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MEMORANDUM FOR FILE

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PRELIMINARY DESIGN PROPOSAL FOR A FACILITY
TO RECOVER IRRADIATED ENRICHED FUEL

The production of tritium in excess of that produced in the reactor control rods involves the use of enriched fuel loadings. After a portion of the U-235 has been consumed, the fuel elements are discharged and the remaining U-235 must be recovered and purified for reuse. In the event that the Savannah River reactors are so loaded, a new 200 Area facility for the recovery of U-235 probably will be required. This memorandum is a preliminary design proposal for such an installation.

Late in 1954, a contract was awarded to the Oak Ridge National Laboratory to assist in the establishment of design criteria for a U-235 recovery plant. At that time planning was in progress for the irradiation of thorium, using enriched fuel elements. Liaison with ORNL was provided by D. S. Webster and the writer and the work was periodically reported (Reference 3) and culminated in a design proposal (Reference 1). This work is the primary source for this proposal.

In summary, it is proposed that in the event that the scope of SRP operations be expanded to include U-235 processing, a new plant should be built in 200-H Area. Maximum possible use should be made of presently existing equipment in 200-H Area. In particular it is recommended that nitric acid dissolution be used and tributyl phosphate extraction be the primary purification means. The proposed equipment maintenance philosophy is the ORNL "Contact Maintenance" type.

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Details of this proposal are discussed under the following headings:

- I. Design Criteria
- II. Chemical Flowsheet Description
- III. Building Description
- IV. Special Equipment Description
- V. Research and Development Required Before Final Design
- VI. Bibliography

I. DESIGN CRITERIA

A. Capacity

It is ambiguous to speak of the capacity of the plant in terms of uranium throughput because the weight of the alloy (U + Al) to be processed determines capacity and the uranium content of the alloy will vary as a function of "burnout". The design capacity of the plant is best expressed as 680 lbs. of U-Al alloy per day. It is assumed that the uranium content of the alloy is about 8.7% (as delivered to the dissolver) and the uranium is 93.4% U-235 before irradiation. With these assumptions, the daily throughput may be expressed as 25 kg U-235 (ignoring burnout). The need for 25 kg/day throughput as a function of tritium production is discussed elsewhere.

B. Post-irradiation Lag (Cooling) Time

Short-lived fission products that saturate in the reactor decay to tolerable levels after 90 days cooling time. However the presence of U-237 which arises from the reaction

would constitute a serious radioactive contaminant to the product. Consequently a 150-day cooling time is required to reduce U-237 activity to such a level that it does not contribute significantly to the radioactivity of the product.

C. Chemical Flowsheet

The successful production experience with the nitric acid-tributyl phosphate (TBP) solvent extraction system in a similar process (Purex) governed the decision to adapt the U-235 recovery process to this system. Two major variations were considered.

Caustic Dissolution Process - The aluminum in the fuel elements is dissolved as NaAlO_2 in strong caustic while the uranium remains in solid state probably as an oxide. The uranium is centrifuged, dissolved in nitric acid and purified in small scale TBP cycles.

The advantages of the caustic dissolution process included low waste volume and small extraction equipment. As research progressed, however, difficulties mounted. A significant portion of the uranium residue from caustic dissolution was less than one micron in diameter which rendered centrifugation very slow requiring several centrifuges (about seven) and at best resulted in the loss of 0.1% of the U-235 through this operation alone. Economic consideration indicated that routine loss of 0.1% of the product more than off-set the waste volume advantage of the caustic dissolution process.

Nuclear criticality control is a serious problem with both schemes. However, the centrifugation of solid U-235 oxide in batch sizes operationally feasible requires the elimination of both batch and concentration control of criticality. As a result, the feed tanks, centrifuges and run tanks must be critically sized which when considered with the volumes required represents a considerable complexity. The ORNL study concluded that the following equipment would be required for separation of the U-235 oxide from aluminum:

- 7 centrifuges (6 receiving and one unloading)
- 2 waste collection tanks (slab design) 9.5' x 4" x 15'
- 3 centrifuge receiver tanks (slab design) 4" x 1' x 20'
(used also as U dissolvers)
- 2 feed tanks (cylindrical design) 7-3/4" ID x 15'

Critically safe centrifuges are not available. Discussions with centrifuge vendors have indicated that design would be difficult but possible.

Acid Dissolution Process - The aluminum and uranium are dissolved in nitric acid (mercury catalyzed). The aluminum and the fission products are removed in larger scale TBP extraction cycles.

The acid dissolution process was chosen. It is quite true that the extraction equipment with the caustic dissolution process is much smaller than that used for this process. However, this margin of advantage is lost when the complex centrifuge separation system and the attendant uncertainties are taken into account.

The adaptability of the acid dissolution process to better demonstrated design philosophies was a deciding factor. The equipment sizing and arrangement will be closely similar to that demonstrated with a number of processes at the ORNL Pilot Plant and somewhat similar to that at CPP Idaho. Both plants are of the "Contact Maintenance" design which is discussed under the succeeding heading.

D. Building Philosophy

The proposed building embodies an equipment maintenance philosophy new to Savannah River separations plants. Present installations involve orientation of the process equipment, piping, instrument leads etc. in a straight line in such a manner that a remotely operated crane, guided through a periscope type optical arrangement, can perform necessary maintenance while the equipment is still very radioactive. The advantages of this philosophy are undeniable where tonnages of material are processed daily as with the natural uranium case. Direct maintenance, on the other hand, involves installation of all contaminated or possibly contaminated equipment in shielding cells that are segmented according to the level of radioactivity and the susceptibility to breakdown. When equipment malfunction occurs, the cell is shut down, the equipment is decontaminated by prearranged procedure and repairs are made, in place, in a normal chemical plant manner. Continuity of operation is of primary concern. The advantage of direct maintenance is capital cost savings. A preliminary cost analysis is in preparation. It is expected that the capital outlay for the proposed installation will be less than one-half that of a comparable crane maintained plant.

It is planned that the most radioactive equipment which will require the most time consuming decontamination will be duplicated. The relatively small equipment and process simplicity of this U-235 recovery process conveniently adapts to a direct maintenance philosophy. Specifically, the equipment from the dissolver charging through first cycle extraction will be duplicated. Should malfunction of this equipment occur, the cell will be shut down and repaired while the duplicate picks up the duty. Such safeguards designed into the process, according to experience at Idaho and Oak Ridge (albeit limited) assure continuity of operation at least equal to that of the conventional crane maintained plant.

E. Process Philosophy and Criticality Control

Safe operation with a fissionable material such as U-235 is a prime design consideration. From a practical standpoint, a critical assembly in water may be avoided by (1) limiting the accumulation of U-235 to less than 800 g; (2) limiting the concentration of U-235 to less than 12.0 g/l; or (3) insuring high neutron loss from the vessel by geometric design.

Batch control, method No. 1, is somewhat unreliable because of its sensitivity to operator error and analytical inaccuracy. In addition, where high throughput rates, as in this case, are necessary, handling of the number of batches of less than critical mass would be extremely complex in design and in operation. This method of control is not assumed in the proposed design.

Geometric design is not subject to operational errors and is used throughout the main line process and all vessels and equipment feeding or receiving from the main line. Two exceptions presently exist because of the large volumes of solution handled. They are the first cycle feed adjustment tank and the first cycle aqueous waste run tank. The adjustment tank normally holds solution containing around 5 g/l U-235 and the run tank around 0.0005 g/l U-235. This affords adequate protection as a solution concentration safeguard but mal-operation could conceivably vary the concentration such that this safeguard would be lost. This problem is under active study.

The extensive use of geometric criticality control substantially defines much of the equipment and the philosophy of operation. Since run and catch tanks must be geometrically safe, the inclusion of appreciable solution hold up would involve many tanks which would introduce serious operational complexity. As a result the main process is essentially continuous from first cycle feed make-up through final product collection. Waste streams are collected and analyzed before discharge and solvent is continuously reconditioned and returned to solvent storage.

F. Product Specifications:

These specifications are similar to those required of the Idaho Chemical Processing Plant.

1. Uranyl nitrate solution containing 15-25% by weight uranium and less than 1.5 M HNO_3 .

2. Cation Contaminants

Fe	< 1000 ppm
Al	< 6000 ppm
Na	< 200 ppm
Si	< 250 ppm
Ca	< 200 ppm (of U_3O_8)
Cr	< 200 ppm
Cu	< 200 ppm
Mg	< 200 ppm
P	< 300 ppm

total < 0.75% by weight of U

3.
 - a. Beta activity less than 30 times that of aged reference standard of pure U-235.
 - b. Gamma activity less than 100% greater than reference standard.
 - c. Plutonium less than 10 parts per billion uranium.

II. CHEMICAL FLOWSHEET DESCRIPTION

Figures 1 and 2 are a flowsheet outline of the process. Material balance flowsheet (Table I) has been tested but is not considered sufficiently demonstrated for final recommendation. The process outline and equipment requirements are firm but final design, if possible, should await further development to avoid expensive assumptions.

The decontamination factors required to meet the specifications as outlined earlier are calculated to be:

$$\beta - 4.6 \times 10^6$$

$$\gamma - 1 \times 10^7$$

$$\text{Pu} - 1 \times 10^4$$

The solvent extraction process has been demonstrated to yield the following:

$$\beta - 2.3 \times 10^7$$

$$\gamma - 2.5 \times 10^7$$

$$\text{Pu} - 3 \times 10^5$$

The silica gel column clean up performance has not been demonstrated but since the major contaminant after solvent extraction should be Nb-Zr, this column should provide valuable backup.

These demonstrated decontamination factors are subject to question. For instance, the effect of the use of recycled solvent is as yet inadequately demonstrated. This must be studied further before a flowsheet can be drawn with confidence.

FIG. 1
PRELIMINARY CHEMICAL
FLOW DIAGRAM (DISSOLUTION AND 1st CYCLE)

----- (6% TBP in Diluent)

———— (Aqueous)

FIG 2
PRELIMINARY CHEMICAL FLOW DIAGRAM
(2nd CYCLE AND SILICA GEL TREATMENT)
----- (18% TBP in Diluent)
———— (Aqueous)

TABLE I.

Process Flowsheet (From ORNL Material Balance E-22683)

<u>Steam</u>	<u>Composition</u>	<u>Units of Flow²</u>
1. Dissolver charge	26.77 kg U ¹ 258 kg Al	per batch
2. Dissolver solvent	50% HNO ₃ (10.39M) .018 M Hg(NO ₃) ₂	1030 gal/batch
3. Dissolver product	1.8 M Al(NO ₃) ₃ 1.0 M HNO ₃ 5.04 g/l U ¹ 0.013 M Hg(NO ₃) ₂	1400 gal/batch
4. 1 AF	Same as dissolver product	58.4 gal/hr
5. 1 AS	3.00 M HNO ₃ 0.75 M Al(NO ₃) ₃ 0.02 Fe(NH ₂ SO ₃) ₂	10.6 gal/hr
6. 1 AX	6% TBP in diluent	23.4 gal/hr
7. 1 BX	0.01 M HNO ₃	11.8 gal/hr
8. 1 BP	0.2 M HNO ₃ 25.0 g/l U ¹	11.8 gal/hr
9. 1 BP Evaporator Product	1.55 M HNO ₃ 195 g/l U ¹	1.51 gal/hr
10. 2 AF	3.0M HNO ₃ 160 g/l U ¹	1.84 gal/hr
11. 2 AS	3.0 M HNO ₃	1.22 gal/hr
12. 2 BX	0.01 M HNO ₃	5.9 gal/hr
13. 2 BP	0.12 M HNO ₃ 50 g/l U ¹	5.9 gal/hr
14. 2 BP Evaporator Product	0.84 M HNO ₃ 350 g/l U ¹	0.84 gal/hr

1 - U refers to U²³⁸ + U²³⁵ assuming no burnout of either.

2 - Batch corresponds to one 24 hour operating day.

Feed Preparation

The U-Al fuel element is dissolved with 10.39 M HNO_3 - 0.018 M $\text{Hg}(\text{NO}_3)_2$ solution. The resulting dissolver solution is 1.8 M $\text{Al}(\text{NO}_3)_3$, 1.0 M HNO_3 , 5.04 g/l uranium and about 0.01 M $\text{Hg}(\text{NO}_3)_2$. The solution is passed through digesters to insure complete dissolution of fines which might prove a criticality hazard if allowed to go directly to a large diameter tank. The solution is evaporated in the coarse feed adjustment tank, centrifuged and adjusted to feed conditions in the feed adjustment tank.

First Cycle Solvent Extraction

This cycle provides gross separation from plutonium, aluminum and fission products. For reasons discussed later, pulse columns are the desired solvent contactors. The solvent recommended by ORNL is composed of 6% by volume TBP in a special Amsco Naptha No. I sulfonated hydrocarbon diluent. This is subject to change as SRL data becomes available. Since the uranium concentration is so low, as compared to Purex, the TBP content has been dropped to 6% by volume. This allows the saturation, or nearly so, of the solvent with uranium at hydraulically stable flow ratios. Since the extractability of uranium is higher than most or all the radioactive impurities, such saturation tends to improve decontamination. Plutonium is converted to the non-extractable form (Pu III) by the addition of ferrous sulfamate to the 1A column scrub stream. The column is operated in acid condition to inhibit emulsion formation and $\text{Al}(\text{NO}_3)_3$ is added to the scrub to insure the extraction of the last traces of uranium. Decontamination factors through the first cycle laboratory demonstration were:

Gross β	Gross γ	Zr-Nb	Ru	Pu
1.5×10^4	1.6×10^4	9.0×10^2	3×10^5	3×10^5

The aqueous product from the first cycle is evaporated and adjusted to about 160 g uranium/liter for feed to the second solvent extraction cycle.

Second Solvent Extraction Cycle

The higher uranium concentration in the second cycle feed requires raising the TBP concentration to 18% by volume. A gross gamma decontamination of 25×10^3 is expected and the uranium concentration of the product is about 50 g/l. This solution is evaporated to 350 g/l and sent to the silica gel columns.

Silica Gel Columns

The principal contaminant of the uranium should be Nb-Zr activities. Silica gel has proved to be a highly effective selective

absorbent for these ions. The product from the second solvent cycle is passed through silica gel beds until the gel surfaces are effectively saturated with Nb-Zr at which time the uranium solution is displaced with nitric acid and the Zr-Nb is removed with oxalic acid.

Waste Handling

The original conception of this plant involved extensive use of the waste disposal equipment (both liquid and gas) in an existing 221 Building then scheduled for conversion to thorium processing. This proposal modifies the original plan.

A. High Activity Waste

The high activity waste from the 1A extraction cycle and feed adjustment evaporator will be about 2000 gallons per day of solution composed of 1.3 M $\text{Al}(\text{NO}_3)_3$, 0.3 M HNO_3 , 0.003 M $\text{Hg}(\text{NO}_3)_2$ plus traces of silica, plutonium, corrosion products, $\text{Fe}(\text{NH}_2\text{SO}_3)_2$, and substantially all the fission products. This solution must be added (with agitation and cooling) to sufficient 50% NaOH to neutralize the acid, precipitate $\text{Al}(\text{OH})_3$ and redissolve it to NaAlO_2 plus 7% excess NaOH. This requires about 1100 gal 50% NaOH. The solution is evaporated back to the original 2000 gallons and transferred to a standard waste storage tank. Overheads from this evaporation must be re-evaporated to produce process water quality. This waste differs so grossly from Purex wastes that it is impractical to try to schedule them into a 221 Building. The new facility must be prepared to handle this task.

B. Low Activity Waste

Low activity waste solution from the second solvent cycle, silica gel column flushes, evaporator overheads and solvent recovery streams constitutes about 650 gallons per day of 0.3 molar HNO_3 plus traces of NaNO_3 and oxalic acid. This solution must be evaporated in a manner similar to that practiced in Purex at SRP. The small volume of this stream suggests most strongly that it be handled in a canyon building.

C. Dissolver Off Gas

Separate facilities, located in the new process building, will be required for treatment of dissolver off gas. Although 150 days cooling time is adequate to reduce I-131 discharge in this gas to tolerable limits, it is recommended that provision be made for installation of a silver nitrate iodine removal tower and dehumidifying and filtration equipment after the manner presently practiced in Purex at SRL.

D. Cell Vent System

The recommended philosophy of cell venting varies from that practiced in the 221 canyons. Only enough negative pressure should be maintained inside the cells to insure inleakage from the surrounding atmosphere. This will minimize the pumping expenditure and will provide a satisfactory system of contamination control as demonstrated at ORNL.

E. Process Vessel Vent Gas

Process vessel vent gas is extremely small in comparison to that of a 221 Building. It is recommended that this gas be passed through the presently installed sand filter and then to the stack.

Product Handling

The product is sampled for accountability and analysis and shipped in critically safe cylinders of less than 5.5" O.D. with guard rails to insure that at least 2' spacing, wall to wall, exists between stored cylinders.

Solvent Recovery

The solvent is recovered by successive washes in sodium carbonate, nitric acid and water. Since a criticality hazard exists in this operation, continuous recovery in critically sized containers is mandatory. The carbonate catch tank will contain uranium entrained in the solvent and must therefore be critically sized.

III. BUILDING DESCRIPTION

Figures 3 through 8 are presented to provide a rough representation of the building as presently visualized. Actually they represent an ORNL design prepared during preliminary planning. The building is to be located in the 200-H Area to facilitate maximum use of presently installed facilities.

The salient features are obvious from the figures. The equipment distribution is discussed in II D. Smaller equipment, subject to breakdown, such as pumps, pulse feeders and valves must be located in small isolated shielding modules adjacent to the main cells. This will enable decontamination and repair of the most troublesome equipment without entering the main cells.

Every effort must be made to anticipate difficulties to be encountered in cell decontamination. Since concrete adsorbs fission products and is difficult to decontaminate, a stainless steel cell liner must be installed. In addition, decontamination sprays and sump collection equipment must be installed.

The supporting laboratories are adjacent to the sampling equipment to reduce the lag time between sampling and analysis. A greatly improved simplified sampling device has been designed and demonstrated at ORNL and is extensively described in Reference 6.

IV. SPECIAL EQUIPMENT DESCRIPTION

A. Dissolver

Attempts to cut tubular fuel elements into smaller lengths resulted in such extensive contamination and mechanical complexity that it was decided to introduce the tubes into the dissolver intact. The dissolver is a slab thick enough to accommodate only one tube and high enough to accommodate the desired number of tubes plus de-entrainment freeboard.

Dissolver charging is a difficult design problem. Figure 9 shows the proposed rotating fuel tube magazine which would be loaded in the 100 Area. The magazine is located on a flat car in such a manner that the magazine can be rotated at right angles to the rail car against the shielding wall adjacent to the dissolver. The fuel tubes are then shoved from the magazine through the shielding wall to a tray above the dissolver. The tray is tipped and the tube falls into the dissolver. Design of this scheme has progressed sufficiently that it can be proposed with confidence. A similar device has been used for several months in charging Thorex material to the ORNL Pilot Plant.

B. Solvent Extraction Column

Pulse or packed columns present the only demonstrated technique for solvent extraction under geometric critically safe conditions. Although the exact column height required is not known, the use of pulsed columns considerably decreases the height requirements as compared to that required for packed columns. In order to save building height, the 1A column recommended is a "concatenated pulse column" which has been demonstrated at ORNL.

Fig 3

DA4-55-417

UPM-55-411

SECTION-B.B

14-5-41

SECTION-CC

2

SECTION-EE

Fig 8

Figure 9
Fuel Tube Carrier

V. RESEARCH AND DEVELOPMENT REQUIRED BEFORE FINAL DESIGN

The experience accumulated at ORNL and CPP at Arco, Idaho with installations similar to that proposed will be extremely valuable. With this as a guide, preliminary design may commence immediately. It is recommended, however, that certain programs be initiated in support of final design. Those programs are outlined below:

Chemical Flowsheet (SRL)

A chemical flowsheet that gave good performance has been partially demonstrated at ORNL. It is recommended that high activity testing of this flowsheet on miniature mixer-settler scale be performed at SRL to test for ruggedness under plant conditions and to provide data for flowsheet improvement. This work also will be necessary to firm basic design criteria for extraction and silica gel equipment. This work can best be done at SRL where the effect of production conditions on extraction processes are best appreciated. The mixer-settlers and associated equipment are set up and can be adapted to this process.

Equipment Design (SRL-TNX)

A large portion of the proposed plant involves equipment that is not demonstrated at SRL. An extensive program at TNX is indicated to demonstrate prototype adequacy and train personnel for plant operation. The most important equipment to be tested is the concatenated 1A pulse column and the 1B column. These columns and attendant equipment must be installed at TNX and demonstrated prior to final building commitment if expensive column height assumptions are to be avoided.

It is probable that technology on the proposed evaporators is adequate so that TNX demonstration will not be required.

The small scale dissolver has been set up at ORNL and promises to provide adequate design information.

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 - E-19680 - Tipper and hopper assembly study remotely maintained.
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 - B-22675 - Infinitely safe vessels, Group II.
 - D-22662 - Solvent recovery turbo-mixers.
 - D-22682 - E-I-E Evaporator.
 - D-22679 - Vapor condensers.
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