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HW-60116

**CHEMISTRY — SEPARATION PROCESSES FOR
PLUTONIUM AND URANIUM**

DESCRIPTION OF PUREX PLANT PROCESS

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May 19, 1959
[Date Declassified]

Hanford Atomic Products Operation
General Electric Company
Richland, Washington

UNITED STATES ATOMIC ENERGY COMMISSION
Technical Information Service

5118

Work performed under Contract W-31-109-Eng-52.

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DESCRIPTION OF PUREX PLANT PROCESS

I. INTRODUCTION

The Purex process is a continuous solvent extraction process for irradiated uranium for the separation and decontamination of plutonium and uranium from each other and from fission products. Much information has been documented (2,11) in the unclassified literature in regard to the process bases, chemistry, and possible flowsheets based on laboratory and pilot plant work. However, since the Purex Plant has been in production operation at the Hanford Atomic Products Operation since 1956, many revisions have been made to the process flowscheme and details. These process improvements have been the result of coordinated in-plant and laboratory development programs. The purpose of this document is to present a brief summary, with reference literature for details, of pertinent and important process flowsheet conditions (12, 19) which are in use in the Purex Plant.

II. OVER-ALL PROCESS DESCRIPTION

The Purex Plant process can be divided into seven major steps or unit operations:

1. irradiated uranium dissolution (including jacket removal) and feed preparation;
2. gross decontamination and recovery of uranium and plutonium from fission products;
3. partitioning of the uranium and plutonium;
4. final decontamination and recovery of plutonium (including anion exchange tail-end treatment);
5. final decontamination and recovery of uranium;
6. solvent recovery; and
7. nitric acid recovery (including waste concentration and disposal).

In addition, several auxiliary steps are required for the coordinated operation of the plant.

Initially, the solvent extraction portion of the plant consisted of: a) a decontamination cycle for gross decontamination of plutonium and uranium from fission products; b) a partition cycle for further decontamination from fission products and separation of plutonium and uranium from each other; and c) final decontamination cycles for both plutonium and uranium. Final products were concentrated by boil-down to concentrated nitrate solutions

for further processing. Wastes from each cycle were concentrated; nitric acid was recovered by distillation for re-use in the process, and the wastes were neutralized and stored. Used solvent was chemically washed and recycled to the process.

As discussed in Reference 11, the above flowscheme is only one of several possible arrangements of the Purex process. A major improvement in the processing scheme has been incorporated in the Purex Plant by conversion from a three-cycle to a two-cycle solvent extraction flowsheet employing the back-cycling of waste (2). Figures 1, 2, and 3 show the details of the over-all process flowsheet, and Table I summarizes the design criteria for the solvent extraction columns (6, 12). Over-all process performance with these flowsheets and process equipment has been demonstrated on a plant scale as follows:

Separation of U from Pu	$> 10^7$
Separation of Pu from U	10^6
Decontamination of F.Ps from Pu	$> 10^8$
First Cycle DF	$= 2 \times 10^4$
Second Cycle DF	$= 2 \times 10^2$
Anion Exchange DF	$= > 3$
Decontamination of F.Ps from U	10^7
First Cycle DF	$= 2 \times 10^4$
Second Cycle DF	$= 5 \times 10^2$
Plutonium & Uranium Recovery	99.9%
Acid Recovery - Dissolving	70%
- Solvent Extraction	95%
Solvent Recovery	99.7%

The major items contributing to the successful transition from the three-cycle to the two-cycle flowsheet include:

1. improved pulse column technology;
2. improved solvent treatment methods;
3. new technology for anion exchange purification of plutonium;
and
4. increased process "know-how".

In the following sections each of the major unit operations will be described briefly in conjunction with reference to appropriate flowsheet Figures 1, 2, and 3. Notes on these figures define the flow bases, temperatures and other process conditions.

III. UNIT OPERATIONS DESCRIPTIONS

A. Uranium Dissolution and Feed Preparation

The uranium dissolving operation is a two-step batch process: coating removal and slug dissolution. Aluminum-canned, irradiated uranium slugs are charged into a dissolving vessel on top of an equal weight of bare uranium slugs (a "heel" left from the prior operation) which are all covered with a sodium nitrate solution. In the dissolution of the aluminum jackets, concentrated sodium hydroxide is added to the dissolver slowly, limited only by the reaction rate. In the presence of nitrate ion, hydrogen formed by the reaction of aluminum and hydroxide ion is converted to ammonia gas which is diluted with air and discharged to the off-gas system. At the end of the coating-removal operation, usually after two to three hours digestion, the solution is jetted from the dissolver; water rinse of the dissolver is made to remove residual chemicals. The resulting coating-waste solution is stored indefinitely in large mild-steel underground tanks (15).

The uranium charge is normally dissolved in two steps. Concentrated nitric acid is next added to the dissolver vessel in a ratio of approximately 3.5 moles of acid per mole of uranium to be dissolved. The solution is heated to boiling, and dissolving occurs for three to six hours until the solution specific gravity reads 1.75 at the boiling point. At this time the solution, still containing free acid, is cooled to prevent further acidity reduction (which would permit polymerization of plutonium to an inextractable form) and jetted from the dissolver to another tank preparatory to further processing. The process is repeated to remove a second batch of solution, leaving a residual "heel" of uranium. Finally, the dissolver is rinsed with water to minimize loss of plutonium and uranium in the subsequent coating-removal operation.

During the uranium dissolution process, the oxides of nitrogen are passed through a water-cooled, downdraft reflux condenser which returns significant acid to the dissolver. Unreacted oxides leave the condenser, are heated to 190°C, and pass through a silver reactor (17) for removal of iodine, are filtered through a glass fiber filter (17), and are discharged to a standard nitrogen oxide recovery unit. Approximately seventy percent of the acid is recovered. All equipment prior to the final acid absorbers is located in the remotely-maintained and operated canyon building (3, 8).

The dissolver solution containing plutonium, uranium, and fission products is next centrifuged (1,000 G's with a ten-minute hold-up) to remove siliceous solids from the solution, primarily aluminum-silicon compounds, from the slug-jacket bond. If radio-iodine suppression is needed, 5×10^{-4} M mercuric nitrate is added to minimize the evolution of iodine during centrifugation and further processing. All process vessels (excluding dissolvers) are vented through a common silver reactor for removal of radio-iodine and filtered through a common glass fiber filter prior to discharge to the atmosphere.

Following centrifugation, the solution is adjusted to the proper uranium and acid concentration for solvent extraction. Normally, no plutonium valence adjustment with sodium nitrite is necessary because of the presence of adequate nitrite ion resulting from radiolysis of nitrate ion in the dissolver solution.

B. Gross Decontamination from Fission Products

With reference to Figures 1 and 2, and solvent extraction process descriptions elsewhere (2, 11), stepwise descriptions of the solvent extraction flowsheets are unnecessary. However, major facets will be mentioned. In regard to the HA-HS Columns, essentially the HS Column is an extension of the HA Column scrub section, but being separate, it may be operated at 70°C with the HA Column operated at 35°C for maximum decontamination as discussed in Reference 2. This procedure has not been adopted in the Purex Plant, however, because all solvent solutions are maintained below 55°C for safety reasons with existing equipment and solvent.

The HAO stream is a solvent stream consisting of backcycled IBSU (returning fission products removed in the IBS Column) and HOO recycled solvent used to minimize uranium reflux in the HA and HS Columns. Sodium nitrite may be added to oxidize any entrained ferrous ion from the IBS Column, but this is not usually necessary.

The 3WB stream, as shown in Figure 3, is a concentrated solution of second cycle wastes being backcycled (2) for recovery of plutonium and uranium losses from these cycles. These waste acid solutions are steam-stripped through six bubble-cap trays for removal of entrained solvent prior to concentration. Most of the nitric acid entering the HA Column enters via this solution.

Both the HA and HS Columns have special cartridges defined in Table I and are operated with the solvent phase continuous. Thus, the aqueous-organic interfaces are located at the bottom ends of the columns; highly radioactive solids which accumulate on these interfaces are entrained with the aqueous phases leaving the columns and thus do not follow the product-bearing solvent phases leaving the columns. The combination of the special cartridges and the bottom-interface locations resulted in a 5- to 10-fold improvement in fission product decontamination compared with top-interface locations.

Operation of the columns with bottom interfaces was dependent on two major developments. Satisfactory plastic plates with adequate radiation stability (integrated exposure greater than 2×10^9 R) were made from linear polyethylene. Satisfactory interface control (under pulsed column and slightly emulsified conditions) was achieved by use of a float-type interface detector (7) for remote locations.

C. Partitioning of Plutonium and Uranium

Two columns, IBX and IBS, are utilized for partitioning of plutonium and uranium. Use of two short columns rather than one long column permits easier installation of the columns in canyon cells. Also, such a flow-scheme permits the IBSU backcycle which provides additional fission product decontamination for both uranium and plutonium.

D. Final Plutonium Decontamination

As shown in Figure 2, the plutonium is further decontaminated from fission products by solvent extraction in the 2A and 2B Columns and from both fission products and uranium by an anion exchange purification step (16). The 2A Column is operated with the solvent phase continuous.

All equipment downstream of the partitioning step is designed to be geometrically safe for plutonium solutions. This feature is important from the standpoint of preventing a criticality excursion when processing quite concentrated plutonium solutions. Two other concepts are used for critical mass control elsewhere in the plant: (a) batch-size control where feasible, and (b) safe solution concentration elsewhere. The primary critical mass control feature, however, is maintenance of chemical conditions (1) to prevent the precipitation and accumulation of a critical mass of plutonium. In the acidic Purex process, the problem exists primarily only where plutonium concentrations become high.

Solvent for the Final Plutonium Cycle is shown to be from the No. 2 Solvent System, the low-activity solvent used also in the Final Uranium Cycle, for maximum fission product decontamination. If the No. 1 Solvent System, the high-activity solvent used in the First Cycle, provides adequate decontamination of the solvent from fission products, 2AX can be supplied from this system; such has proved to be possible in the plant when the alkaline-permanganate solvent washing process (described later) is used. In this way the 2BW can be backcycled to IBXF, but caution must be used to prevent routing of excessive nitrite ion (very extractable) to the IBXF and oxidizing the IBX Column solutions.

The anion exchange process is performed in Higgins (9, 10) continuous anion exchange columns using Permutit SK resin (20 - 40 mesh). A 0.045 M H_2SO_4 solution is used in 2BX in order to complex Pu IV to permit a low 2BP flow; thus, the XAF flow rate is maintained at a minimum to maximize the hold-up time in the XA Column for anion resin adsorption, kinetics of adsorption being the controlling factor. Following elution of the plutonium from the XC Column, the solution is concentrated in a titanium concentrator to minimize introduction of impurities from corrosion; tantalum would also be suitable for this purpose.

E. Final Uranium Decontamination

As shown in Figure 2, the ICU stream is first steam stripped (through six bubble-cap trays) for removal of entrained solvent and then concentrated.

(Steam pressure is limited to a maximum of 30 psig for safety reasons.) Ferrous sulfamate is added to the 2DF to reduce trace amounts of plutonium so that the plutonium will not be extracted in the 2D Column. The 2D Column is operated with a high degree of saturation, resulting in approximately five percent of the uranium in the 2D Column feed being backcycled to 3WB, in order to maximize fission product decontamination. The 2EU is concentrated in equipment identical to that used for ICU and 3WB and transferred to a storage tank for further processing by calcination to the oxide.

The 2D Column is operated with the solvent phase continuous and a bottom interface similar to the HA Column. A twenty-fold increase in fission product decontamination was effected by the conversion of this column from aqueous to solvent phase continuous even though the "solvent extraction" decontamination factor should not have been different. This significant improvement resulted because decontamination in this column is limited by particulate rather than soluble fission products (primarily Zr-Nb).

The plastic plates in the 2D Column are fluorothene because they were installed a year before the new linear polyethylene plates used in the HA Column. The fluorothene plates have adequate radiation resistance (10^8 R) for use in this low-activity field.

F. Solvent Recovery

Two solvent systems, low-activity (No. 2) and high-activity (No. 1) systems, are used in the Purex Plant. The low-activity system simply employs a dilute sodium carbonate wash of the solvent in the 2D Column, followed by centrifugation or decanting for removal of solids and aqueous solution. Such a procedure normally maintains solvent of adequate quality. Since approximately 0.3 percent of the solvent processed in the plant is lost by chemical reaction, entrainment, and volatilization, fresh solvent is occasionally added to the No. 2 system, and solvent from the No. 2 system is then added to the No. 1 system for make-up solvent.

With the much more severe conditions of exposure of the No. 1 system solvent, a more comprehensive washing procedure is used as shown in Figure 3. The washing consists of good contacting of the solvent with an alkaline-permanganate solution first, where the permanganate is reduced to manganese dioxide, a good scavenger for diluent degradation products and particulates. The solvent is next washed with dilute acid for removal of residual manganese dioxide and finally washed with dilute sodium carbonate alone.

The contactors for all three operations in the plant are different because of the presence of original equipment in the plant. However, all three contacts could be made in identical equipment achieving adequate contacting and separation of 15 to 30 minutes. No centrifugation is required with the above process.

Chemical solutions used for solvent washing are combined with the neutralized IWW from solvent extraction processing, concentrated, and stored in large underground mild-steel tanks. Infrequently, gross amounts of solvent have been discarded to separate storage areas because of contamination with grease or paint resulting from leaks out of process equipment. No solvent which has remained in routine process use has required discarding.

G. Nitric Acid Recovery and Waste Disposal

As shown in Figure 3, the only acid waste from solvent extraction is HAW. Nitric acid is recovered from this waste by a double-distillation, absorption, and fractionation according to the flowsheet specified. No steam-stripping of this acid is performed; the entrained solvent is decanted and returned to process, and the dissolved solvent is distilled or hydrolyzed. The acid product recovered from these operations has been decontaminated from fission products by a factor of 1×10^6 and is re-used in the process wherever possible without introducing excessive fission products. No problem of ruthenium volatilization has resulted from the acid recovery process.

The IWW waste solution containing essentially all of the fission products is neutralized with sodium hydroxide and stored in large, underground mild-steel (SAE-1020) tanks under boiling conditions as described in Reference 15. The concentration of these wastes must be maintained below 8 M sodium ion to prevent precipitation of sodium salts. The precipitates (iron salts primarily) in the tank scavenge most of the Zr-Nb, Ce, and Sr fission products leaving Cs and Ru in the supernate. Because of this tendency for concentration of the high heat-generating Zr-Nb and Ce fission products in the solids, large air-lift recirculation tubes are used to turn the solution in the tanks over at rates of several thousand gallons per minute to alleviate a tendency for localized over-heating, which would result in uneven boiling. The vapors from the tank are condensed, and most of the condensate is returned to the tank. The remainder of this condensate is returned to the plant and used for acid distillation water prior to disposal to the ground via caverns (15).

Use of the formaldehyde process in the Purex Plant is contemplated for denitration of nitric acid in IWW (5, 14). This process has many economic advantages in addition to its main justification of reducing the solids content (six-fold) and volume (three-fold) of high-level wastes for ultimate storage.

IV. SPECIAL FEATURES

A. Process Control

Many of the process control techniques and devices used in the plant are standard for chemical plants. However, special equipment is used for

sampling the radioactive solutions, and special handling is required for analyses. Several in-line analytical instruments (18) are used for continuous measurement of pH, plutonium and uranium concentrations, and gamma activities of solutions. These instruments provide much assistance for detecting off-standard conditions at an early time in their development and for aiding in the understanding of the process during normal and test operations.

Continuous flow streams are generated either by pumps (4) or gravity-flow, and flow measurement and control (4) are achieved by remotely-operated rotameters and diaphragm-operated valves, respectively.

The evaluation of the allowable departures from flowsheet and the sensitiveness of process variables is largely dependent upon which type of flowsheet is being evaluated. For example, a flowsheet employing extensive waste backcycle can be extremely insensitive to deviations from flowsheet as compared to a process involving no backcycle. Sensitiveness of process variables is also dependent upon specific details, such as: 1) interface position, 2) cartridge design, 3) critical mass considerations, 4) product specifications, 5) how close the plant is being pushed to capacity, 6) frequency of analytical checks for off-standard conditions, 7) degree of technical process supervision, 8) the existence of secondary indications of off-standard conditions, and 9) rate of process response or turnover.

In general, instrumentation and analytical techniques should be capable of two percent precision. An equally high degree of accuracy is not generally necessary, except in isolated cases such as nuclear materials accountability. Operating and process control personnel rapidly develop a "feel" for the various biases in the data.

HAPCO experience has shown that ± 5 percent variation in composition or flow rate of most streams can be tolerated. However, certain key streams should be controlled more closely. For example, in the uranium extraction columns, close control of solvent uranium saturation is required to optimize decontamination without incurring excessive plutonium losses. Temperatures should be controlled with a precision of $\pm 2^\circ\text{C}$ as wider fluctuations can result in column upsets due to changes in phase behavior.

A thorough evaluation of allowable flowsheet departures cannot be made sufficiently general to be realistic, and such an evaluation should be made specifically for the proposed flowsheet with consideration given to the points outlined above.

B. Waste Rework

Off-standard aqueous streams are reworked occasionally to recover uranium or plutonium. Such streams usually consist of concentrated acid wastes, solvent recovery wastes, cell drainage, flushes and spill solutions. The procedure for handling all aqueous streams is generally the same. The

rework stream is acidified, if necessary, to 4 - 7 M nitric acid and then boiled or refluxed until all TBP and its degradation products are hydrolyzed to phosphoric acid and the diluent boiled off. The time required to destroy TBP depends on temperature, the concentration of nitric acid, salts present, and on the TBP concentration. Unfortunately, due to difficulties involved in measuring TBP concentrations in waste streams, no fixed formula for reflux time has been obtained. Rather, a trial method is used to determine completion of the hydrolysis. A small portion of the rework solution is blended into the First Cycle feed at volume ratios of about 1 to 20, and the recovery efficiency, decontamination performance, and stability of the first extraction column is observed. If the batch is processed satisfactorily, the remainder of the rework is blended off in the same manner. However, if the trial is not successful, the rework solution is refluxed for a longer period. To date, concentrated acid wastes which contain a minimum of entrained TBP and which have boiled more than 50 hours have been processed successfully by blending into the feed for First Cycle feed make-up solution. Other aqueous rework solutions mentioned above have required 12 to 22 days of refluxing before they could be processed. In refluxing rework material, as in concentrating any Purex solution, care must be taken not to exceed 135°C (the de-nitration temperature of "Red Oil").

Recently processes have been developed to rework aqueous waste solutions, particularly concentrated acid waste, through anion exchange resins for the recovery of plutonium. Several problems remain to be resolved in this process, one of which is solids removal. The composition of concentrated acid waste is being studied, and the ion exchange method of recovery appears promising. One item of interest learned from plant experience is that a solid iron salt of dibutylphosphate is formed in concentrated acid wastes which has a tendency to coagulate and plug lines. This salt is slurryable in dilute (5 weight percent) sodium hydroxide.

The processing of off-standard organic solution, notably large volumes of spilled organic which have dissolved paint and grease from cell floors, has not been always successful. Amercoat paint used at Hanford acts as an emulsifying agent and upsets the extraction columns so that blending is not possible. If the organic contains recoverable product and the volume is such that it cannot be conveniently distilled and hydrolyzed, then batch contacting is used. Usually carbonate or caustic solutions will adequately strip the product from the solvent so that the stripped solvent can be discarded. The stripped solution is then treated as off-standard aqueous rework solution.

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TABLE 1

SOLVENT EXTRACTION COLUMN DATA

Column		Plate or Packed		Cartridge Details					Louver		Pulser		Phase	
		Section		Plate Type	Plate Mat'l.	Hole Size in. Dia.	Free Area %	Plate Spacing in.	Plates		Ampl., in.	Freq. Range c/m	Contin-uous	Pulsed
		Height, ft.	I.D. in.						No.	Free Area %				
HA	Ex.	13.9	24	Nozzle	ss	3/16	23	2	4	23	1.1	35-110	Org.	Org.
	Scrub	19.2	32	Sieve	(a)	(a)	(a)	1	4	16	0.6			
2D	Ex.	14.0	24	Nozzle	ss	3/16	23	2	4	23	1.1	35-110	Org.	Org.
	Scrub	13.2	32	Sieve	(b)	(b)	(b)	1	3	16	0.6			
HS		18.6	34	Sieve	(a)	(a)	(a)	1	4	16	0.53	35-110	Org.	Aq.
IC, 2E		18.0	34	Nozzle	ss	1/8	10	4	--	--	0.53	35-110	Aq.	Aq.
IBX		28.0	27	Sieve	ss	3/16	33	4	6	16.5	0.84	35-110	Aq.	Org.
IBS		13.3	8	Sieve	ss	1/8	23	2	6	20	0.84	25-110	Aq.	Aq.
2A	Ex.	20.9	7	(c)	(c)	--	(c)	(c)	--	--	1.1	25-110	Org.	Aq.
	Scrub	9.8												
2B		21.0	7	Sieve	ss	1/8	23	2	--	--	1.1	25-110	Aq.	Aq.
IO, 20		26.3	34	Sieve	Fluoro- thene	3/16	23	4	--	--	0.53	35-110	Org.	Aq.

(a) Cartridge composed of 21% free area stainless steel (ss) plates and 23% free area linear polyethylene plates, grouped alternately four stainless steel plates and two polyethylene plates. Hole diameters in stainless steel plates = 0.085 in.; polyethylene plates = 0.1875 in.

(b) Cartridge composed of alternate pairs 21% free area stainless steel plates and 23% fluorothene plates. Hole diameters in stainless steel plates = 0.085 in.; fluorothene plates = 0.1875 in.

(c) Packed with 1 in. fluorothene Raschig rings.

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FIGURE 1
PUREX TWO-CYCLE
FLOWSHEET

IODINE BACKUP
FEED PREPARATION
FIRST DECONTAMINATION
AND PARTITION CYCLE

NOTES:

- All flows are relative to 100 flows equals 747 gallons/ton uranium.
- All temperatures are ambient except as otherwise noted.
- Specific gravities are given for all solutions at 25°C, regardless of operating temperature.
- Solvent refers to 30 ± 1 vol. per cent TBP in a hydrocarbon diluent of specific gravity equal to 0.801.
- Plutonium concentrations are based on uranium irradiated to g Pu/ton U.
- Anion exchange resin is Permutit SK, 20 - 40 mesh. Flow volumes of resin are for wet settled resin in the nitrate form.
- Process control allows ± 5% deviation on all specifications except TBP (See note 4).

FIGURE 2
PUREX TWO-CYCLE
FLOWSHEET

FINAL URANIUM CYCLE
FINAL PLUTONIUM CYCLE
PLUTONIUM ION EXCHANGE

WASTE CONCENTRATION
ACID RECOVERY
BACKCYCLE CONCENTRATION
ORGANIC RECOVERY