"	Stainless Steel					x	x													x		x		x
J-1	CCP Receiver Tank	15.5	100	120	31.6	8" x 26" x	35"	Stainless Steel			x		x		x			x		x				x	x			x	x	x	
J-2	CCP Sample Tank	24	55	68.8	18.2	8" x 23" x	34"	Stainless Steel							x		x	x		x						x		x	x	x	
J-3	CCP Sample Tank	24	55	68.8	18.2	8" x 23" x	34"	Stainless Steel							x			x		x				x	x	x		x	x	x	
J-4	CCP Weigh Tank	—	—	10.5		8" x 8" x	10"	Stainless Steel											x	x								x		x	
J-5	CCP Overflow Tank	—	—	19.2		8" x 12" x	12"	Stainless Steel			x			x														x	x		
J-6	CCP Vacuum Trap	—	—	10.5		8" x 8" x	10"	Stainless Steel			x			x														x	x		
J-7	CCP Evaporator	—	—	—		10"	16"	Stainless Steel								x					x	x						x			
J-8	CCP Condenser	—	—	—		4"	24"	Stainless Steel								xx															
J-9	CCP Condensate Receiver	—	—	14.4	4	10"	12"	Lucite																				x			
K-1	CAX Feed Tank	12	229	286	75	29"	36"	Stainless Steel		xx			x		x			x		x				xx	xx	x			x	x	
K-2	CCl_4 Make-Up Tank	12	146.2	182.6	48	23"	28"	Stainless Steel											x												
K-3	TBP Make-Up Tank	12	25.8	32.2	9	13"	16"	Stainless Steel											x												
K-4	CT1 Column	—	—	—	—	10"	40"	Stainless Steel	x	x		x																	x		
K-5	CTX Make-Up Tank	12	11.5	14.4	4	10"	12"	Stainless Steel											x	x										x	x
K-6	CTX Feed Tank	24	23.0	28.7	8	12"	17"	Stainless Steel					x	x															x		
K-7	CTP Vacuum Trap	—	—	7.6	2	9"	10"	Stainless Steel			x			x														x	x		
K-8	CTP Storage Tank	12	229	286	75	29"	36"	Stainless Steel						x												x		x	x		
K-9	CTW Receiver Tank	12	11.5	14.4	4	10"	12"	Stainless Steel							x			x		x								x	x	x	
K-10	CTW Storage Tank	12	23.0	28.7	8	12"	17"	Stainless Steel																				x			
K-11	CTW Overflow Tank	—	—	108	28.4	19"	23"	Stainless Steel			x			x						x						x		x	x	x	
K-12	CT2 Column	—	—	—	—	10"	40"	Stainless Steel	x	x		x																	x		
K-13	CCl_4 Addition Tank	—	—	—	2	9"	10"	Lucite																			x	x			
K-14	TBP Addition Tank	—	—	—	2	9"	10"	Lucite																			x	x			
L-1	CAW Receiver Tank	12	509	565	149	33"	41"	Stainless Steel							x			x					x					x	x		
L-2	CAW Treatment Tank	12	1018	1135	300	42"	52"	Stainless Steel							x			x		x			x			x		x	x	x	
L-3	CAW Treatment Tank	12	1018	1135	300	42"	52"	Stainless Steel							x			x		x			x			x		x	x	x	
L-4	CAW Overflow Tank	—	509	565	149	33"	41"	Stainless Steel			x			x									x					x	x		
L-5	CAW NaOH Addition Tank	—	—	—	50	25"	28"	Lucite						x														x	x		
L-6	CAW Neutralized Make-Up Tank	—	—	—	100	31"	39"	Stainless Steel										x											x		x

LEGEND

DPR	Differential Pressure Recorder	SGR	Specific Gravity Recorder
FRC	Flow Recording Controller	TR	Temperature Recorder
HLA	High Level Alarm	VI	Vacuum Indicator
IRC	Interface Recording Controller	WFR	Weight Factor Recorder
LLA	Low Level Alarm	WI	Weight Indicator
LLI	Liquid Level Indicator		

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vessels was not attempted, key equipment pieces were duplicated and such equipment as spare pumps, overflow tanks, and vacuum traps were included to insure maximum operating time without shutdown. Safe criticality conditions were maintained in the sizing of the vessels to contain concentrated feed. Figure H-2-15211 presents the preliminary specifications for the reception-and-blending equipment.

(iv) Solvent extraction operations.

The equipment indicated in Figure H-2-15081 for the solvent-extraction, solvent-treatment, waste-treatment, and product-storage steps of the Recuplex Process is simple enough to permit enclosure within one hood. The extraction-and-scrubbing column is three inches in diameter and the stripping column two inches in diameter. The number of theoretical stages required to perform the extraction, scrubbing, and stripping operations are 6.5, 1, and 5, with corresponding HETS values of approximately 3, 3, and 4 feet. Since the recommended column heights, including end sections and appropriate safety margins, are each twenty-nine feet, the columns must extend above the main body of the hood in a special shroud. The dimensions of the solvent-treatment tower, given in the preliminary equipment specifications of Figure H-2-15212, are approximate; more firm values have not as yet been determined. Proper disposition of the aqueous CTW stream has yet to be determined. Although the plutonium loss to this stream is extremely low, it is believed that a suitable recovery procedure may be developed. The aqueous waste stream from the CA column requires special underground piping facilities and storage. The annual volume of the gelatinous, neutralized CAW solution is estimated to be approximately 10,000 gallons. An evaporator is included downstream of the CCP receiver tank to be employed in the event that the dilute slag-and-crucible solution would be the only feed stream to the extraction column. The product receiver, evaporator, and storage tanks were designed to be infinitely safe with regard to criticality conditions.

(c) Equipment arrangement studies.

There is presently available within the 234-5 Building unoccupied floor space which is suitable for additional chemical make-up facilities (Room 337) and a limited amount of room suitable for process operations (Room 221). One of the prime factors in the Recuplex feasibility study is the containment of the requisite process equipment within these two rooms, since the construction of a new and separate building would diminish greatly the advantages to be gained by the process. Equipment arrangement studies based upon the preceding engineering flow sketches have indicated that this can be accomplished satisfactorily. Sketches have been prepared to indicate plan and elevation view of the most suitable arrangements of the process vessels and the accompanying instrumentation.

(i) Chemical make-up facilities.

Room 337 in the 234-5 Building can more than adequately accommodate the reagent-preparation vessels required in the Recuplex Process. As Figure H-2-15083 indicates, the installation of the proposed Recuplex equipment would still permit expansion of the present make-up room in the event of other plant requirements.

(ii) Present floor plan of Room 221.

The present floor plan of Room 221, presented in Figure H-2-15075, demonstrates the location of the supernatant-recovery hoods (numbers 29, 30, 31, and 32) the skull-recovery hood (number 40) and the space available for the Recuplex processing equipment. Each of the present hoods follows the Barrier-type construction pattern.

(iii) Recuplex equipment arrangement No.1.

A feasible arrangement of the Recuplex process equipment within Room 221 is presented in Figure H-2-15076. Replacement of the present Barrier-type supernatant hoods with Greenhouse construction would enlarge the available operating area in Room 221 and provide a satisfactory solution to the Recuplex special requirements. It is proposed that the reception-and-blending hood and the hood for the solvent-extraction operations both be of the Greenhouse-type construction, the latter having a special shroud extending through the ceiling to house the pulse columns. The location of the barrier for Hood No. 40 and the contamination problems associated with handling the slag-and-crucible material and periodically replacing the dissolver pot, suggested the extension of the barrier for Hood No. 40 and the construction of the S-&-C hood according the Barrier pattern. For purposes of convenience the PR Can unloading station was also included in the Barrier pattern, although it is not believed to be necessary. Elevation view of the equipment and the instrument panels are presented in Figure H-2-15078, sheets 1 and 2.

(iv) Recuplex equipment arrangement No.2.

A second arrangement of the Recuplex process equipment within Room 221 is given in Figure H-2-15077. The individual hoods are essentiall the same as in Figure H-2-15076, with the exception of the supernatant-recovery facilities. The Barrier-type process hoods can be retained in the room and the Recuplex facilities installed in the remaining area. The use of the present-supernatant-recovery facilities in the Recuplex Process, however, presents the alternatives of either expanding Room 221 to include a portion of the adjacent locker rooms or appreciably reducing the working space between the hoods. Elevation views of the equipment and the instrument panels in the second arrangement are presented in Figure H-2-15078, sheets 1 and 2.

Of the two equipment-arrangement plans presented in Figures H-2-15076 and H-2-15077, the former permits the more efficient utilization of the available floor-space. The replacement of the present Barrier-type recovery hoods with one revised and consolidated Greenhouse hood releases the present servicing area for use as a combined servicing and operating area. The resulting arrangement will provide a greater facility and efficiency in the operation and maintenance of the processing equipment. The proposed substitution of sulfur in the place of hydriodic acid prior to the Wet-Chemistry reduction step will permit the Wet-Chemistry supernatants to be recycled directly to the 224 Building (without a preliminary reduction in volume) during the construction of the new facilities. On the basis of these considerations, the equipment-arrangement plan of Figure H-2-15076 is recommended as the more suitable and advantageous for the Recuplex Process.

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Preliminary estimates indicate that the cost of installation would be approximately $1,300,000. It is estimated that the preparation of a detailed Project Proposal will require 8 to 12 man-months of engineering effort. In the event the project is approved, the completion date for equipment fabrication and installation will be primarily dependent upon material procurement.

[redacted]

VI. FUTURE WORK.

The Recuplex program has reached the stage of development wherein the preparation of a detailed Project Proposal is indicated. It is estimated that this will require 8 to 12 man-months of engineering effort. Although the feasibility of the Process has been established, certain phases of the laboratory, semi-works, and engineering studies have not as yet been completed. Some of these data should be obtained on a plant-scale basis in the installed process equipment, as for example, the optimum operating conditions (temperature dissolving times, etc.) for dissolution of the slag-and-crucible residues and the stability and operating characteristics of the solvent extraction columns under conditions of varying feed stream quantities and compositions. The following work should be performed concurrently with the preparation of a firm Project Proposal and a detailed engineering design:

A. Column Feed Preparation.

(1) Slag-and-Crucible Dissolution.

The three approaches to the corrosion problem associated with the one-step, total-plutonium-dissolution procedure must be evaluated: (1) adjustment of the chemistry to provide satisfactory dissolution in a non-corrosive environment; (2) development of special materials of construction for increased resistivity to chemical attack under dissolving conditions; and (3) periodic replacement of the dissolver. Preliminary data indicate that the corrosion rates with presently available stainless steels would make dissolver replacement economically sound. In the event the Los Alamos dissolution procedure is used, the most advantageous method of processing the silica cake must be determined.

(2) F-10-P Clarification.

The presence of oil emulsions and solid contaminants in the F-10-P solution seriously affects column operation by increasing the disengaging time of the two phases. The solution must consequently be clarified prior to its blending in the CA-column feed stream. This can be accomplished either by washing with carbon tetrachloride or by a filtration operation similar to that presently employed in the 231 Building. The two methods must be evaluated and the better procedure incorporated into the Recuplex system. Preliminary data indicate that the carbon-tetrachloride wash will provide an effective treatment.

B. Solvent Extraction.

Extraction coefficients and column operating characteristics should be determined for the individual 2BP, F-10-P, and S-&-C streams to provide the necessary data to insure Recuplex operability over the full range of feed-stream quantity and composition. Studies pertaining to the purity of the CAP stream and empirical diffusion rates in the dilute extraction end of the CA column and in the CC column should be completed.

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C. Waste Disposal.

Although the waste loss to the organic phase is only 0.03 per cent, the periodic treatment of the solvent with a 5 per cent caustic wash results in

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an aqueous solution having a volume of approximately 17 liters and a plutonium concentration ca 4 g/1. It is believed that an effective procedure can be developed for the recovery of the plutonium.

D. Design and Equipment-Arrangement Studies.

Although the equipment proposed for the Recuplex Process is capable of accommodating varying feed streams, proper adjustments will have to be made in the chemical flowsheets and engineering flow sketches to compensate for future process changes in the Bismuth Phosphate, Redox, and Purification and Fabrication operations. It will be necessary to maintain close coordination between future development investigations and the equipment-arrangement studies to insure the containment of the Recuplex equipment in the space available in the 234-5 Building.

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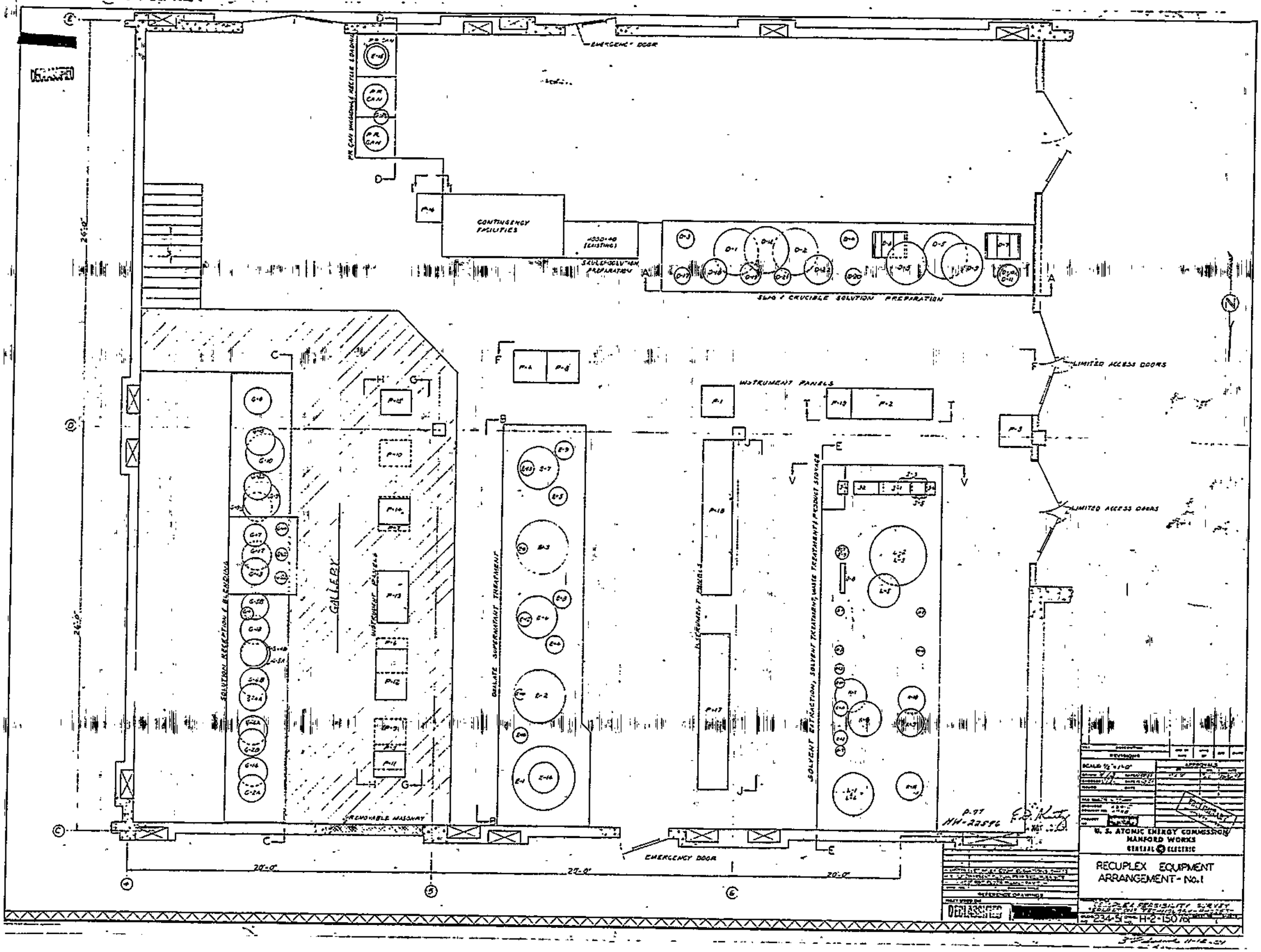

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HW-22596

SCALE 1/8" = 1'-0"

REFERENCE DRAWINGS

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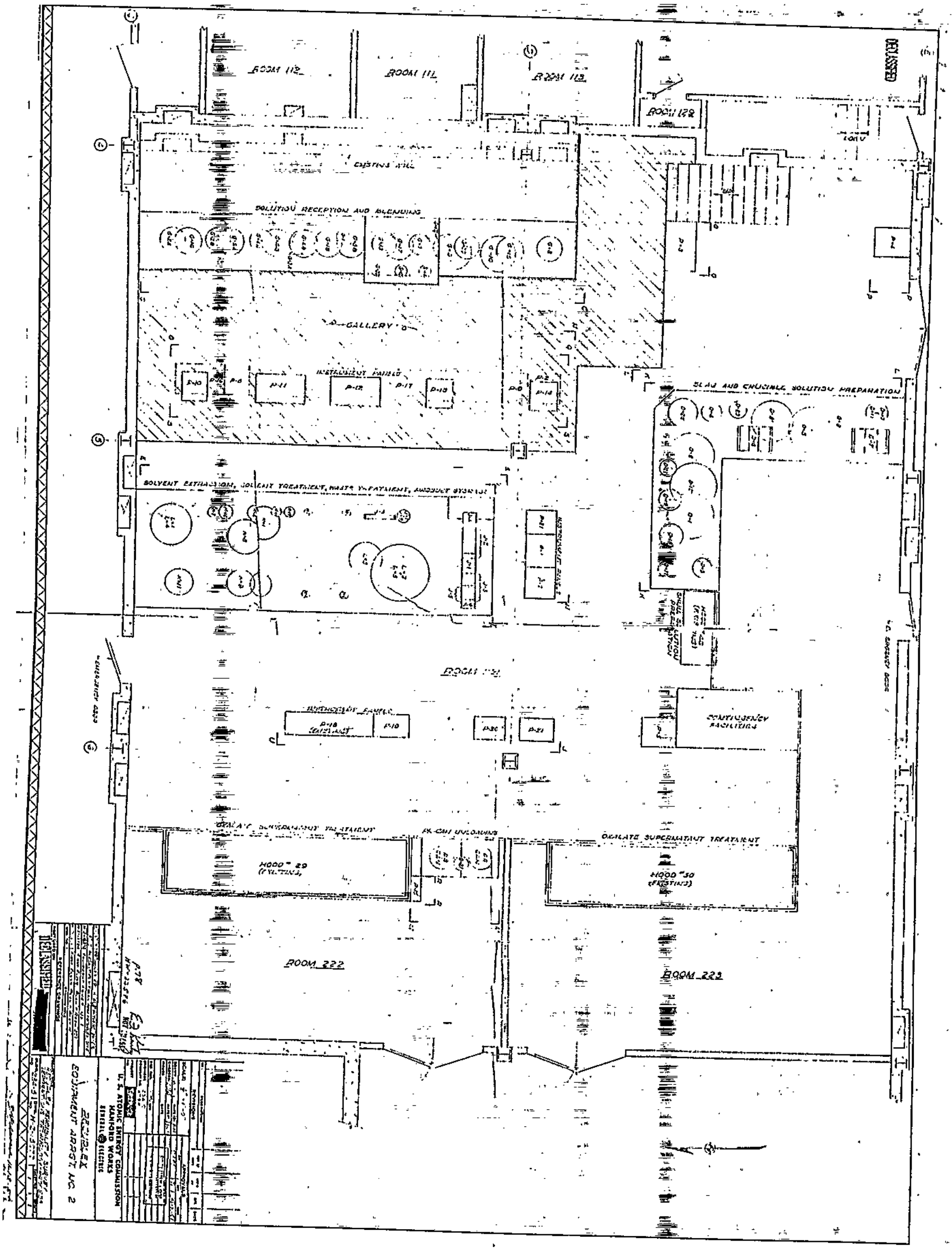
HANFORD WORKS
GENERAL ELECTRIC

RECUPLEX EQUIPMENT
ARRANGEMENT - No.1

RECUPLEX FEASIBILITY SURVEY

234-5 H-2-15076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ROOM 112
ROOM 111
ROOM 113
ROOM 128
EXISTING WALL
SOLUTION RECEPTION AND BLENDING
GALLERY
INSTRUMENT PANELS
P-10
P-11
P-12
P-13
P-14
P-15
P-16
P-17
SLAG AND CRUCIBLE SOLUTION PREPARATION
SOLVENT EXTRACTION, SOLVENT TREATMENT, WASTE TREATMENT, PRODUCT SYSTEMS
CONTINGENCY FACILITIES
P-18
P-19
P-20
P-21
OXALATE SUPERNATANT TREATMENT
HOOD #29 (EXISTING)
HOOD #30 (EXISTING)
ROOM 222
ROOM 223
P78
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HANFORD WORKS
GENERAL ELECTRIC
234-5
RECUPLEX
EQUIPMENT ARRGT NO 2
SCALE

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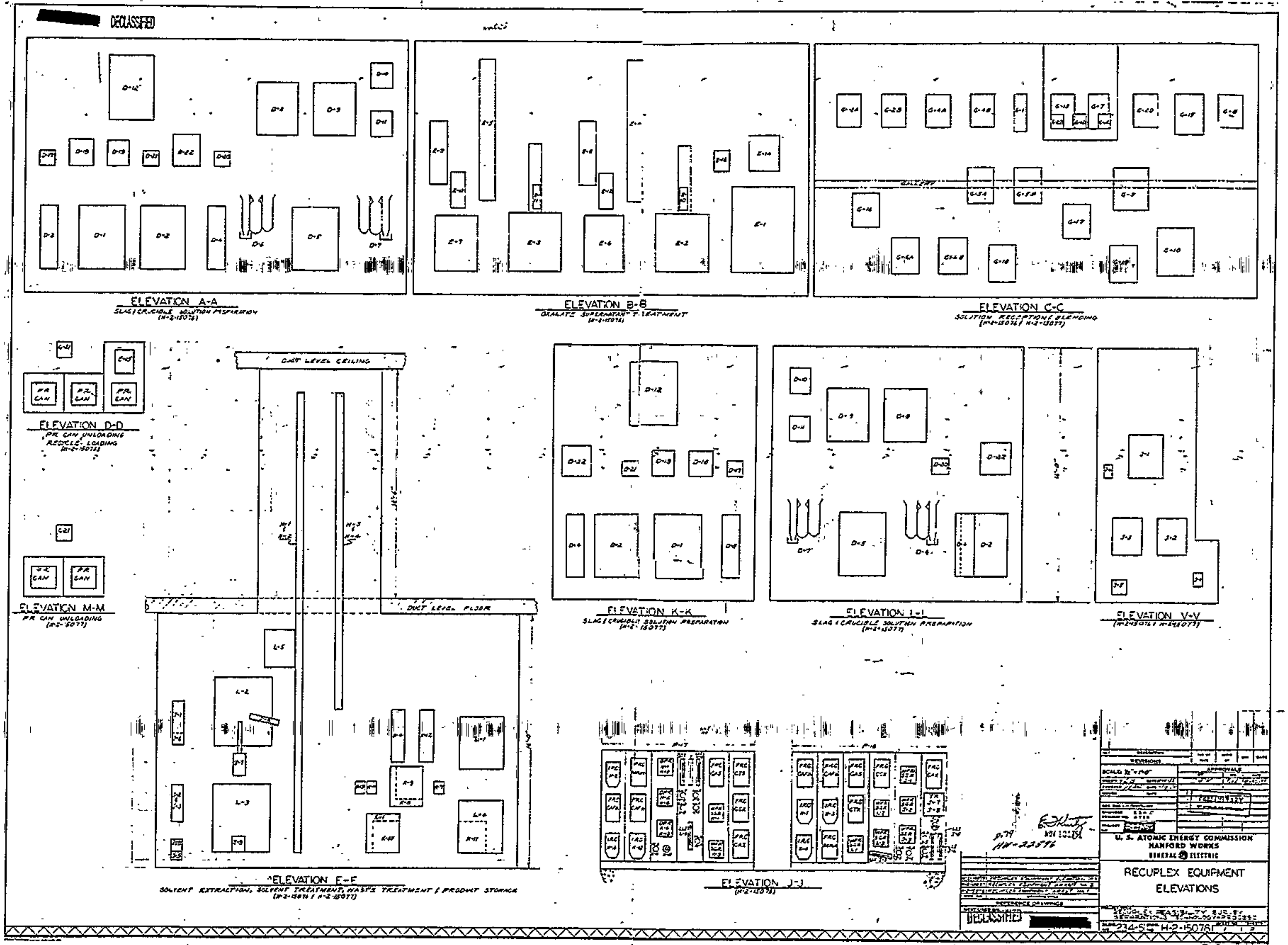

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SCALE ½" = 1'-0"

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HANFORD WORKS
GENERAL ELECTRIC

RECUPLEX EQUIPMENT
ELEVATIONS

REFERENCE DRAWINGS

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234-5 H-2-15076

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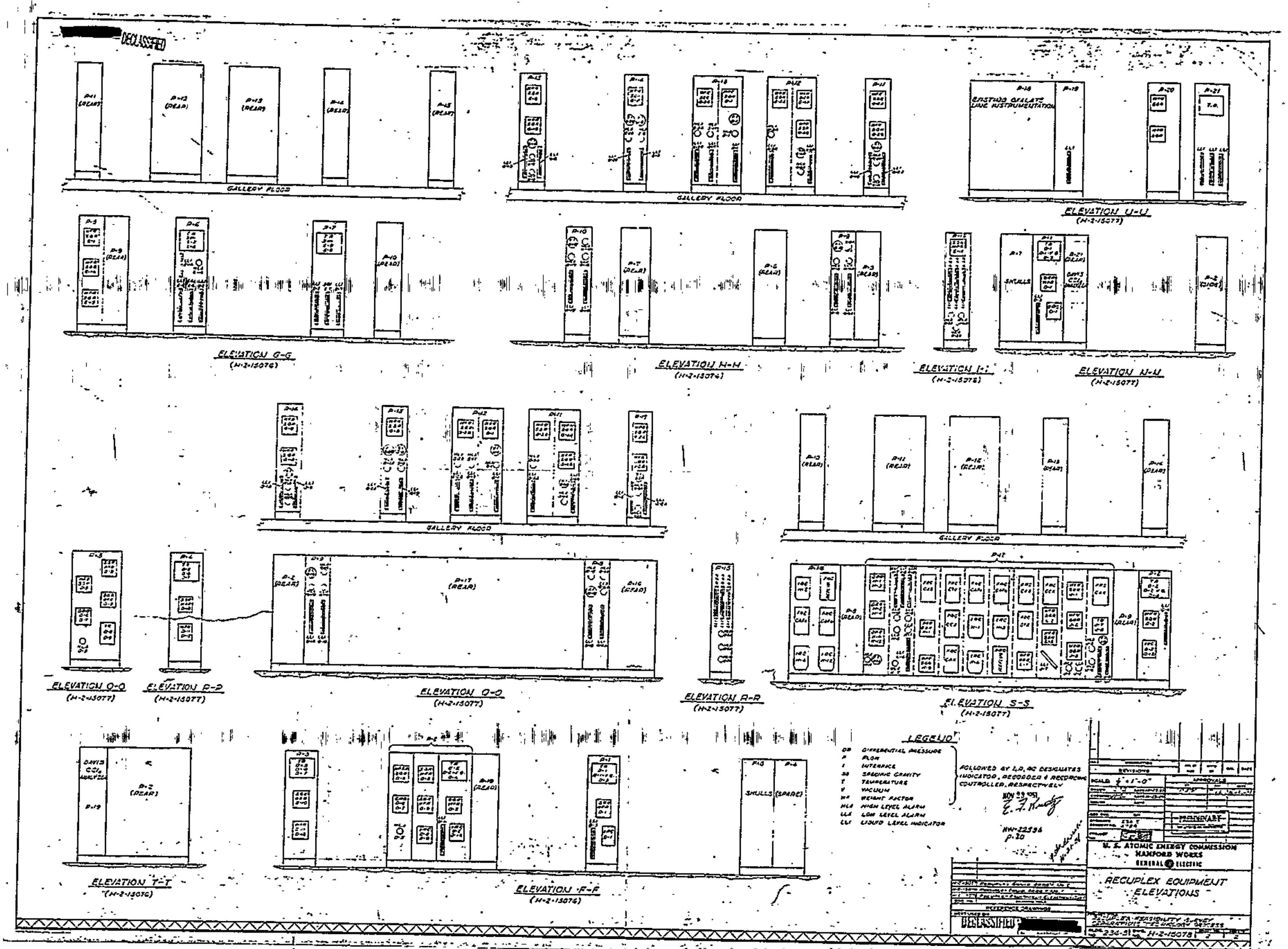

LEGEND

DP	DIFFERENTIAL PRESSURE
F	FLOW
I	INTERFACE
SG	SPECIFIC GRAVITY
T	TEMPERATURE
V	VACUUM
WF	WEIGHT FACTOR
HLA	HIGH LEVEL ALARM
LLA	LOW LEVEL ALARM
LLI	LIQUID LEVEL INDICATOR

FOLLOWED BY I, R, RC DESIGNATES INDICATOR, RECORDER & RECORDING CONTROLLER, RESPECTIVELY

NOV 23 1951
E. F. Kurtz

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p. 30

REFERENCE DRAWINGS

SCALE 3/4" = 1'-0"

PRELIMINARY

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

RECUPLEX EQUIPMENT
ELEVATIONS

RECUPLEX FEASIBILITY SURVEY
SEPARATIONS TECHNOLOGY SECTION

234-5 H-2-15078 2 2

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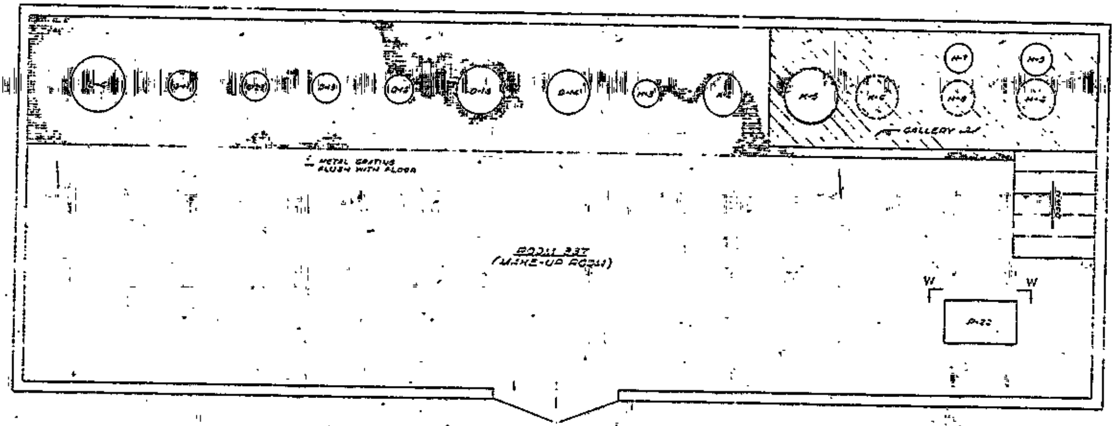
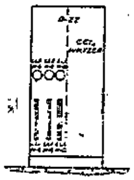
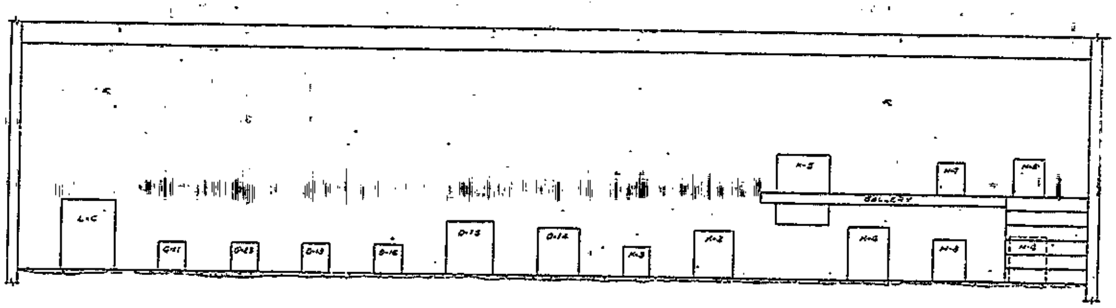


PLAN



ELEVATION W-W



ELEVATION
SCALE 1/8" = 1'-0"

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NOV 23

REFERENCE DRAWINGS

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REVISIONS
SCALE 1/8" = 1'-0"
APPROVALS

U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

CHEMICAL MAKE-UP ROOM

REDOX FEASIBILITY SURVEY
SEPARATION TECHNOLOGY PROCESS

234-5 H-2-15033

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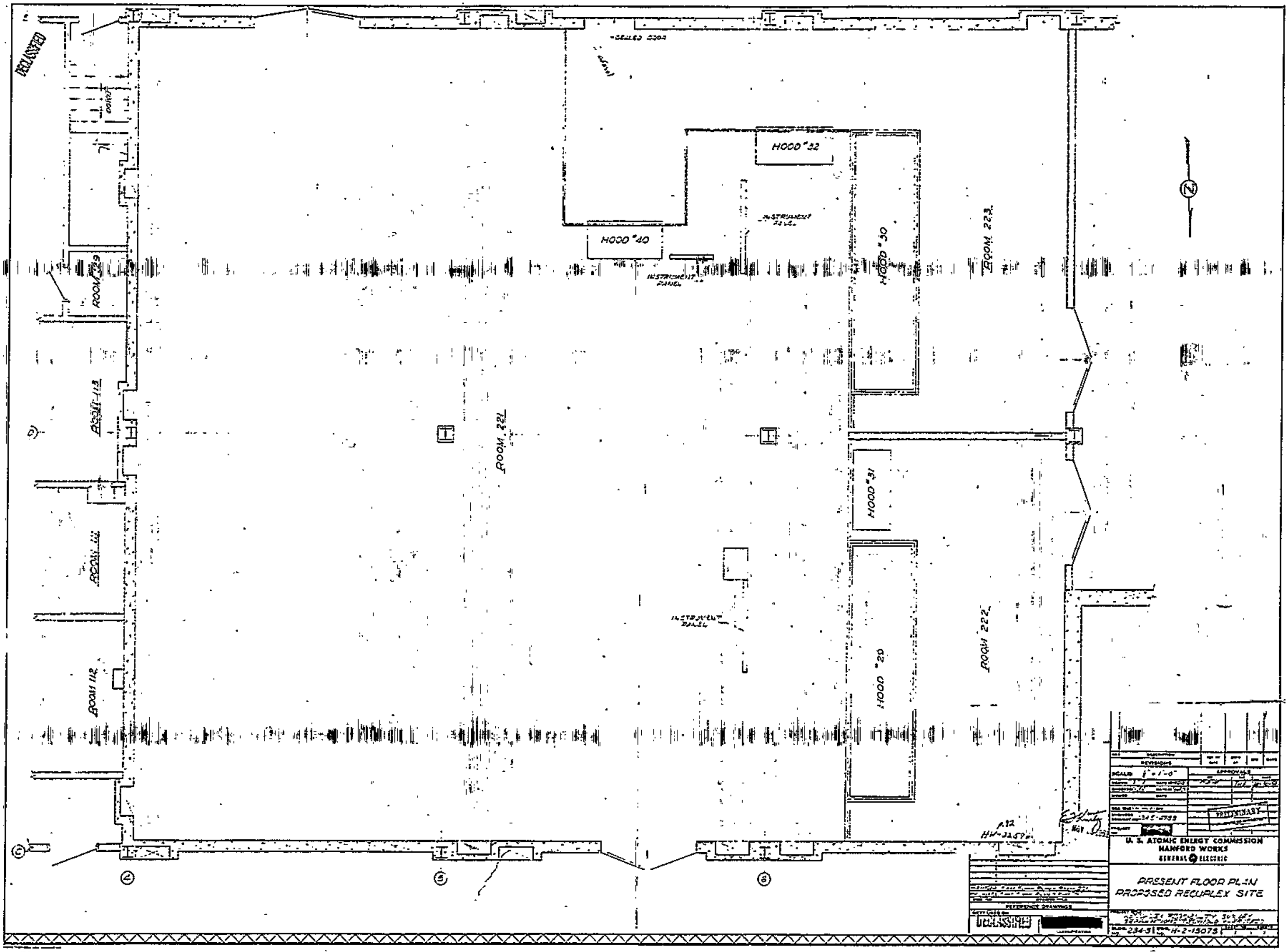

SCALE 1/8" = 1'-0"

PRELIMINARY

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U. S. ATOMIC ENERGY COMMISSION
HANFORD WORKS
GENERAL ELECTRIC

PRESENT FLOOR PLAN
PROPOSED RECUPLEX SITE

BLDG. NO. 234-5 DWG. NO. H-2-15075