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ARH-SA-78

CONF-7104M--3

Plutonium-Uranium Partitioning by a Reflux Extraction Flowsheet

MASTER

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August 18, 1970

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AEC Contract AT(45-1) -2130

Atlantic Richfield Hanford Company
Richland, Washington

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PLUTONIUM-URANIUM PARTITIONING
BY A REFLUX EXTRACTION FLOWSHEET

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August 18, 1970

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For presentation at the

International Solvent Extraction Conference, 1971
The Hague, The Netherlands
April 19-23, 1971

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ABSTRACT

A new solvent extraction flowsheet has been developed for the processing of unirradiated plutonium-uranium scrap. By refluxing plutonium product as Pu(III) in a multi-stage plutonium stripping-partitioning system, uranium is salted into the organic and pure plutonium is quantitatively separated from uranium. A hydrofluoric acid strip removes the final traces of plutonium from the organic phase containing the uranium. The flowsheet was plant-tested; and problems evident in the plant test were solved by comprehensive laboratory tests. The fully-developed flowsheet is ready for plant application.

PLUTONIUM-URANIUM PARTITIONING
BY A REFLUX EXTRACTION FLOWSHEET

INTRODUCTION

A new solvent extraction flowsheet has been devised for the separation and purification of plutonium from systems containing uranium. The flowsheet permits the purification and concentration of plutonium in a single solvent extraction cycle. This new concept was developed by Atlantic Richfield Hanford Company personnel in the Plutonium Reclamation Facility, a plutonium scrap processing plant operated under prime contract for the Atomic Energy Commission.

It appears that this flowsheet could be applied directly to the processing of scrap from fuel fabrication for both the light water and fast breeder reactors. With further development, it might be useful in the processing of irradiated fuels.

The development of the flowsheet and the parameters pertinent to process performance are described in this paper.

SUMMARY

After the plutonium-uranium scrap is dissolved or leached, the resulting solution is adjusted for salting strength and the plutonium is adjusted to the extractable (IV) and (VI) states. Uranium and plutonium are coextracted in a multi-stage extractor (Unit I, Figure 1), using 20 percent TBP (tributyl phosphate) in CCl_4 (carbon tetrachloride) as the solvent. In the same extractor, the loaded solvent is scrubbed for impurity removal.

In a second multi-stage unit (Unit II, Figure 2), plutonium

is reduced to the aqueous favoring plutonium(III) state with hydroxylamine nitrate. The purified Pu(III) leaving the concentrated end of this second multi-stage unit is split into a product stream and a reflux stream which flows to a hold-up tank where it is oxidized to the extractable Pu(IV) state by merely adding nitric acid. This Pu(IV) stream is re-introduced as a scrub stream in the first extractor. Plutonium(III) product is withdrawn from the system when a 30 to 60 g Pu/liter concentration is attained. Uranium is removed from the Pu(III) by a 20 percent TBP-CCl₄ scrub in the initial stages of this second extractor (see Figure 1).

The uranium quantitatively leaves the second extractor via the organic stream. The plutonium impurity (about 1 percent) in this uranium stream is removed by a novel hydrofluoric strip in a third extractor (Unit III, Figure 2). Further conventional extractors (not discussed in this paper) remove the uranium and organic impurities from the organic stream.

The flowsheet has been tested in the laboratory and the plant to identify and correct process difficulties. Full-scale plant application is planned.

DISCUSSION

BASIC FLOWSHEET

Scrap bearing plutonium and uranium is dissolved or leached, and appropriate salting and plutonium valence adjusting agents are added as required. Both plutonium and uranium are quantitatively coextracted from the feed by a mixture of 20 percent TBP in a CCl₄ diluent. This extraction operation can be performed in some type of multi-stage equipment; *e.g.*, a mixer-settler unit or a packed or pulsed column unit. The solvent extraction system is depicted in Figure 1.

Addition of nitric acid and sometimes nitrite ion to the

FIGURE 1

REPRESENTATION OF THEORETICAL FLOWSHEET

aqueous extraction unit feed ensures a predominance of Pu(IV), with some Pu(VI). This valence combination is extractable within 10 mixer-settler stages. The uranium in the stable (VI) valence is easily extractable. Sufficient stages are used to ensure negligible plutonium and uranium losses in the salt waste.

Additional scrub stages are required for removing entrained and extracted impurities from the plutonium and uranium in the organic phase. Two scrub streams are used. The initial two-stage scrub section uses refluxed plutonium product as the scrub stream and removes the bulk of the impurities from the uranium and plutonium. One additional scrub stage uses a pure nitric acid scrub stream and removes the final impurities from the refluxed plutonium product with repeated washings. The number of stages required will depend upon the degree of impurity removal required.

The coextracted plutonium-uranium flows to a combination plutonium stripping and plutonium-uranium partitioning unit--Unit II in Figure 1. The organic stream enters Unit II at the fourth stage, leaving three stages of scrub section for removal of the uranium from the plutonium stream. The number of scrub stages used depends upon the desired capacity. For the maximum-sized equipment allowable to provide criticality control (e.g., geometrically-favorable 6-inch diameter sieve plate pulse column) and using a 20 percent TBP extractant, three stages of uranium scrub provide a potential of 100 kg per day uranium capacity. Organic of the same composition as used in Unit I is used as scrub in Unit II and is introduced in Stage 1 (see Figure 1).

Two stripping solutions are introduced into the stripping partitioning unit, Unit II--one at the 24-stage and one 2 stages below the organic feed stream entry point, the sixth stage. The stripping streams contain various amounts of reducing agents,

hydroxylamine nitrate and nitric acid. An estimated 20 stages are required for quantitative stripping of the plutonium from the organic phase.

The two effluent streams from Unit II are the pure plutonium product and the organic effluent, containing the uranium and a minor amount of the plutonium [usually less than 0.1 gram per liter]. Almost all of the plutonium product is refluxed to the thirteenth stage of Unit I as the intermediate scrub. When the refluxed plutonium stream reaches the desired concentration, plutonium is drawn off as product.

The partition principle is based on maintaining a high concentration (30 to 60 grams per liter) of predominantly Pu(III) in the scrub section and the initial stripping stages (stages 5 and 6). Plutonium(IV) and plutonium(VI) favor the organic in a salted medium--whereas plutonium(III) favors the aqueous phase even in a salted medium. The high concentration of plutonium(III) nitrate salts the uranium into the organic phase. The Unit II scrub stream (20 percent TBP in CCl_4) removes the uranium quantitatively, and the plutonium is only slightly refluxed in this scrub section.

The high plutonium(III) nitrate salting strength is maintained with a reducing agent. Selection of the reducing agent is the key to the one-cycle system. The reducing agent must be strong enough to cause a predominance of plutonium(III) in the scrub section and the concentrated product portion of the stripping section. The reducing agent must not introduce impurities, since much of it remains with the concentrated plutonium product.

The reducing agent found to be optimum was hydroxylamine nitrate (HN). This chemical can reduce plutonium(IV) to plutonium(III) in low nitric acid mediums (0.5 to 1.5M). Other reaction products are gases-- N_2O (major constituent), N_2 , and a small amount of NO_2 . However, the equilibrium shifts

quantitatively to plutonium(IV) in nitric acid concentrations greater than 3.5M.

One problem with HN is the formation of the NO_2 gas from the reaction of plutonium(IV) with HN. Some of the NO_2 is solubilized and forms a nitrite ion which oxidizes plutonium(III) to plutonium(IV). The NO_2 reacts with HN, forming N_2O , N_2 , and small amounts of additional NO_2 . The conversion back to plutonium(IV) can be minimized.

The stripping agent introduced at the dilute plutonium end of the stripping-partition unit, Unit II (see Figure 1, Stage 24), forms plutonium(III). As the aqueous or light phase goes from Stage 24 to 23, etc., an autocatalytic breakdown of HN takes place due to the formation of the nitrite ion in the plutonium(IV) to plutonium(III) reaction. The excess NO_2 oxidizes plutonium(III) to plutonium(IV), and plutonium(IV) may predominate in the middle stages (13, 14, 15, etc.). If plutonium(IV) predominates, the flowsheet is not workable. [This will be discussed later under *Plant Testing*.] If a second HN stream is added at about the sixth stage, a plutonium(III) medium can be maintained at the top of the column; and the formation of excessive plutonium(IV) below the addition point is minimized. This second stream, 1.2M HN and 1.0M HNO_3 is added approximately two stages below the organic stream entry point. It cannot be added too many stages from the plutonium end because, again, the kinetics involved with the formation of nitrite ion may cause a predominance of plutonium(IV) at the product end of the unit.

One additional unit is important to the one-cycle system--Unit III. This unit is necessary due to the formation of DBP (dibutyl phosphate), the breakdown product of tributyl phosphate (TBP). Dibutyl phosphate complexes plutonium as an almost unstrippable plutonium(IV) compound, $\text{Pu}(\text{NO}_3)_4 \cdot 2\text{DBP}$. Hydroxylamine nitrate (HN) will not efficiently strip plutonium from

DBP. However, hydrofluoric acid will quantitatively strip plutonium from DBP. The stripping agent used is 2.5M HNO_3 -0.25M HF. Nitric acid serves two purposes: (1) to ensure the bulk of the uranium stays in the organic phase [due to salting action of nitric acid], and (2) to prevent PuF_4 precipitation. The solubility of PuF_4 increases from 1 to 8 grams per liter as the nitric acid concentration is increased from 0.0 to 2.5M.

With an estimated 8 stages in Unit III, the plutonium may be reduced to the parts-per-billion level. The organic stream is transferred to a simple water strip system; and the uranium is removed for further processing as a relatively pure uranium stream. Dibutyl phosphate is removed from the organic in another unit by use of a carbonate wash. The organic is re-acidified and returned to Unit I and Unit II as extractant and scrub, respectively (see Figure 1).

PLANT APPLICATION

The Atlantic Richfield Hanford Company's Plutonium Reclamation Facility was designed and built as a plutonium scrap (no uranium) recovery facility.⁽¹⁾ Plutonium-bearing scrap is dissolved or leached with a mixture of nitric and hydrofluoric acids. The resulting solutions are purified in a single cycle solvent extraction system. The extraction column, called the CA Column (see *Nomenclature* for description of plant columns and streams, and Figure 2 for flow sketch of Plutonium Reclamation Facility columns); and the the plutonium stripping column, the CC Column, are 4-inch diameter sieve plate pulsed columns. The CA Column represents Unit I and the CC Column represents Unit II (see Figure 1). A short packed column (CO Column, Unit III) is used for removing the final plutonium from the organic by an HF wash.

This column system appeared adaptable to the basic flow-sheet approach previously described, and Pu-U scrap was

available; hence, the decision was made to test the Pu(III) reflux principle in the Plutonium Reclamation Facility (PRF). Plant stages available were not sufficient for high capacities (2 to 3 organic scrub stages, Unit II; and 2 to 3 stages in HF strip column, Unit III), but a significant capacity (10 kg uranium + plutonium per day) could be realized with only minor changes and additions. Later, additional stages could be added to increase plant capacity (up to 30 kg U + Pu/day is deemed possible).

Initial separation flask laboratory studies showed that a single stripping solution (last stage, Unit II) in the plutonium purification-partition column should be adequate. To study the overall valence balance and kinetics, a plant test was needed.

Plant Testing

Several hundred pounds of plutonium-uranium scrap were processed through the Plutonium Reclamation Facility. However, the first attempt was not successful since plant kinetics using a single stripping agent were such that a Pu(IV) medium predominated in the top of the CC Column. A brown Pu(IV)-color was apparent where a blue color [characteristics of Pu(III)] should have been present. Although Pu(IV) has a smaller extraction coefficient than U, the law of mass action prevailed and the Pu(IV) took over the bulk of the immediate available organic sites in the organic scrub stream. The end result was Pu refluxing down the column and U building up in the recycled Pu product stream to as high as 10 percent.

Plutonium(IV) and uranium accumulated in the center of the column, resulting in over-saturation of the organic and precipitation of the Pu(IV) nitrate DBP complex [only Pu(IV) can form this complex]. Also, the high Pu(IV) accumulation resulted in high Pu losses in the CC Column organic effluent

called the CCW. The high amount of Pu overloaded the final Pu clean-up unit, the CO Column. The HF strip precipitated PuF_4 , and high Pu in the U product resulted. Although the first plant test was unsuccessful, flowsheet problem areas were identified for subsequent comprehensive laboratory investigation.

LABORATORY TESTING

Laboratory testing was conducted in two different sets of equipment. One was a minimixer-settler unit to study the kinetics in certain areas; and the other, a mock-up of the plutonium stripping-partitioning column to study HTU's (height of a transfer unit) and column gassing.

Mixer-Settler

In this unit, plant solutions could be used and the number of plant stages and residence times could be duplicated. The scale-down ratio was 500:1. All stages were visible, permitting color determination. The tests were set up to first duplicate plant problems, and then varied to solve those problems.

Plant problems were successfully duplicated. By using only a single CC Column stripping agent at the last stage [bottom stage in the plant], a plutonium(III) medium could not be attained and the plutonium product soon contained excessive amounts of uranium.

Two flowsheet changes were made and each produced excellent results. One was the addition of a second HN stripping stream added two stages below the organic inlet, as previously discussed. The other was use of hydrazine as a holding agent in the CC Column bottom stripping stream. The hydrazine reacted with excess nitrite and the mixer-settler system showed a blue Pu(III) -color throughout. Although inexpensive, hydrazine

has a serious flowsheet disadvantage. Nitric acid alone will not convert the Pu(III) returning to the CA Column back to Pu(IV) if hydrazine is present. Nitrite must be added to destroy the hydrazine, and a "snowballing" effect can easily occur if excess nitrite is added. Serious gassing and reversals to predominant plutonium(IV) mediums can result. Therefore, the more easily controlled hydroxylamine nitrate (HN) approach was selected.

The mixer-settler study concluded with a long demonstration run.⁽²⁾ It demonstrated that the plant CA, CC, and CO Columns could probably handle 5 kg U/day (plus associated plutonium) with only the addition of a side HN stream.

Mock-Up of CC Column

The mock-up column duplicated the top 15 stages of the CC Column.⁽³⁾ Uranium was used; but plutonium could not be used in the pilot plant area available. To simulate the plutonium (NO₃)₃ salting strength, sodium nitrate was added to the HN side stream. The same type of perforated stainless steel plates as in the PRF plant CC Column (Unit II) were used. This was followed by a test with Raschig rings between the plates in the scrub section of the laboratory column (rings were also added to the PRF column).

The results showed

- Gassing in the CC Column actually improved column efficiency. To test severe gassing, excess nitrite was added to the system; however, no column flooding occurred. Gassing should be minimized in the plant to avoid organic entrainment into the plutonium recycle stream and eventual cavitation of pump in the product recycle hold-up tank.
- Height of a transfer unit (HTU) is lowered only slightly by the addition of 3/4-inch plastic Raschig rings

between the plates in the scrub section of the column. The rings were added to increase mixing in the lower volume velocity scrub section.

- Height of a transfer unit studies showed that with optimization of column amplitude and frequency, at least four transfer units could be realized in the plant scrub section.

PLANT FLOWSHEET

The final plant flowsheet for processing unirradiated plutonium-uranium scrap in the PRF is depicted in the flow sketch shown in Figure 2. The abbreviations used in Figure 2, plus other abbreviations and symbols used through the article are explained in the *Table of Nomenclature*, page 15. Existing equipment was used so there are some capacity limitations. For example, the CC Column originally designed as a plutonium stripping column was divided into a stripping-scrub column. Hence, some stripping area was lost and not as many scrub stages as desired could be added. The CO Column is a simple packed column with only two to three stages (eight shown in Figure 1) available.

A color code is also used showing the valence balance throughout a column system. The dark brown-to-black color shows a high concentration of plutonium (IV) [some green plutonium(VI) is present, but it is dwarfed by the predominant plutonium(IV)]. Plutonium (III) in the aqueous phase (the light phase) is blue. The organic, when free of actinides, is a yellowish-color.

The CA Column is an organic continuous unit with the aqueous being the dispersed phase. Here, the interface is at the top. In the CC and CO Columns, the aqueous phase is continuous and the interface is at the bottom (I stands for interface, Figure 2).

FIGURE 2
PLANT FLOWSHEET

BROWN - Pu - IV (1-15 g/l)

BLACK - Pu - IV (15-30 g/l)

BLUE - Pu - III (5-60 g/l)

YELLOW - 20% TBP IN CCl₄ NEGLIBLE Pu

ORANGE-BROWN-U-VI (1-20 g/l)

* REFER TO PAGE 15 FOR DESCRIPTION OF EACH STREAM AND COLUMN

CONCLUSIONS

A new plutonium-uranium scrap processing flowsheet (see Figure 2) has been developed from theoretical considerations, laboratory testing, and plant testing. Important conclusions based on this work include the following:

1. A Pu stripping column or combination Pu-U partition - Pu stripping column (as discussed above) operating on the reflux flowsheet principle can operate on a Pu(III) system. Use of the reducing agent, hydroxylamine nitrate and minimal use of nitrite in the extraction feed can give an easily controlled Pu(III) column. The Pu(III) system not only has the advantage of an excellent column profile (variation of Pu concentration with column height), but it avoids Pu polymer precipitation (possible in low-acid mediums like the CC Column) and $\text{Pu}(\text{NO}_3)_4 \cdot 2\text{DBP}$ precipitation in the middle of the stripping column.
2. A flowsheet for Pu-U scrap processing has been developed that can avoid additional Pu and U cycles. The extra Pu cycles can be avoided by the reflux flowsheet principle, which gives a concentrated, pure Pu product. Many solvent extraction systems are once-through systems (*i.e.*, no refluxing) with several Pu and U cycles. Such systems demand high nitric acid concentrations for partitioning; hence an impurity-laden (iron), powerful reducing agent like ferrous sulfamate is required. In once-through systems, the Pu product from the partitioning column will frequently be low in Pu (especially in a Pu-U scrap processing facility with its variable Pu content in the scrap), and metallic impurities will be excessive. At least one more complete cycle is required--and

probably two--to attain required purity level if the once-through principle is maintained in the additional cycles.

Further U cycles may be avoided by use of the HF strip column. The PRF uses an existing column, and the facility layout was such that column dimensions could not be economically altered to meet U specifications (10 parts Pu/billion parts U). A new facility can avoid this problem.

3. Geometrically-favorable equipment is required with the reflux flowsheet system. However, geometrically-favorable systems are usually selected for final plutonium purification with any flowsheet. Geometrically-favorable means nuclear safe by geometry (usually minimum diameter, annulus, or slab thickness) for the worst foreseeable abnormal occurrence in the facility.
4. An adaption of the proposed flowsheet might be useful in power reactor fuel reprocessing, especially where breeder reactors are concerned (high Pu concentrations, *e.g.*, 20 percent Pu). A new facility could conceivably process 500 kg/day of U plus Pu if limited to a cylindrical geometrically-favorable facility.
5. Plutonium can be removed from the organic degradation product DBP with dilute hydrofluoric acid-nitric acid mixtures. Other reducing agents (like ferrous sulfamate) are unsatisfactory for this purpose.

TABLE OF NOMENCLATURE

ARHCO	Atlantic Richfield Hanford Company.
PRF	Plutonium Reclamation Facility.
Geometrically-Favorable Facility	A facility processing fissionable material that is safe from criticality by equipment design.
TBP	Tributyl phosphate, the extractant used in this solvent extraction process.
CCl ₄	Carbon tetrachloride, the diluent used for the TBP.
CA Column	The PRF sieve plate, air-pulsed, continuous extraction column. This column co-extracts the plutonium and uranium and scrubs the impurities from the plutonium and uranium in the organic stream.
CAF	The aqueous feed to the CA Column.
CAX	The organic stream used in the CA Column. It contains 20 percent of the TBP extractant in the CCl ₄ diluent. The organic phase is the continuous and the heavy phase.
CAIS	The refluxed plutonium product stream. This stream gives extra volume velocity to the scrub section, improving the plutonium-uranium cleanup efficiency.
CAS	A nitric acid scrub at the bottom of the CA Column. For most feeds, a 2M HNO ₃ is used. With refluxing product [CAIS], the CAS is able to recontact the plutonium product for purification many times before the plutonium product is removed from the system.
CAP	The organic effluent from the CA Column containing all the co-extracted plutonium and uranium. The plutonium

concentration will normally be higher than the uranium, since plutonium is refluxed.

I	Interface between the two phases. The interface will either be in the bottom or top disengaging section, depending upon which is the continuous phase; <i>e.g.</i> , organic phase--I at top; aqueous phase--I at bottom.
CC Column	The combination plutonium stripping-partitioning column. It is a sieve plate, air-pulsed column.
CCS	The organic scrub which removes the uranium from the plutonium product in the CC Column.
CCX	The initial CC Column strip, 0.2 to 0.5M HN and 0.15M HNO ₃ , added at the bottom of the column.
CCXF	The second CC Column strip, 1.2M HM-1.0M HNO ₃ , added about two stages below the CAP entry point.
CCW	The CC Column organic effluent containing all the uranium and usually less than 0.1 g/liter plutonium.
CCP	The final purified plutonium product.
CO Column	The column for removing the final plutonium from the uranium-laden organic.
COX	The hydrofluoric acid strip for the CO Column, 0.25M HF-2.5M HNO ₃ .
COW	The CO Column organic effluent. This stream goes to conventional uranium and organic handling equipment; <i>i.e.</i> , a packed water strip column for removing nitric and uranium from the organic and a column for removing such degradation products as DBP from the organic by a carbonate strip.
COP	The aqueous effluent from the CO Column which contains the remaining plutonium

and 10 percent of the uranium entering the CO Column via the CCW. The COP flows to the extraction feed system for rework.

HN	Hydroxylamine nitrate.
DBP	Dibutyl phosphate, a degradation product of TBP.
HF	Hydrofluoric acid.
Pu(III)	The plutonium 3 valence state.
Pu(IV)	The plutonium 4 valence state.
Pu(VI)	The plutonium 6 valence state.
U	Uranium
Pu	Plutonium.
g/liter	Grams per liter.

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