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## CONTINUOUS ANION EXCHANGE PROCESSING OF PLUTONIUM\*

G. C. Oberg and W. H. Swift
October 3, 1961

## HANFORD ATOMIC PRODUCTS OPERATION

RICHLAND, WASHINGTON



\*Prepared for presentation American Institute of Chemical Engineers. Los Angeles National Meeting, Ion Exchange Symposium, February 4-7, 1962

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## Abstract

The Hanford continuous anion exchange process for purification and concentration of plutonium nitrate solutions is discussed. Information is given on the unique process chemistry and aspects peculiar to plutonium processing. The continuous contactor design and operating experience is reported.

#### CONTINUOUS ANION EXCHANGE PROCESSING OF PLUTONIUM

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### Introduction

Throughout the United States, Canada and Europe, various ion exchance processes have been developed and adopted for the final purification and concentration of plutonium nitrate solutions. These processes have been applied to the plutonium product streams from primary reactor fuel processing plants at both the Savannah River and Hanford Purex Plants. In addition, ion exchange processes have been applied to the aqueous reprocessing of plutonium metallurgical scrap.

The first major process application was the tri-valent plutonium cation exchange technique developed at Oak Ridge National Laboratory, Hanford, and the Knolls Atomic Power Laboratory. This system is employed as a conventional fixed-bed process at the Savannah River Plant (1) (2) The primary advantages of the cation exchange process are its adaptability to processing very dilute plutonium solutions and its ability to produce a highly concentrated plutonium product.

At the Hanford Purex Plant, evaporative concentration of plutonium was employed initially. However, difficulties were encountered with corrosion, formation of plutonium (IV) nitrate polymer and high product acidity. More recently, the quadravalent plutonium anion exchange process has been extensively developed and applied at Hanford and Savannah River. In the latter case, the application is again in conventional fixed beds and applied to processing of metallurgical wastes.

<sup>(1)</sup> Tober, F.W., "Concentration and Purification of Uranium and Plutonium and Neptunium and by Ion Exchange in Nuclearly Safe Equipment", Proc. 2nd Inter. Conf. Peaceful Uses of Atomic Energy, Geneva, 17, P/520 pp 574-85, 1958.

<sup>(2)</sup> Orth, D. A., E. L. Field, and J. H. Rodke, Ind. Eng. Chem 53, 685 (1961)

The subject of this paper is the application of anion exchange to the processing of the Hanford Purex Plant plutonium product wherein the scale of operation demanded the use of continuous ion exchange contacting equipment. The primary function of the continuous anion exchange process at Hanford is the decontamination of plutonium from last traces of fission products and other undesirable metallic ions. Decontamination factors for zirconium-niobium-95 ranging up to ten are required to meet product specifications. This is not only being met but is exceeded in the operation of the present continuous anion exchange unit. In addition, a tenfold increase in plutonium concentration is attained in the process. At this writing, because of the success of the operating unit, a second continuous anion exchange unit is being installed at Hanford in the Redox Plant.

### Process Chemistry

The basic process chemistry of the plutonium anion exchange process has been extensively described by Ryan and Wheelwright(3)(4). The more important process aspects are briefly reviewed here:

#### A. Equilibrium Considerations

Plutonium is one of the few elements that form anionic nitrate complexes and can readily be absorbed on anion exchange resin from nitrate solutions. Figure 1, (Distribution Coefficient versus Nitrate Molarity) illustrates this absorption behavior by plotting the distribution coefficient ( $K_d$ ) for plutonium versus the nitrate molarity for a typical anion exchange resin. In nitric acid systems, the distribution coefficient increases with increasing nitrate concentration up to about 7.3  $\underline{M}$  HNO3 and then decreases gradually at higher acidities. Temperature also affects the distribution with lower coefficients

<sup>(3)</sup> Ryan, J. L., E. J. Wheelwright, Ind. Eng. Chem. 51, 60 (1959)
(4) Ryan, J. L., E. J. Wheelwright, USAEC Declassified Report, HW-55893, January 2, 1959.

being obtained at higher temperatures. It is believed that plutonium is absorbed as the  $Pu(NO_3)_6^-$  complex and, as the nitric acid concentration is raised from 1 M to 7.3 M the increasing nitrate activity promotes formation of  $Pu(NO_3)_6^-$ . Above about 7.5 M rapidly increasing hydrogen ion activity promotes the formation of the acidic complexes  $HPu(NO_3)_6^-$  and  $H_2Pu(NO_3)_6$ . At low nitric acid concentrations, i.e. below 1 M the equilibrium is reversed and plutonium can be readily eluted from anion exchange resins. These two equilibrium aspects form the basis for the plutonium anion exchange process.

## B. Kinetic Considerations

Kinetic factors are also of importance in the process as can be seen in Figure 2 (Plutonium Absorption Rates on Dowex-1 X-4). Here plutonium absorption on Dowex-1 is shown as a function of time and temperature. As shown by shallow bed experiments, increased temperature markedly improves the rate of plutonium absorption; a loading of 200 mg of plutonium per gram of resin requires 300 minutes at 25°C and only 25 minutes at 70°C. Thus, the less favorable equilibrium at high temperatures is overcome by improved kinetics, and in fact, it was this observation that made feasible the concept of Pu anion exchange process. Figure 3 (50% Break Through Capacity for Deep Bed versus Temperature) data taken on deep static beds indicate that process-wise the optimum temperature i.e. maximum plutonium absorption occurs at temperatures between 60 and 70°C. Increasing absorption rates achieved with increased temperature of operation are overcome by the decreasing distribution coefficients. Nevertheless, because of the highly favorable equilibrium and strong kinetic effects even at elevated temperatures, kinetic properties are process controlling.

## C. Choice of Resin

The system properties discussed above are of general applicability. Resin type is another variable with considerable impact on process capacity. Figure 4 (Loading versus Time for Various Cross Linkages) indicates the effect of resin cross linkage on plutonium absorption. The more porous resin absorbs plutonium much more rapidly.

From an engineering standpoint, it is desirable to use a resin of as large a particle size as possible and for this reason the absorption characteristics of several commercial resins were examined in shallow bed experiments. Figure 5 (Absorption Rates of Commercial Resins) shows kinetic data for several resins of varying mesh size, cross-linkage, and functional exchange site nature. The loading rates at 60°C indicate that Dowex-1 X4 (50 to 100 mesh) is superior. Permutit SK (20 to 50 mesh) is, however, sufficiently fast for process purposes to permit taking advantage of its larger mesh size, the latter desirable for continuous contactor use.

Radiolytic and chemical damage to the ion exchange resin must be considered in the choice of resin. Plutonium as an alpha emitter is not generally considered as a radioisotope capable of radiation damage to materials. Radiation damage is normally associated with beta, gamma, and neutron radiation rather than with short range alpha particles. However, in the case of ion exchange resin loaded with plutonium to the extent of 50 grams plutonium per liter of resin, and consequently under conditions of intimate contact of the resin with the radiation, the alpha radiation is absorbed and the dose rate is on the order of 10<sup>5</sup> roentgens per hour. Since most elastomeric organic materials begin to show considerable radiation damage at levels of 10<sup>8</sup> roentgens, the ability of various resins to withstand damage is of considerable process importance. Radiation damage can affect both exchange capacity and the effective cross-linkage percentage. Of the resins examined, Permutit SK was found most resistant to damage by radiation (3). When irradiated to 3.4 x 10<sup>8</sup> roentgens, exchange capacity decreased 35 percent but physical damage i.e. cross-linkage reduction was small.

Strange nitric acid solution at 60°C is not a desirable environment for organic materials such as ion exchange resins. This is particularly true in static, non-flow systems where the oxidation of organic material by nitric acid results in the generation of nitrate ion, further accelerating the attack. However, in continuously flowing systems, the oxidation products are continuously removed and the stabilities of commercial resins are adequate for process use.

## Equipment and Operation

The Hanford plutonium ion exchange unit which is patterned after the Higgins continuous unit, consists of absorption (XA) and elution (XC) columns connected by a downcomer which permits flow from the top of the absorption column to the bottom of the elution column as shown in Figure 6 (Continuous Plutonium Anion Exchange Flow Diagram). Connected to the top of the elution column is a resin receiver which drains into the resin reservoir directly below. The bottom of the reservoir is connected to the bottom of the absorption column. Large two-way plug valves are located at the bottom of the absorption and elution columns with a three-way valve connecting the top of the elution column and the resin receiver to the resin reservoir. A push tank with its connecting air line joins the resin reservoir and supplies motive power for the resin movement. Connections for the introduction of liquids are located at the middle of the extraction column (XAF) and on top of both the absorption (XAS) and elution (XCX) columns. Aqueous waste streams exit the bottom of the absorption (XAW) and elution (XSW) column and the product stream (XCP) leaves from the bottom of the elution column.

Both the absorption and the elution columns are maintained completely full of resin with the reservoir about three-quarters full. With the large plug valves open, resin flow is accomplished by supplying air pressure to the surface of the liquid in the push tank which moves the liquid down through the reservoir, up through the Unclassified -9- HW-SA-2290

absorption column, down the downcomer, up through the elution column, and into the resin receiver. Movement of this liquid carries with it a quantity of resin, thus the resin is intermittently moved around the circuit whenever the liquid is "pushed". Upon completion of the "push", the large plug valves close to isolate the columns and aqueous streams start to flow into and out of the column.

The feed stream consists of the plutonium product stream from solvent extraction equipment, made 6 M in nitric acid and heated to 70°C before entering the feed tank. This feed solution is pumped into the absorption column above the midpoint by a remote diaphragm metering pump. During its downward travel, plutonium in the solution is absorbed on the resin. A 6 M nitric acid scrub stream is metered into the top of the absorption column by a diaphragm pump and washes occluded fission products and other foreign ions down the column toward the waste end. The waste stream exits the bottom of the column and is backcycled to the solvent extraction process for recovery of any plutonium it may contain. One molar nitric acid is pumped into the top of the elution column by a diaphragm metering pump to remove the plutonium from the resin and form the plutonium product stream which exits near the bottom of the elution column. This product stream is concentrated in a small thermo-siphon concentrator to about 250 g/l plutonium. The highly concentrated nitric acid solution which is pushed into the bottom portion of the elution column during resin movement is undesirable in the final product, and therefore is purged from the system via a waste stream exit at the bottom of the elution column about one foot lower than the product stream exit.

The valves in the installation, which are either ball or plug type, are operated by air cylinders actuated by solenoid valves in the air lines. The impulses which operate the solenoid valves are supplied by a master programmer or by local instrumentation such as conductivity probes. The overall sequence of operations is controlled by the programmer, which provides electrical switching of the various operating equipment pieces on a definite time cycle. At the start of the "push" cycle,

a number of events occur simultaneously:

- (1) The following valves close:
  - (a) Plutonium feed valve (the feed is routed back to the feed tank via a bypass valve).
  - (b) The plutonium product valve.
  - (c) The waste stream valve from the absorption column (waste stream valve from the elution column is already closed).
  - (d) The vent valve on the push tank.
  - (e) The overflow valve on the push tank.
- (2) The following valves open:
  - (a) Air supply valve to the push tank.
  - (b) The large plug valves at the bottom of the absorption and elution columns.
  - (c) The three-way plug valve aligns the exit from the top of the elution column with the resin receiver.

Immediate buildup of air pressure in the push tank starts the liquid in the push tank moving through the equipment. A conductivity probe regulates the amount of liquid pushed from the push tank by the air pressure, which in turn determines the amount of resin moved from the reservoir through the system. After the solution in the push tank has dropped to the predetermined level, the conductivity probe signals the end of the "push". At this time another series of events occur simultaneously:

- (1) The following valves close:
  - (a) The air supply valve to the push tank.
  - (b) The large plug valves at the bottom of the absorption and elution columns.
  - (c) The top of the elution column is isolated from the resin receiver by the turning of the three-way plug valve which aligns the resin receiver with the resin reservoir.

- (2) The following valves open:
  - (a) The vent valve on the air push tank.
  - (b) The overflow valve on the push tank.
  - (c) The waste valves at the bottoms of the absorption and elution columns.
  - (d) The feed valve to the absorption column.

The valves controlling the flow of scrub solution into the top of the absorption column and the elutant into the top of the elution column do not close during the push cycle. After all the highly acid solution pushed into the bottom of the elution column has flowed from the bottom of the column into the solvent extraction backcycle tank as determined by a conductivity probe, the waste valve closes and the product valve opens. Product solution flows into the concentrator until the start of the next resin "push".

Process solutions enter and exit the anion exchange columns via special distributors and collectors (Figure 6A). The stream inlets distribute the solutions over the column cross-section while the outlet streams are collected from the periphery of the columns. The screen used in each case is stainless steel, single warp, plain dutch twill. A 14 x 88 mesh is used for the two waste stream collectors while the product collector and feed inlet distributors are fabricated of 24 x 110 mesh. The larger mesh used for the waste collectors allows resin fines, formed by mechanical attrition, to exit the system with the waste and not buildup to interfere with resin "pushing".

Under normal operating conditions, the typical overall time cycle for the unit (from the start of one push to the start of the next push) is about five minutes. This includes approximately 25 seconds for resin pushing, 2-1/2 minutes for elimination of the highly acidic solution from the bottom of the elution column (XSW) and two minutes for collection of the plutonium product solution (XCP). These times are adjusted somewhat to provide optimum operation of the unit.

Until recently used resin could only be removed from the system by draining it from a valved connection at the bottom of the elution column. Thus, after the unit

was removed from service, the resin in the other equipment pieces was "pushed" into the elution column and removed. Since an equipment modification, a water jet may be used to transfer the resin from the receiver to a waste tank after each push, and thus remove the resin without interfering with the operation of the unit. New resin is added via either the resin receiver or the push tank depending on whether the inventory is being increased or replaced.

## In-Line Instrumentation

The task of maintaining proper process control during operation of the unit has been simplified by the installation of several in-line monitors. Plutonium concentration in the solvent extraction product stream is determined by a counter which measures alpha radiation from the solution. A zinc-sulfide phosphor in a fluorothene mount and protected by a one-quarter mil Teflon film is exposed to the process solution (5). Scintillations produced by alpha bombardment of the phosphor establish the plutonium concentration in the solution. Plutonium concentration in the loaded resin in the lower portion of the extraction column (just below the feed point) is measured by the attenuation of cesium-137 gamma rays through the column (6); the higher the plutonium concentration, the greater the attenuation. Initially, concentration of plutonium in the product streams was measured in the same manner using an americium-241 source shining through a fluorothene cell through which the solution flowed. This latter instrument has been removed from service because the value of the information obtained did not justify the maintenance time required to keep it in operation. An "Alpha Printer" (7) which automatically deposits

<sup>(5)</sup> Upson, U.L and Brown, P. E., "A Liquid-Contact Scintillation Alpha Counter", USAEC Report, HW-49003 (Declassified) March 1, 1959.

<sup>(6)</sup> Connally, R. E., Upson, U.L., Brown, P. E., and Brauer, F.P., "Uranium Analysis by Gamma Absorptiometer", USAEC Report, HW-54438 (Unclassified), May, 1958.

<sup>(7)</sup> Hildreth, N. T., "Design Criteria In Line Alpha Monitor", USAEC Report, HW-41525 (Unclassified) February 22, 1956

a small aliquot of solution on an aluminum tape, dries it, and counts the residue for alpha particles, is used to measure th plutonium concentration in the low-level waste solutions from the absorption and elution columns.

## Special Problems Associated with Plutonium Handling

Plutonium acts much the same as other heavy metal poisons, whenever it enters the body. However, plutonium is a radioactive poison and is much more damaging within the body than ordinary heavy metals and those radioisotopes that irradiate the body from the outside for the following reasons: (8)

- (1) Radioisotopes within the body irradiate tissue continuously until eliminated.
- (2) Plutonium remains in the body for years because it is almost impossible to increase its elimination rate from the body.
- (3) It accumulates mainly in the bones but also some in the lymph nodes, liver, spleen, and other soft tissues.
- (4) Bombardment of the bone marrow by alpha particles is thought to produce bone cancer or a blood disorder such as leukemia. The total lifetime body burden for plutonium is 0.04 microcurie (9) which is calculated to be one-tenth of the concentration believed to be relatively harmless.

Because of the small body burden, the admission of plutonium into the body by ingestion, by inhalation or by skin punctures must be prevented. About seventy-five per cent of the plutonium inhaled remains in the body, thus the maximum allowable plutonium concentration in breathing air is  $2 \times 10^{-12}$  microcuries per cc(5). Consequently, special handling procedures are required for plutonium processing and all equipment is enclosed in a sealed lucite hood that operates under a negative

<sup>(8)</sup> Schubert, Jack and Lapp, R. E., "Radiation, What It Is and How It Affects You" Viking Press, New York, 1957. Pages 110 to 131.

<sup>(9) &</sup>quot;Manual of Radiation Protection Standards" USAEC Report, HW-25457, Rev. 2 March 1, 1960

three-quarter inch water pressure. Figures 7, 8, 9, and 10 show different views of the hood with installed equipment. Air is drawn into the hood through a 24 in. x 24 in. absolute filter to prevent backflow into the work area, and the air exhausted from the hood is also filtered to prevent discharge of plutonium to the atmosphere. Equipment maintenance is performed through glove ports located in a regular pattern along the sides of the hood.

In addition to its extreme toxicity which produces unique problems in handling, plutonium is fissionable. This means that an accumulation of as little as 510 grams of plutonium (10) under the proper conditions of concentration and geometry, could result in a nuclear reaction. Critical mass control can be exercised by proper design of processing equipment which prohibits favorable geometry for nuclear reaction. Thus, the initial step in critical mass control may be accomplished by the design of nuclear "safe" equipment. Such equipment may safely contain more than 510 grams of plutonium because of geometrical shape. In the ion exchange processing equipment, the feed storage tank is a "safe" slab tank 8 feet long x 5 feet high x 2-5/8 inches inside thickness, and the columns are the maximum "safe" internal diameter of 5.3 inches. Not only must the equipment be of a "safe" size but spacing between adjacent pieces must exceed a certain critical dimension so as to prevent nuclear interactions among the various pieces of equipment containing plutonium. As a result of these imposed conditions of nuclear safety control and in-hood maintenance, the design engineer is somewhat limited in his choice and arrangement of equipment.

#### Equipment Performance

Despite the complexity of the operating equipment, the performance of the ion exchange unit has been good with about ninety per cent on-stream efficiency being attained. The mechanical difficulties which for the most part have been minor

<sup>(10)</sup> Kruesi, F. E., et al, "Critical Mass Studies of Plutonium Solutions", USAEC Report HW-24514 DEL, (Declassified) February 15, 1960

have been associated with the valves and/or air cylinders, check valves in the pumps and some of the control instrumentation.

Decontamination factors of three to five were obtained initially under normal processing conditions. This meant that the concentration of undesirable components in the product solution had been reduced by a factor of three to five when compared to the concentration in the feed solution. However, this decontamination performance was not adequate for plant needs and, although the length of the scrub section was increased from two to four feet, decontamination performance was not significantly improved. After laboratory tests indicated improvement from the addition of fluoride ion to the feed, the feed was made 0.005 M in fluoride ion by the addition of sodium fluoride and the decontamination factor was improved to the ten to twenty five range. Aluminum nitrate is also added to reduce fluoride corrosion in the stainless steel equipment. The recovery efficiency of the ion exchange unit has been highly satisfactory with only about 0.3 to 0.5 per cent of the plutonium processed lost from the absorption column and one to two per cent recycled via the slip-water stream (XSW) from the bottom of the elution column. In both cases, these streams are recycled to solvent extraction equipment for plutonium recovery.

The first resin used in the unit was Permutit SK, 20-50 mesh; however a switch to 20-40 mesh occurred when the former became unavailable from the vendor. The use of a resin size mixture (25 per cent 10-20 mesh, 75 percent 20-40 mesh) has been used recently with apparently better "pushing" success, and no decrease in absorption efficiency.

In plant practice the normal resin usage has usually been sixty to seventy days of continuous operation. Figure 11 (Stability of Permutit SK Resin) compares the appearance of new resin and that used 120 days in the plant unit. Degradation of the resin is due to chemical attack, radiolysis by plutonium alpha bombardment and mechanical attrition with the main recognizable effect being a softening of

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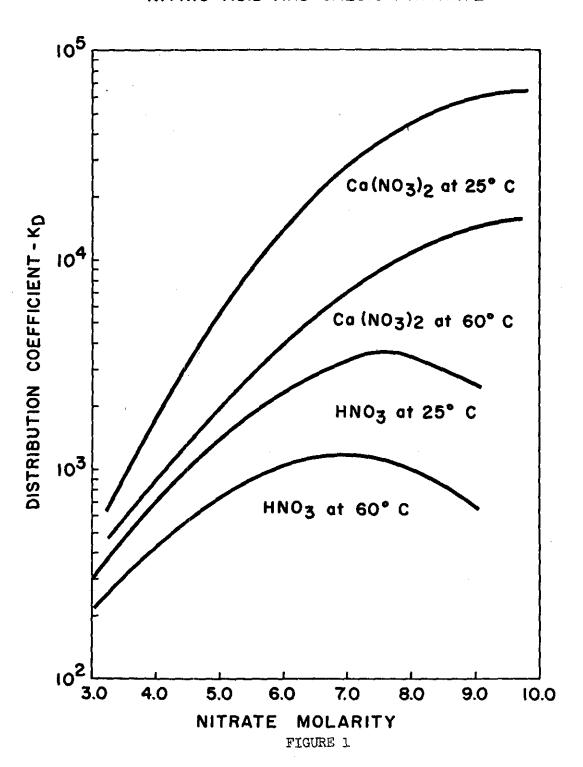
of the resin beads. Significant resin degradation is usually recognized by an increase in "push" time. Softening of the individual beads apparently produces resin packing when the push pressure is applied thus lengthening the "push" time required. If the "push" time increases as little as ten seconds, the entire resin inventory in the unit should be changed within 48 hours otherwise resin movement may stop completely.

Since it was previously necessary to shut down the unit to remove the resin, used resin was removed routinely during scheduled plan shutdown periods in order to prevent possible shutdown of the unit during an operating period. The recent equipment changes described above now allow changing the resin while the unit is operating, thus a longer useful resin life is anticipated because the resin will be operated until degradation is apparent.

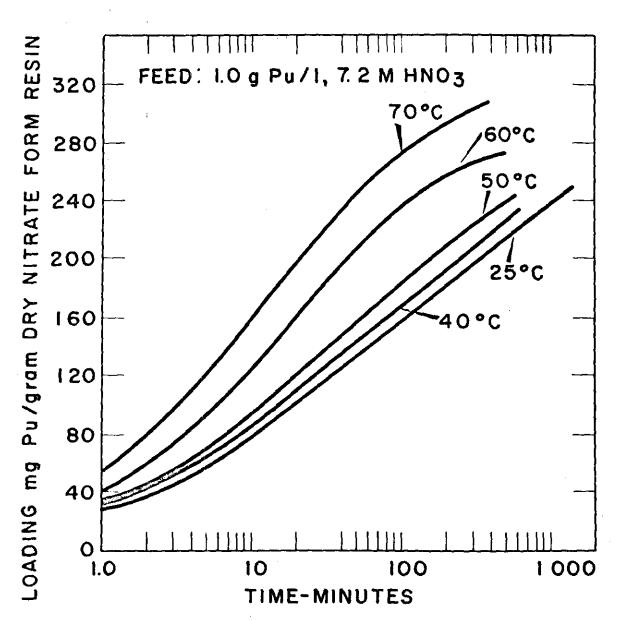
### Conclusion

In summary, the anion exchange process has proved to be a highly satisfactory method for purification and concentration of plutonium. The Higgins continuous contactor was successfully adopted to this process. Product purity from fission and corrosion products has been improved by a factor of approximately ten while producing a product of lower acid to plutonium ratio. In general, the anion exchange unit has operated with greater than 90 percent on stream efficiency and by virtue of improved plutonium product decontamination, made possible economics in the Hanford Purex Plant Operation. Subsequent improvements in process and equipment incorporated as a result of plant experience has warranted the installation of a similar unit in the Redox Plant at Hanford.

## ON DOWEX-I, X-4 (50-100 mesh) FROM NITRIC ACID AND CALCIUM NITRATE



## PLUTONIUM LOADING KINETICS OF DOWEX-I X-4 (50-100 MESH)



50% BREAKTHROUGH CAPACITY OF 45 cm COLUMNS DOWEX 1, X-4 (50-100 MESH) FROM 7.2 M HNO3, 0.55 g Pu/1 FEED

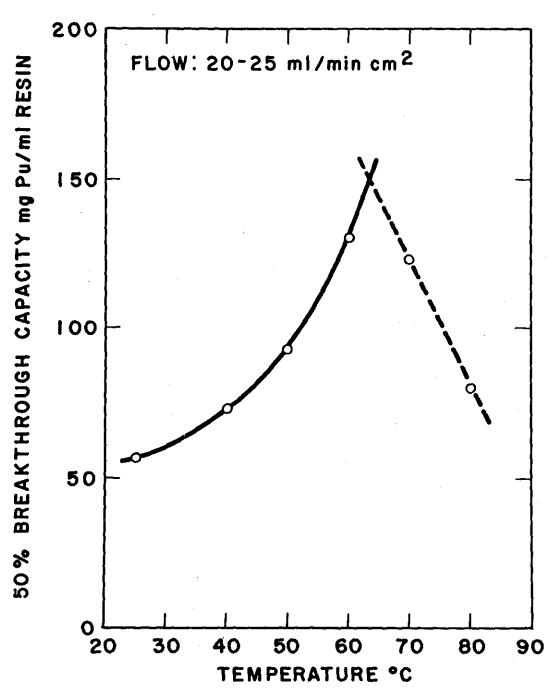
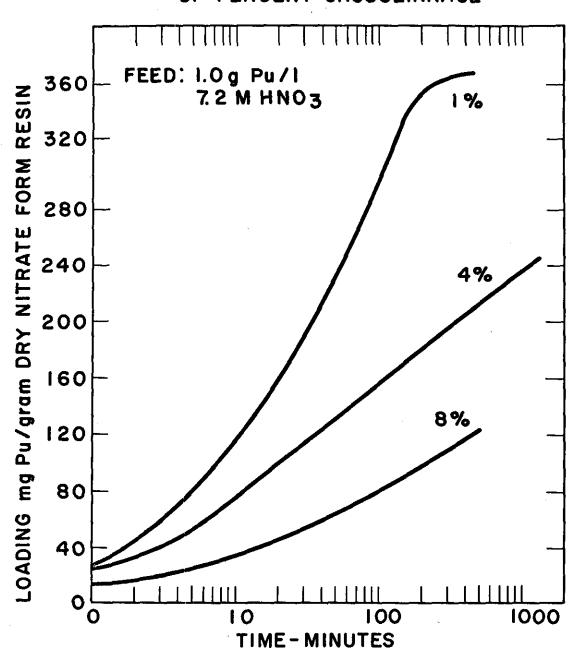


FIGURE 3

## PLUTONIUM LOADING KINETICS OF DOWEX-1 (50-100 MESH) AT 25°C AS FUNCTION OF PERCENT CROSSLINKAGE



## PLUTONIUM LOADING KINETICS OF VARIOUS RESINS AT 60°C

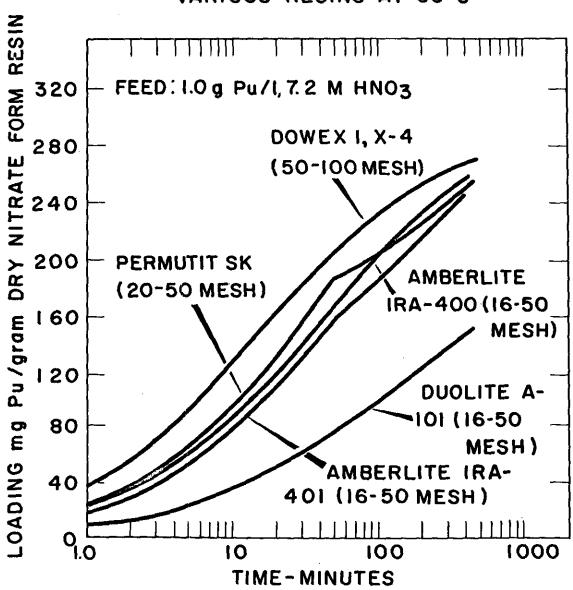
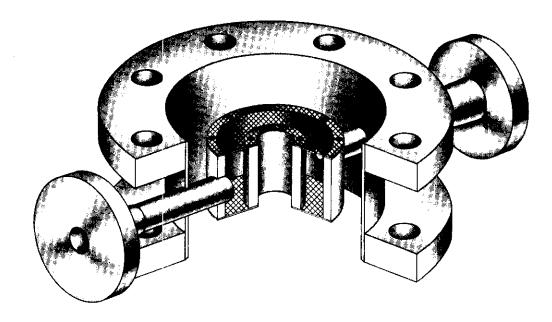


FIGURE 5

FIGURE 6

## ION EXCHANGE SOLUTION DISTRIBUTOR



ION EXCHANGE SOLUTION COLLECTOR

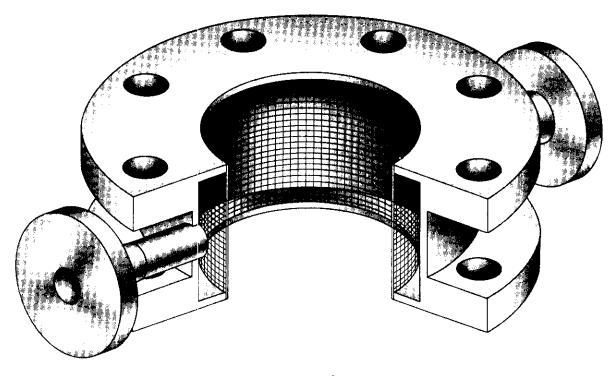


FIGURE 6A

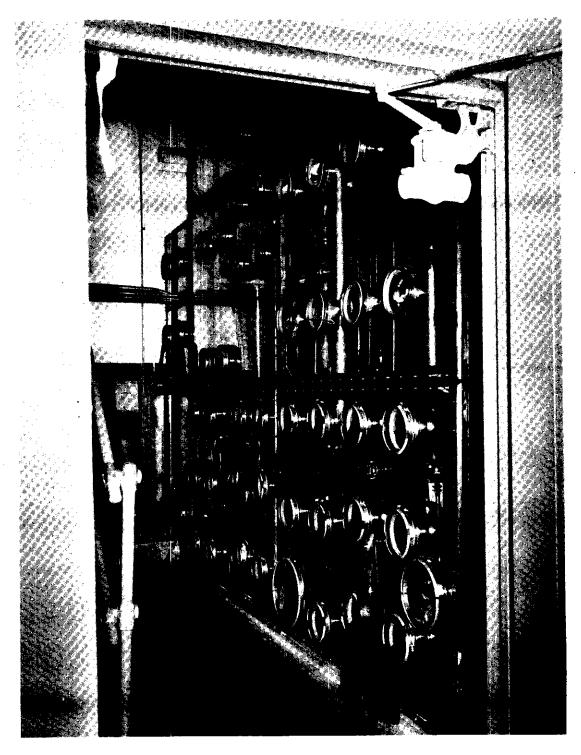
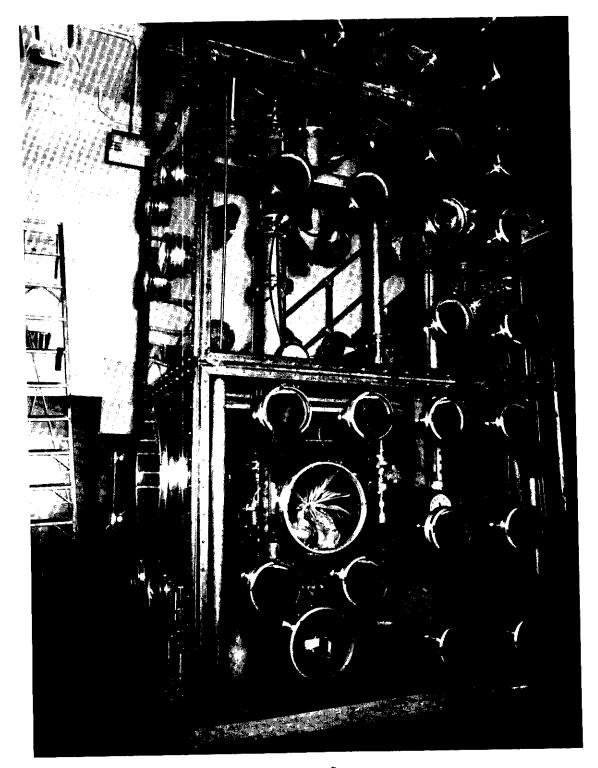


FIGURE 7
Lengthwise View of Anion Exchange Hood





Anion Exchange Hood Showing Concentrator and Condenser

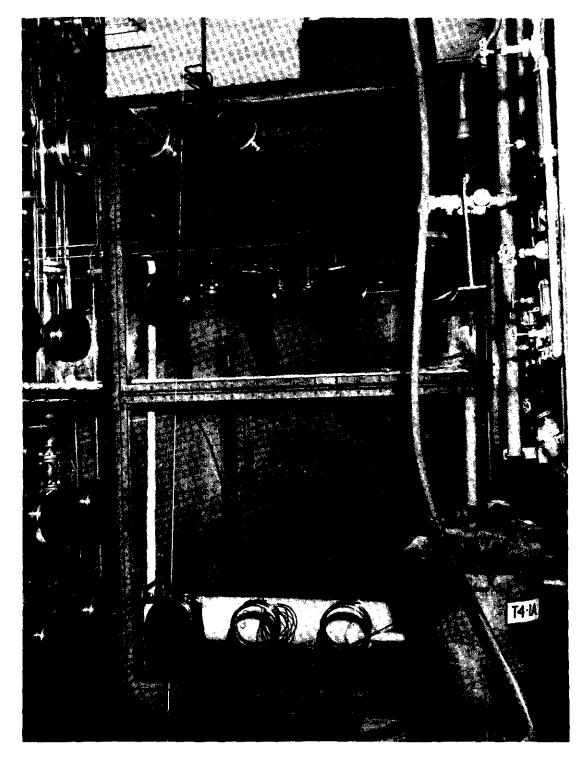
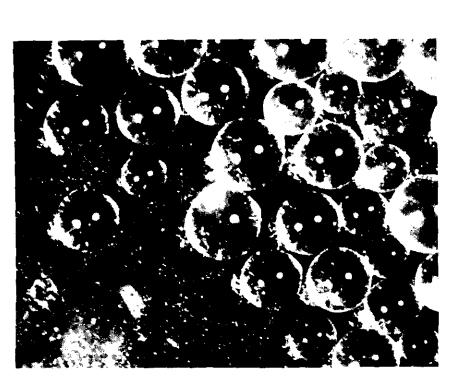
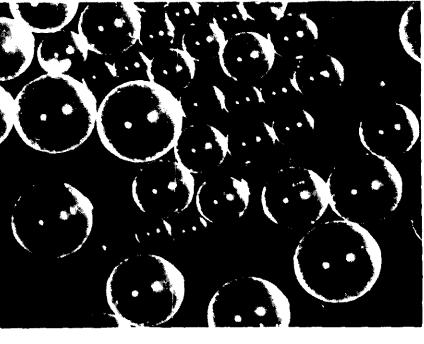


FIGURE 10

Anion Exchange Hood Showing Feed Tank



Resin after 110 days of plutonium processing at approximately 60 C in Purex plant continuous contactor.



Nitrate form resin as charged to Purex plant continuous contactor. (25X)

# FIGURE 11

Stability of Permutit SK Resin to Operation in Plant Scale Plutonium Anion Exchange Continuous Contactor

March 15, 1965

3. K. Sharp, Patent Attorney Hickland Operations Office

### PATRIC CLEARANCE

I have given patent clearence to the following document which you will receive on standard distribution.

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#### Title

N#-3101

Progress in Flutonium Utilization

## te mile

Discloses the subject setter of the following invention reports:

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4/18/2000 Date

TO: -

## PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
PROGRESS IN PLUROSIUS UTILIZATION	B4~33601
AUTHOR O. P. Hill	
PROPOSED DISTRIBUTION	
PATENT REVIEW	
NO OBJECTION TO PROPOSED DISTRIBUTION	
DOES NOT DISCLOSE AN INVENTION.	2, 1595, 25, 1130, 1210,
† DISCLOSES THE SUBJECT MATTER OF HWIR- 1505. 1016. 1745. Q	16, 1057, 1227, 1577, 1003 115, 1503, 1607, 1605, 1513
DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION.	···/, ·/··/, 1···/, 1···/, 1/13
DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HWIR HAS BEEN FILED.	
DISCLOSES SUBJECT MATTER OF HWIR-	
REMARKS: BUTR-1745, 1227, 3414, 1605, and 1537 have been	m dropped. Patent
applications have been filed on all the other cases.	
GENERAL LEGAL REVIEW	
NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.	
LEGAL CLEARANCE WITHHELD OR QUALIFIED.	
REMARKS:	
cc: To Be Suported File HVIR Files	
Publications File Robert Keith	Maria was
PATENT ATTORNEY COUNSEL'S OFFICE	,



COMPANY

RICHLAND, WASHINGTON . . TELEPHONE AREA CODE 509, 942-1111 99352

HANFORD

ATOMIC

PRODUCTS

**OPERATION** 

Extension 3601

3760 Bldg., 300 Area

JAN 1 7 1964

bec: HWIR Files

IJ James, Jr. Publications File

Record Copy

LB

Mr. George H. Lee, Chief Chicago Patent Group U. S. Atomic Energy Commission Chicago Operations Office 9800 South Cass Avenue

Subject: RELEASE OF DOCUMENT

HWIR-

AEC CASE NO

1051 (-19,5)

1057

Dear Mr. Lee:

Argonne, Illinois

In accordance with your letters of April 8, 1960 and July 18, 1961, I have given patent clearance for distribution or publication of the following document, a copy of which is enclosed:

Document Number:

HW-SA-3232

Title: "New Meptunium Purification Facility at the Purex Plant"

Author: J. P. Duckworth and J. R. LaRiviere

Type of Distribution: American Chemical Society Meeting - Division of Nuclear Chemistry and Technology, Denver, Colorado 1-20-24-64. The above invention report(s) is (are) involved.

Remarks: Patent applications have been filed on the above cases.

Titles

HWIR-1051 - "Separation of Neptunium and Plutonium by Anion Exchange"

(HW-57945) by J. L. Ryan and F. P. Roberts Very truly yours,

HWIR-1057 - "The Use of Fluoride to Increase the

Separation of Phytonium from Fission Products by Nitric Acid Anion Exchange

Process' by E. J. Wheelwright.

Robert Keith Sharp Patent Attorney

RKS:eg

Enclosure

a PRIME CONTRACTOR FOR THE U.S. ATOMIC ENERGY COMMISSION cc: Roland A. Anderson - USAEC-HDQ (w/encl.)

OEC 23 1963

## PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	<del></del>		DOCUME	NT NUMBER	
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AUTHOR	rtere		-		
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Technology, Deswor, Colore D. January		424 4 7 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4			
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ce: JF Duckworth JR LaRiviero					
Publications File	Robert Kel	th Sherry			
Mark Piles	PATENT AT			,	
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COMPANY

RICHLAND, WASHINGTON . . . TELEPHONE AREA CODE 509, 942-1111 HANFORD ATOMIC PRODUCTS

OPERATION

Extension 3601

3760 Bldg., 300 Area

December 17, 1963

Mr. George H. Lee, Chief Chicago Patent Group U. S. Atomic Energy Commission Chicago Operations Office 9800 South Cass Avenue Argonne, Illinois

bcc: HWIR Files IJ James, Jr. Publications File Record Copy

Subject: RELEASE OF DOCUMENT

HWIR-

1017 1051

1057

Dear Mr. Lee:

In accordance with your letters of April 8, 1960 and July 18, 1961, I have given patent clearance for distribution or publication of the following document, a copy of which is enclosed:

Document Number:

HW-SA-3283

Title: "Neptunium Recovery and Purification at Hanford"

Author: B. F. Judson

Type of Distribution: American Chemical Society National Meeting . January 21, 1964 at Denver, Colorado. The above invention report(s) bax (are) involved.

Remarks:

AEC Case S-18,905 has been dropped. Patent applications have been

filed on the other two cases.

Very truly yours,

Robert Keith Sharp Patent Attorney

RKS: eg

Enclosure

A PRIME CONTRACTOR FOR THE U.S. ATOMIC ENERGY COMMISSION cc: Roland A. Anderson - USAEC-HDQ (w/encl.)

TΩ		100

Marriso Moridan

## PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
BEFFUREN RECOVERY AND FORTFRACTOR AT MARK	III III-6A-3263
AUTHOR	
B. F. Judaon	
PROPOSED DISTRIBUTION Anariogn Chamical Society Bution	al Mosting - Jumery Sl, 196 et
PATENT REVI	E W
NO OBJECTION TO PROPOSED DISTRIBUTION	
DOES NOT DISCLOSE AN INVENTION.	A STATE OF THE PARTY OF THE PAR
DISCLOSES THE SUBJECT MATTER OF HWIR-	1, 1057
DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION	
DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HWIR HAS E	EN FILED.
DISCLOSES SUBJECT MATTER OF HWIR-	
REMARKS:	polications have been filed on
- the clima two states.	<u> </u>
<u>GENERAL LEGAL R</u>	EVIEW
NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.	
LEGAL CLEARANCE WITHHELD OR QUALIFIED.	
LEGAL CLEARANCE WITHHELD OR GOALIFIED.	
REMARKS:	
ee: Wik Files	
Publications File	
PATE	Hobert Batth Sharp
	EL'S OFFICE

## PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
BEFERENSE SECONERT AND PERIFICATION AS	
	B-3a-3203
AUTHOR	
B. F. Judaca	
PROPOSED DISTRIBUTION AMERICAN CAMBONI SOCIETY	Butional Meeting - Jenuary 21, 1964 at
Degree, Colorado.	
PATENT	REVIEW
NO OBJECTION TO PROPOSED DISTRIBUTION	
DOES NOT DISCLOSE AN INVENTION.	
DISCLOSES THE SUBJECT MATTER OF HWIR-	, 1051, 1057
DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIB	UTION.
DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HW	YIR HAS BEEN FILED.
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- ca the other two exame.	
GENERAL LEGA	L REVIEW
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NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.	
LEGAL CLEARANCE WITHHELD OR QUALIFIED.	
REMARKS:	
eo: W Judnon	
Buytumium Resovery File	
BOR Piles	School Enith Sharp
Publications File	PATENT ATTORNEY
	COUNSEL'S OFFICE

W. H. Swift 326 Building 300 Area

G. C. Oberg 202-A Building 200-Heat Area

## PARENT AND LEGAL REVIEW OF THI-SA-2290 CONTINUES ANION EXCHANGE PROCESSING OF PLUTONION

I have given patent and legal clearance to your paper. There are, however, certain modifications which, if they can be made, would be desirable from the legal and patent aspects.

First, it is desirable to avoid the appearance of endorsement of one company's products as against those of another and it is therefore desirable to identify products by composition rather than by trade name, wherever possible, particularly when making comparisons. I realize that it is impossible to fully adhere to this principle in the field of ion exchange resins. However, the "advertising" aspect is reduced if the differences in composition are given as far as possible. The comparisons then become scientific, rather than trade, comparisons.

On page 7, you state: "Figure 5 shows kinetic data for several resins of varying mesh size, cross-linkage, and functional exchange site nature." Home of these properties, except size, are given, however. It would be desirable for the above reasons to give the cross-linkage and functional exchange site nature in so far as you know them.

It is also desirable from the patent standpoint that the technical information be as complete as possible, since trade name identifications are given very little weight in the patent field. At the time the patent application was filed on B/IR-1057 (BF-58327) we were able to obtain sufficient identification of only Bowex-1.

Under all the circumstances, I did not feel justified in withholding legal clearance, but I would appreciate your making any changes possible along the lines indicated.

ORIGINAL SIGNED BY ROBERT KEITH SHARP

Petent Attorney

RK Sharp: 1b 3760 Bldg., 300 /rea

CG: M. Sheridan

MIR-1057

Publications File
LB

τη,	X.	Sheridan		
1111			 	

4..7000-012 ( 9-61)

### PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE		DOCUMENT NUMBER			
11122	Continuous Anion Exchange Processing of Plutonium				
		My-8A-2290			
AUTHOR					
AUTHOR	W. R. Swift and G. C. Oberg	1			
	D DISTRIBUTION Symposium on Ion Exchange at Feb. 1962 AICHE Me				
and f	or possible publication in an AICHE periodical such as Chem	ical Engineering Progres			
	PATENT REVIEW				
DX N	O OBJECTION TO PROPOSED DISTRIBUTION				
	DOES NOT DISCLOSE AN INVENTION.				
	DISCLOSES THE SUBJECT MATTER OF HWIR- 1057				
	O NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION				
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XX ·	IO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.				
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· '	EGAL CLEARANCE WITHHELD OR QUALIFIED.				
R	EMARKS: See attached letter.				
	•				
_					
•					
		L SIGNED BY			
	Publications File PATENT ATTORNEY	KEITH SHARP			
	TWIR-1057 COUNSEL'S OFFICE				



COMPANY

RICHLAND, WASHINGTON . . TELEPHONE WHitehall 2-1111

HANFORD ATOMIC **PRODUCTS** 

**OPERATION** 

Extension 3601

3760 Bldg., 300 Area

➤ HWIR-1057 File

Record Copy

Publications File

October 27, 1961

 $\mathbb{I}\mathbb{B}$ 

Mr. George H. Lee, Chief Chicago Patent Group U. S. Atomic Energy Commission Chicago Operations Office 9800 South Cass Avenue Argonne, Illinois

Subject: RELEASE OF DOCUMENT

HWIR-

aec case no

1057

Dear Mr. Lee:

In accordance with your letters of April 8, 1960 and July 18, 1961 I have given patent clearance for distribution or publication of the following document, a copy which is enclosed.

Document Number: 18-5A-2290

Title: Continuous Anion Exchange Processing of Pintonius.

Author: W. H. Swift and O. C. Oberg

Type of Distribution: AICHE Mosting, Los Angeles, Feb. - 1962.

The above invention report(s) is (are) involved.

Remarks:

Very truly yours,

Robert Keith Sharp

Patent Attorney

RKS:1b

Enclosure A PRIME CONTRACTOR FOR THE U.S. ATOMIC ENERGY COMMISSION cc: Roland A. Anderson - USAEC-HDQ w/encl.



## UNITED STATES ATOMIC ENERGY COMMISSION CHICAGO OPERATIONS OFFICE

9800 South Cass Avenue Argonne, Illinois

August 1, 1960

Mr. Robert Keith Sharp, Patent Attorney Hanford Atomic Products Operation General Electric Company 3760 Building, 300 Area Richland, Washington

Subject: CASE S-19,530 (HWIR-1057)

Dear Mr. Sharp:

The above application has been filed in the U. S. Patent Office on June 16, 1960 and assigned Serial No. 36,693. The work covered by this application was done under Contract No. W-31-109-Eng-52 with General Electric Company.

Very truly yours,

George H. Lee, Chief Chicago Patent Group Mr. George E. Lee, Chief Chicago Patent Group U. S. Abanic Energy Commission Chicago Operations Office 9800 South Case Avenue Argonne, Illimois

Door Mr. Lee:

#### MIR-1057 - AEC CASE S-19.530

Enclosed herewith are the Assignment and carbon copy of the Specification for the above case, which has already been filed in the Patent Office.

Yery truly yours,

HARF

Robert Keith Sharp Patent Attorney 3760 Building, 300 Area

MKS: pag

Encl.

bcc: Records Management

File LB W. Sale, Manager Financial Operation Monford Laboratories Hanford Atomic Products Operation

#### PATRIC MAND - SAME J. AMEDIAN DIREC

We are enclosing one copy of Notice of Award and four copies of an approved Notice of Award.

We believe this is all the information necessary for your office to preceed with the processing of request for issuence of sward for the period June 1, 1960 through July 3, 1960.

> (4) (12) (1) (94 8 (4) (1) (2) (4) (1) (4<del>1</del> Robert Keith Sharp Patent Attorney

RES:

Reclosures

cc: AJ Wheelwright personnel folder

bcc: PB Lamphere MT Walling

File - Patent Award File - INIR-1057

Records Management

LB

July	3, 19	00		
Trans	Cor L	ist	<b>3</b> 0.	
MIT.	LIST	<b>30</b> .		99

L. L. Weise, Memager Consolidated Payroll Reports and Statistics General Electric Company Schoosetsdy, New York

Period: June 1, 1960 through July 8, 1960 (MA-55)

The following employee has signed a potent application and has been granted an enerd of \$125, payable in part in shares of Guarral Electric Congany common stock and remainder in cash:

	ČÁŠ D.	DATE OF DEPENDENCE REPUBLICA	APPLICATION STANDARD	PILL BANK AND ANDRESS	ACCOUNT TO BE CHARGED
10.7	5 <b>-1</b> 9,5 <b>3</b> 0	12 <b>-10-</b> 53	9 <b>-26-€0</b>	Geri J. Wheelwright (L.) Cetekill Giobland, Venhington	

Mail certificate to: W. Jele, Manager

Pinsocial Operation

Hunford Laboratories Operation Hanford Atomic Products Operation

Richland, Kashington

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werd.	Authorized	1	The second section of the second seco	A SIMIT	cartified:	Box.; X-17***** enterlandament frahrindle station, allient all the factories designed

Mr. Earl J. Mascluright (1) Catabill Michland, Vachington

Bear Mr. Wheel wright:

## MIR-10-7 - AND CASE S-10,540

In connection with your execution of a potent application on the majort matter of the above case and in token of the Company a appreciation for the contribution which your invention represents, you are being awarded \$12>, payable in part in shares of General Alectric Company common stock and remainder in qual-

You will be advised of the market value of the stock at the time the stock certificate is delivered to you. You may expect to receive the certificate in the mear future.

Acta rikra mortes

General Aspegar

W. Johnson: ME: THE

ca: Al Masolwright personnel folder

boc: PR Lemphere 1906

W. Sele

MT Walling

WE Johnson

RK Sharp (3) Kild

Records Management



## UNITED STATES ATOMIC ENERGY COMMISSION CHICAGO OPERATIONS OFFICE

9800 South Cass Avenue Argonne, Illinois

June 24, 1960

Mr. Robert Keith Sharp, Patent Attorney Hanford Atomic Products Operation General Electric Company 3760 Building, 300 Area Richland, Washington

Subject: CASE S-19,530 (HWIR-1057)

Dear Mr. Sharp:

We have received from Mr. Anderson two original assignments which have been executed by Mr. Wheelwright. Mr. Anderson believes that it would be preferable to use the assignment executed by Mr. Wheelwright on June 10, 1960, the same day on which the application was executed.

We are also enclosing a carbon copy of the application, as filed, for presentation to Mr. Johnson for his signature on the assignments. After the assignments have been executed by Mr. Johnson, please return one copy to us together with the enclosed application.

Very truly yours,

George H. Lee, Chief Chicago Patent Group

Enclosures:

1. Spec. & Claims

2. Assignment by Wheelwright (2)



## UNITED STATES ATOMIC ENERGY COMMISSION

#### CHICAGO OPERATIONS OFFICE

9800 South Cass Avenue Argonne, Illinois

June 24, 1960

Mr. Robert Keith Sharp, Patent Attorney Hanford Atomic Products Operation General Electric Company 3760 Building, 300 Area Richland, Washington

Subject: CASE S-19,530 (HWIR-1057)

Dear Mr. Sharp:

We have received from Mr. Anderson two original assignments which have been executed by Mr. Wheelwright. Mr. Anderson believes that it would be preferable to use the assignment executed by Mr. Wheelwright on June 10, 1960, the same day on which the application was executed.

We are also enclosing a carbon copy of the application, as filed, for presentation to Mr. Johnson for his signature on the assignments. After the assignments have been executed by Mr. Johnson, please return one copy to us together with the enclosed application.

Very truly yours,

George H. Lee, Chief Chicago Patent Group

#### Enclosures:

1. Spec. & Claims

2. Assignment by Wheelwright (2)

### ASSIGNMENT

WHEREAS,	I, Earl J. Wheelwrig	ght,	a citizen of the United States residing
in]	Richland	, County of	Benton ,
State of	Washington	, have invented	certain new and useful improvements in
	RECOVERY OF PLUTONIUM	M FROM FIS	SION PRODUCTS ,
or which l	_	on for United S	States Letters Patent identified as
ınd;			
			acquire the entire right, title and in- ers Patent wherever they may be issued
ble considered transfer to the constant of the control of the cont	ration, the receipt of which is her r unto the said Government of the y Commission, and/or its assigns, t and in and to any and all Letters Pa i extensions of said Letters Patent United States to the full end of th	reby acknowledge United States, the entire right atent wherever the same to be term or terms	ed States, and for other good and valu- d, I by these presents do sell, assign, as represented by the United States title and interest in and to the said they may be granted thereon as well as be held and enjoyed by the said Govern- for which Letters Patent are or may be same would have been held or enjoyed by
eissue appl ents as may epresentati ell as afte nterference ay become i rotecting t ssigns, exc	ications, statements, or other ins y be required by the United State we, in or incident to the prosecut in the issuance of any Letters Pate is or other actions or proceedings involved, and I agree that I will as the invention as may be requested	truments in suc es Atomic Energion or conduct nt thereon, or that said appli id the Government by the United Strough extending	es, affidavits, renewal, divisional and the usual or other forms, terms, and congrego Commission, or its duly authorized of any and all applications, before as in the adjustment or settlement of any exactions may encounter or in which they not of the United States in every way in States Atomic Energy Commission or its g such assistance will be paid for by
	MONY WHEREOF, I have set my hand an		al this /D day of
STATE OF	Idaho Washington	, Cl	arl J Meelwight (SEAL)
	Dannock )		
o me well k	ly appeared before me the above-name nown, this <u>10th</u> day of <u>June</u> knowledged the same to be his volun	<u>, 1960</u> ,	who signed the foregoing in my pres- ed.
(NOTARIA)	L SEAL)	D	Notary Public Residing at Pocatello, Idaho
Approved	and consented to this 12 2	day of	My Come Expires: Oct. 13, 1963
TTEST.	Mrs. Ha -	GE1	

Mariom Powell 3760 Building, 300 Area

### HWIRe 1037, 1057, 1072, 1091, 1101, 1112, 1123, and 1155

There is no longer an objection from the patent standpoint to removal of any "Official Use Unly" marking and public release of the following reports:

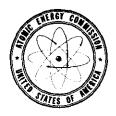
- (1) HW-62431, "The Preparation of Uranium Dioxide from a Molten Salt Solution of Uranyl Chloride" by W. L. Lyon and E. E. Voiland.
- (2) EW-62000, "Plutonium Recycle Program Annual Report, Fiscal Year 1959" by the Staff of Hanford Laboratories.

ORIGINAL SIGNED BY ROBERT KEITH SHARP

Robert Keith Sharp Patent Attorney 3760 Building, 300 Area

RKS:p

cc: EWIR Files (8)



## UNITED STATES ATOMIC ENERGY COMMISSION CHICAGO OPERATIONS OFFICE

9,800 South Cass Avenue Argonne, Illinois

May 18, 1960

Mr. Robert Keith Sharp, Patent Attorney Hanford Atomic Products Operation General Electric Company 3760 Building, 300 Area Richland, Washington

Subject: CASE S-19,530 (HWIR-1057) - EARL J. WHEELWRIGHT

Dear Mr. Sharp:

We have made all the changes requested in your letter of April 28, 1960 and rewritten the application which is enclosed together with the formal papers. We made two additional changes, namely we changed the concentration range for the elutriant from between 0.3 and 0.8, as we had it, to that given in pencil on the carbon copy of our letter returned to us, that is to between 0.5 and 2 M; this change was made in the specification and in claim 8. We also changed the last line or so of the paragraph meant by you as a substitute for our paragraph starting on page 4, line 14, and we inserted there the concentration range of from 5 to 9 M.

Please arrange to have the inventor, Earl J. Wheelwright, review the application and sign the papers at the places indicated by metal tabs. All signatures must be notarized and must correspond with the manner in which his name is typed in the body of the papers.

The city, county, and state of residence should be filled in toward the top of the oath, and the post office address should be inserted near the middle of the oath. The city, county, and state of residence should be filled in toward the top of the assignment.

The subject matter of the disclosure on the above docket is of value. The accompanying patent application covering said disclosure was prepared and will be filed by the Government, and the initial determination of rights has been made as to the subject invention.

Please return all of the enclosed papers to us when they have been executed with the exception of the extra original assignment which is for your files. This application and S-19,539 (HWTR-1051) should be filed on the same day and not later than June 15, 1960. Therefore, please return this application together with application S-19,539 which is also being sent to you today.

Very truly yours,

George H. Lee, Chief Chicago Patent Group

Enclosures:

- 1. Spec. & Claims
- 2. Oath
- 3. Assignment for Wheelwright (2)



#### UNITED STATES ATOMIC ENERGY COMMISSION

CHICAGO OPERATIONS OFFICE
9800 SOUTH CASS AVENUE

ARGONNE, ILLINOIS

May 18, 1960

Mr. Robert Keith Sharp, Patent Attorney Hanford Atomic Products Operation General Electric Company 3760 Building, 300 Area Richland, Washington

Subject: CASE S-19,530 (HWIR-1057) - EARL J. WHEELERIGHT

Dear Mr. Sharp:

We have made all the changes requested in your letter of April 28, 1960 and rewritten the application which is enclosed together with the formal papers. We made two additional changes, namely we changed the concentration range for the elutriant from between 0.3 and 0.8, as we had it, to that given in pencil on the carbon copy of our letter returned to us, that is to between 0.5 and 2 M; this change was made in the specification and in claim 8. We also changed the last line or so of the paragraph meant by you as a substitute for our paragraph starting on page 4, line 14, and we inserted there the concentration range of from 5 to 9 M.

Please arrange to have the inventor, Earl J. Wheelwright, review the application and sign the papers at the places indicated by metal tabs. All signatures must be notarized and must correspond with the manner in which his name is typed in the body of the papers.

The city, county, and state of residence should be filled in toward the top of the oath, and the post office address should be inserted near the middle of the oath. The city, county, and state of residence should be filled in toward the top of the assignment.

The subject matter of the disclosure on the above docket is of value. The accompanying patent application covering said disclosure was prepared and will be filed by the Government, and the initial determination of rights has been made as to the subject invention.

Please return all of the enclosed papers to us when they have been executed with the exception of the extra original assignment which is for your files. This application and S-19,539 (HNTR-1051) should be filed on the same day and not later than June 15, 1960. Therefore, please return this application together with application S-19,539 which is also being sent to you today.

Very truly yours,

George H. Lee, Chief Chicago Patent Group

#### Enclosures:

- 1. Spec. & Claims
- 2. Oath
- 3. Assignment for Wheelwright (2)

June 9. 1960

Mr. Joland A. Andorson Ascistant Consul Council for Patents V. S. Atomic Bayery Constanton Washington 25, D. C.

Dance Mr. Andrewsons

#### MIA-1057 - AND CASE 8-19,530

This is a composite case to 5-19,539 (MCM-1051). As indicated by Mr. Lee's letter of June 3, 1960, those two cases are to be filled on the some day and not later than June 17, 1960.

Since Mr. Macharight has been called out of torq, and the time is critical. I am having him forward all the papers and this letter directly to you.

Due to those elementamore, the continuents do not bear the approval and compact in behalf of General Electric Company. Mr. Low has an analyzonet dated May 26, 1960 which does bear this approval. If you consider the new analysment acceptancy, places return both copies to me, together with a confirmed copy of the application so that I may prepare them to Mr. Johnson for his electure.

Very tarkly present.

ORIGINAL SIGNED BY ROBERT KEITH SHARP

Robert Keith Sharp Patent Missing 3760 Bullding, 300 Area

**PE** 

Incl. Systification

Outh, Person of Attorney, and Potition

Antiquent (2)

bcc: Records Management

File LB

June 9, 1960

Mr. Barl J. Wheelwright c/o R. J. Wheelwright 146 South 17th Street Pocatello, Idaho

Dear Mr. Wheelwright:

#### BR-EXECUTION OF PATENT APPLICATION BWIR-1057 - AND CAME 8-19,530

As I explained to you on the telephone, the AEC attorneys have had the application retyped to incorporate the changes made in ink and initialed by you and have returned the papers for re-execution.

Please take them to a notary and insert the date and your signature, using your full first name, and have the notary fill out his certificate and affix his seal. The notary should see that the "venue" is correct. To call it to his attention, I have, in pencil, changed "State of Washington, County of Benton" to "State of Idaho, County of Bannock" on the Outh, Power of Attorney and Petition, which would, I believe, be correct if the papers are signed in Pocatello. He should make the proper change on all three papers.

When the papers are signed please forward them together with the attached letter to Roland A. Anderson, Assistant General Counsel for Patents, U. S. Atomic Energy Commission. A stemped and addressed envelope is enclosed for your convenience.

Very truly yours,

ORIGINAL SIGNED BY ROBERT KEITH SHARP

Robert Keith Sharp Patent Attorney 3760 Building, 300 Area

HKB: pmg

Encl.: Specification

Oath, Power of Attorney and Petition

Assignment (2)

Letter to Roland A. Anderson

Envelope

bea: File

LB



## UNITED STATES ATOMIC ENERGY COMMISSION CHICAGO OPERATIONS OFFICE

9800 SOUTH CASS AVENUE
ARGONNE, ILLINOIS

June 3, 1960

Mr. Robert Keith Sharp, Patent Attorney Hanford Atomic Products Operation General Electric Company 3760 Building, 300 Area Richland, Washington

Subject: REVIEW AND RE-EXECUTION OF S-19,530 AND S-19,539

Dear Mr. Sharp:

We send herewith the above applications for re-execution, including the originals of the specification. The changes initialed by the inventors have been made.

The applications must be filed in the Patent Office on the same day and by June 17, 1960. Therefore, we ask you to send them directly to Mr. Anderson, with a copy of your covering letter to us.

Very truly yours,

George M. Lee, Chief Chicago Patent Group

H. Lee\_

#### Enclosures:

1. Spec. & Claims for S-19,539

2. Oath for S-19,539

3.- Assignment for Ryan for S-19,539 (2)

4. Assignment for Roberts for S-19,539 (2)

5. Spec. & Claims for S-19,530

6. Oath for S-19,530

7. Assignment for Wheelwright for S-19,530

CC: Roland A. Anderson, Assistant General Counsel for Patents, USAEC, Washington 25, D. C.

AIR MAIL

Mr. George M. Lee, Chief Chicago Patent Group V. S. Atemie Emergy Consission Chicago Operations Office 9800 Bouth Case Avenue Argomne, Illinois

> MIN-816 - ARC CARE 8-15,473 MIR-1051 - ARC CARE 8-19,539 MIR-1057 - ARC CARE 8-19,539

> > ATTENTION - MAIN CAMES

Door Mr. Lee:

Enclosed are the executed formal papers for the above three encon-

Very truly yours,

or CMT WHITE IN Flour New York

Robert Keith Shary Patent Attorney 3760 Building, 300 Area

MAS:p

Inclosures:

Specifications (3)
Print of Drawings (1)
Outh, Power of Attorney and Petition forms (3)
Annigomests (4)

bec: Records Management HWIR Files (3) LB

## ASSIGNMENT

reissue applications, statements, or other instruments in such usual or other forms, terms, and contents as may be required by the United States Atomic Energy Commission, or its duly authorized representative, in or incident to the prosecution or conduct of any and all applications, before as well as after the issuance of any Letters Patent thereon, or in the adjustment or settlement of any interferences or other actions or proceedings that said applications may encounter or in which they have become involved, and I agree that I will aid the Government of the United States in every way in protecting the invention as may be requested by the United States Atomic Energy Commission or its assigns, except that any expenses arising through extending such assistance will be paid for become arrangement with the Government of the United States.  IN TESTIMONY WHEREOF, I have set my hand and affixed my seal this	WHEREAS, 1, Earl J. Wheelwright	, a citizen of the United States residing
RECOVERY OF PLUTONIUM FROM FISSION PRODUCTS  or which I am about to file an application for United States Letters Patent identified as (S-19,530)  and;  WHEREAS, the Government of the United States desires to acquire the entire right, title and interest in and to the said invention and in and to any Letters Patent wherever they may be issued hereon;  NOW, THEREFORE, to all whom it may concern, be it known that for and in consideration of the sum of One Dollar to me in hand paid by the Government of the United States, and for other good and valuable consideration, the receipt of which is hereby acknowledged, I by these presents dos sell, assign and transfer unto the said Government of the United States, as represented by the United States tomake Energy Commission, and/or its assigns, the entire right, title and interest in and to the said invention and in and to any and all Letters Patent wherever they may be granted thereon as well as invention and in and to any and all Letters Patent, the same to be held and enjoyed by the said Government of the United States to the full end of the term or terms for which Letters Patent are or may be granted, reissues and extensions of said Letters Patent, the same would have been held or enjoyed by the half this assignment not been made.  I agree to make, execute, and deliver unto the Government of the United States, or to the United States Atomic Energy Commission, any and all papers, documents, affidavits, renewal, divisional and reissue applications, statements, or other instruments in such usual or other forms, terms, and concerts as may be required by the United States Atomic Energy Commission, or incident to the prosecution or conduct of any and all applications, before an enters are may be required by the United States Atomic Energy Commission or it is saigns, except that any expenses arising through extending such assistance will be paid for by protecting the invention as may be required to the United States.  IN TESTIMONY WHEREOF, I have set my hand and affixed my se	, <u>Kickland</u> , County of	Benton,
WHEREAS, the Government of the United States desires to acquire the entire right, title and inverest in and to the said invention and in and to any Letters Patent wherever they may be issued hereon;  NOW, THEREFORE, to all whom it may concern, be it known that for and in consideration of the sua of the Consideration, the receipt of which is hereby acknowledged, I by these presents do sell, assign, and transfer unto the said Government of the United States, as represented by the United States in and to any and all Letters Patent wherever they may be granted thereon as well as recissues and extensions of said Letters Patent, the same to be held and enjoyed by the said Government of the United States, as represented to the said invention and in and to any and all Letters Patent wherever they may be granted thereon as well as reissues and extensions of said Letters Patent, the same to be held and enjoyed by the said Government of the United States to the full end of the term or terms for which Letters Patent are or may be granted, reissued or extended, as fully and entirely as the same would have been held or enjoyed by the had this assignment not been made.  I agree to make, execute, and deliver unto the Government of the United States, or to the United States, or to other instruments in such usual or other forms, terms, and concerns as may be required by the United States Atomic Energy Commission, or its duly authorize representative, in or incident to the prosecution or conduct of any and all applications, before a reliased applications and entered the issuance of any Letters Patent thereon, or in the adjustment or settlement of am interferences or other actions or proceedings that said applications may encounter or in which the pay become involved, and I agree that I will aid the Government of the United States Atomic Energy Commission or it which the inspiration of the invention as may be requested by the United States Atomic Energy Commission or in the said applications in the paid for by order than the follo	eate of Mashington, have inve	ented certain new and useful improvements in
MHEREAS, the Government of the United States desires to acquire the entire right, title and increst in and to the said invention and in and to any Letters Patent wherever they may be issued hereon;  NOW, THEREFORE, to all whom it may concern, be it known that for and in consideration of the sus of One Dollar to me in hand paid by the Government of the United States, and for other good and value ble consideration, the receipt of which is hereby acknowledged, I by these presents do sell, assign and transfer unto the said Government of the United States, as represented by the United States to the United States, as represented by the United States are insured and in and to any and all Letters Patent wherever they may be granted thereon as well a cissues and extensions of said Letters Patent, the same to be held and enjoyed by the said Government of the United States to the full end of the term or terms for which Letters Patent are or may be ranted, reissued or extended, as fully and entirely as the same would have been held or enjoyed by the had this assignment not been made.  I agree to make, execute, and deliver unto the Government of the United States, or to the United States Atomic Energy Commission, any and all papers, documents, affidavits, renewal, divisional and ecissue applications, statements, or other instruments in such usual or other forms, terms, and concents as may be required by the United States Atomic Energy Commission, or its duly authorized epresentative, in or incident to the prosecution or conduct of any and all applications, before a reliance of any Letters Patent thereon, or in the adjustment of settlement of any neterior the insurance of any Letters Patent thereon, or in the adjustment of settlement of any notecting the invention as may be requested by the United States Atomic Energy Commission or its duly authorized epresentative, in or incident to the prosecution or conduct of any and all applications, before a rote of the United States in every way in the protecting the invention as may	RECOVERY OF PLUTONIUM FROM	FISSION PRODUCTS ,
WHEREAS, the Government of the United States desires to acquire the entire right, title and increase in and to the said invention and in and to any Letters Patent wherever they may be issued nereon;  NOW, THEREFORE, to all whom it may concern, be it known that for and in consideration of the sum of One Dollar to me in hand paid by the Government of the United States, and for other good and valuable consideration, the receipt of which is hereby acknowledged, I by these presents do sell, assign and transfer unto the said Government of the United States, as represented by the United States of Commission, and/or its assigns, the entire right, title and interest in and to the said neutrino and in and to any and all Letters Patent wherever they may be granted thereon as well as eissues and extensions of said Letters Patent, the same to be held and enjoyed by the said Government of the United States to the full end of the term or terms for which Letters Patent are or may be remated, reissued or extended, as fully and entirely as the same would have been held or enjoyed by a had this assignment not been made.  I agree to make, execute, and deliver unto the Government of the United States, or to the United states Atomic Energy Commission, any and all papers, documents, affidavits, renewal, divisional and eissue applications, statements, or other instruments in such usual or other forms, terms, and contents as may be required by the United States Atomic Energy Commission, or its duly authorized expresentative, in or incident to the prosecution or conduct of any and all applications, before as ell as after the issuance of any Letters Patent thereon, or in the adjustment or settlement of any neterferences or other actions or proceedings that said applications may encounter or in which they are proceedings that any expenses arising through extending such assistance will be paid for by reoper arrangement with the Government of the United States.  IN TESTIMONY WHEREOF, I have set my hand and affixed my seal this 24 day of		ted States Letters Patent identified as
NOW, THEREFORE, to all whom it may concern, be it known that for and in consideration of the sur of One Dollar to me in hand paid by the Government of the United States, and for other good and value bele consideration, the receipt of which is hereby acknowledged, I by these presents do sell, assign, and transfer unto the said Government of the United States, as represented by the United States tomic Energy Commission, and/or its assigns, the entire right, title and interest in and to the said novention and in and to any and all Letters Patent wherever they may be granted thereon as well as eissues and extensions of said Letters Patent, the same to be held and enjoyed by the said Government of the United States to the full end of the term or terms for which Letters Patent are or may be ranted, reissued or extended, as fully and entirely as the same would have been held or enjoyed be had this assignment not been made.  I agree to make, execute, and deliver unto the Government of the United States, or to the United states Atomic Energy Commission, any and all papers, documents, affidavits, renewal, divisional and eissue applications, statements, or other instruments in such usual or other forms, terms, and con ents as may be required by the United States Atomic Energy Commission, or its duly authorized epresentative, in or incident to the prosecution or conduct of any and all applications, before as ell as after the issuance of any Letters Patent thereon, or in the adjustment or settlement of any interferences or other actions or proceedings that said applications may encounter or in which they as become involved, and I agree that I will aid the Government of the United States in every way in rotecting the invention as may be requested by the United States Atomic Energy Commission or its assigns, except that any expenses arising through extending such assistance will be paid for by roper arrangement with the Government of the United States.  IN TESTIMONY WHEREOF, I have set my hand and affixed my seal this	nd:	
f One Dollar to me in hand paid by the Government of the United States, and for other good and value ble consideration, the receipt of which is hereby acknowledged, I by these presents do sell, assign, and transfer unto the said Government of the United States, as represented by the United States tomic Energy Commission, and/or its assigns, the entire right, title and interest in and to the said needs on and in and to any and all Letters Patent wherever they may be granted thereon as well as eissues and extensions of said Letters Patent, the same to be held and enjoyed by the said Government of the United States to the full end of the term or terms for which Letters Patent are or may be ranted, reissued or extended, as fully and entirely as the same would have been held or enjoyed by the had this assignment not been made.  I agree to make, execute, and deliver unto the Government of the United States, or to the United states Atomic Energy Commission, any and all papers, documents, affidavits, renewal, divisional and eissue applications, statements, or other instruments in such usual or other forms, terms, and con ents as may be required by the United States Atomic Energy Commission, or its duly authorized epresentative, in or incident to the prosecution or conduct of any and all applications, before as well as after the issuance of any Letters Patent thereon, or in the adjustment or settlement of any neterferences or other actions or proceedings that said applications may encounter or in which they have been proceeding the invention as may be requested by the United States Atomic Energy Commission or its assigns, except that any expenses arising through extending such assistance will be paid for by roper arrangement with the Government of the United States.  IN TESTIMONY WHEREOF, I have set my hand and affixed my seal this   Aday of   TATE OF   Machine II have set my hand and affixed my seal this   Aday of   TATE OF   Machine II have set my hand and affixed my seal this   Aday of   TATE OF   Machine II have set	erest in and to the said invention and in and to any I	
States Atomic Energy Commission, any and all papers, documents, affidavits, renewal, divisional and reissue applications, statements, or other instruments in such usual or other forms, terms, and contents as may be required by the United States Atomic Energy Commission, or its duly authorized representative, in or incident to the prosecution or conduct of any and all applications, before as well as after the issuance of any Letters Patent thereon, or in the adjustment or settlement of any interferences or other actions or proceedings that said applications may encounter or in which they have become involved, and I agree that I will aid the Government of the United States in every way in protecting the invention as may be requested by the United States Atomic Energy Commission or its assigns, except that any expenses arising through extending such assistance will be paid for by proper arrangement with the Government of the United States.  IN TESTIMONY WHEREOF, I have set my hand and affixed my seal this	f One Dollar to me in hand paid by the Government of the ble consideration, the receipt of which is hereby acknowing transfer unto the said Government of the United Stromic Energy Commission, and/or its assigns, the entire extension and in and to any and all Letters Patent where eissues and extensions of said Letters Patent, the same ent of the United States to the full end of the term or example, reissued or extended, as fully and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and entirely as the same of the United States to the full and the same of the United States to the full and the same of the United States to the full and the same of the United States to the full and the same of the United States to the full and the same of the United States to the full and the same of the United States to the full and the same of the United States to the full and the same of the United States to the same of the United States to the full and the same of the United States to the same	United States, and for other good and valu- ledged, I by these presents do sell, assign, cates, as represented by the United States right, title and interest in and to the said ever they may be granted thereon as well as to be held and enjoyed by the said Govern- terms for which Letters Patent are or may be
IN TESTIMONY WHEREOF, I have set my hand and affixed my seal this day of	tates Atomic Energy Commission, any and all papers, doctorissue applications, statements, or other instruments in ents as may be required by the United States Atomic I representative, in or incident to the prosecution or concell as after the issuance of any Letters Patent thereon interferences or other actions or proceedings that said any become involved, and I agree that I will aid the Governotecting the invention as may be requested by the Unissigns, except that any expenses arising through extended	uments, affidavits, renewal, divisional and n such usual or other forms, terms, and con- Energy Commission, or its duly authorized duct of any and all applications, before as , or in the adjustment or settlement of any applications may encounter or in which they ernment of the United States in every way in ted States Atomic Energy Commission or its ending such assistance will be paid for by
State of Washington SS:	IN TESTIMONY WHEREOF, I have set my hand and affixed:	my seal this day of
COUNTY OF Benton SS:		8.000 11 · H
COUNTY OF Benton SS:	FATE OF Washington	Way Meeswright (SEAL)
Personally appeared before me the above-namedEarl J. Wheelwright		
o me well known, this 26th day of may, 1961, who signed the foregoing in my presence and acknowledged the same to be his voluntary act and deed.	o me well known, this 26 day of May, 19	<u>L</u> , who signed the foregoing in my pres-
Loh K. Klyser		Notary Public
(NOTARIAL SEAL)	/www.a	
Approved and consented to this 26th day of	(NOTARIAL SEAL)	
GENERAL ELECTRIC COMPANY HAN FORDY ATOMIC PRODUCTS OPERATION	$\sim$	of <u>man</u> , 19 6 x

bcc: Records Management

File LB

April 88, 1960

Mr. George H. Lee, Chief Chiengo Patent Group V. S. Atomic Energy Consission Chiengo Operations Office 9800 South Ones Avenue Argome, Illinois

Dear Mr. Les:

## INTR-1097 - AND CARE 8-19.53

This is in response to your letter of Murch 39, 1960. The dreft of the specification has been reviewed in detail by the inventor.

The product lover RIK is stated by the inventor to be chamically identical with Bower 1. Apparently there is some physical difference, but he does not even know what that is. He know nothing about the chamistry of Fermatit EK beyond that already given. The most that I have been able to find is that it is a genterousy assentant regin (Emin, Ion Embange Regime, Second Edition, page 93).

In my own past experience I found that more information regarding the chanical identity of synthetic regime could be obtained by going to hiv. 50 of the February Office than by any other means. They have an extensive collection of manufacturer's literature. Feelings common in your Vaskington office could help us cut. I fear, however, that Fermutit may be one of the "close-mouthed" organizations.

I am returning the earten copy of your letter, on which the inventor has given some of the data requested by you. So has also extered proposed ementments in pencil.

I should purhaus emplain that the figures on page 6, lines 17 and 18 mmm "gamm-disintegrations (or gamm photons) per minute per milliliter". The "gamm count" has been corrected to give those fundamental values.

The other changes proposed by the inventor are given as follows in the conventional "assaulment" form.

Form 2, owned the extire year and substitute:

--This invention deals with the recovery of plutonium values from an aqueous nitric acid solution by rudon emchange and in particular with the separation of plutonium values from fiscien product values.

The amion emchange process is readily adaptable to a vide variety of process applications. The process is besically a three step operation——an absorption step wherein emionic species of plutonium are absorbed into the resin matrix, a resin washing step (if necessary), and an elution, step. The optimum choice of operational variables is dependent upon the requirements of a particular application. Some operating conditions are rather closely defined and apply to all applications of the process, others are flexible and must be determined by the requirements of the given application.

When the process is used for recovering and concentrating plutonium which has been previously decentaminated from all objectionable impurities, no decentamination is necessary and the washing cycle can be eliminated from the process. If, however, anion exchange is used as a process where decentamination from uranium, fission products, or other ionic or nomical impurities is desired, a washing cycle must be inserted between the absorption and elution cycles. The quantity and composition of wash solution used will depend upon the efficiency of the equipment design, the degree of decontamination required, the wash solution flow rate, and the resin being used.

Hitric acid solutions containing plutonium and fission product values are obtained during the processing of neutron-irradiated uranium, for instance by entracting the uranium and the bulk of the plutonium with tributyl or other triallyl phosphates and subsequently back-entracting the plutonium with a solution of a reducing agent. The recovery of plutonium from much an aqueous nitric acid solutions with strong-base amion emakings resime by use of an all nitric acid or metal nitrate processes have been tried. However, such processes have the drawback that when the plutonium loaded resim is washed with strong nitric acid or metal nitrate solutions to remove conhected fission products, some fission products are not removed; in particular, sironnium and nichium are held rather strongly by the resim. Consequently, these fission products, or at least part of them, are cluted together with the plutonium when the resim is treated with dilute nitric acid, which means that the plutonium product solution is conteminated with fission products.--

Page 4, cancel the paragraph beginning on line 14 and substitute:

--The plutonium content of the solution to be treated, the feed solution, may vary videly, depending on the specific application; however, since the resin capacity begins to decrease when the consentration of plutonium in the feed solution is decreased below a value of 0.4 grams of plutonium per liter of solution, the plutonium content of the feed solution should not be lower than 0.4 grams of plutonium per liter of solution in those applications where a high process rate is necessary. In processes where less efficiency can be tolerated; such as the recovery of plutonium from "off-standard" solvent extraction made solutions which are waste solutions containing unusually high consentrations of plutonium, plutonium feed consentrations as low as 0.010

to 0.050 grams of plutonium per liter of solution can be tolerated. The proferred average concentration is 1 to 10 grams of plutonium per liter of solution but the process suffers no adverse consequence from a much higher consentration of plutonium in the food, even as high as 50 grams, of plutonium per liter of solution. The wranium consentration in such solutions is about 0.08 M, and the proferred mitric acid concentration for the process of this invention is approximately 7 M.---

Page 6, lines 1-8, cancel "en-----resin" and substitute:

--Single cycle sipecnium-nicbium decentumination factors in the range of  $2 \times 10^5$  to  $5 \times 10^5$  are readily obtained by the process of this invention and with special ears sireonium-nicbium decentemination factors as high as  $5 \times 10^7$  can be achieved in one cycle. (The decentemination factor is the semant of finaism products in the feed solution times the amount of plutonium in the eluted product solution divided by the amount of plutonium in the feed solution times the amount of finaism products in the eluted product solution).--

As regards the requested example based on pages 98-108, the immediar states that this was a highly theoretical proposal as to which he cannot give the necessary data. He has proposed the following Enumple III to show separation of uranium. It does not immive the invention facture, however, and you my feel that it would confuse the Losse rather than help. That is a matter that I leave to your judgment. It does show applicability of the basic ion exchange method to unprocessed dissolver solutions.

#### Describe III

The process of this invention has application in the processing of plutanium reactor fuels, more particularly the recovery of partitled plutanium from plutanium recycle reactor fuels. Three runs were made to determine the effect the high unanium content of such dissolved fuels vould have on the maion embange process. The feet solution for those three runs contained uranium and plutonium at a male ratio of 300 to 1; corresponding to natural uranium irrediated to about 5000 magnetit days per ten of unanium. The three experimental runs were conducted to determine the degree of separation of plutonium from uranium that could be achieved in one cycle with only a underste amount of resin washing when the uranium and plutonium are present in the 7 M MMO<sub>2</sub> feed solution at a male ratio of 300 to 1. The operating conditions and the decontemination results are compiled in Table III.

#### THE LIE

	1	2	3
Food Sulution			
Plutonium Concentration (grams per liter) Urunium Concentration (grams per liter) Botio Urunium/Flutonium	0.405 190 296	0.00k 60.6 278	0.626 184 294
Product Sciution			
Flutenium Commentantiem (grams per liter) Granium Commentantiem (grams per liter)	38.4 0.006	34.8 0.410	46.5 0.002
Urmius Decembersinsties Factor	1.9006	1.0006	6.0006

Nomin: Novem-1, X-4 (50-100 much) Temperature: 60 C for all operations

Work Solution: 16 Column Winnes of 7 M 1880;

The results of those three runs alongly show that plutonium can be almost completely decembrated from unusing in one spale. A second syste would reduce the unusing continue c

I have discussed the compasion case with the inventor thereof but he has not completed his detailed review. I will fewered it to you as come as I receive it.

Tury truly pours.

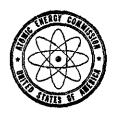
Nobert Solith Many Patent Attorney 3760 Bullding, 300 Area

#### MA: year

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# UNITED STATES ATOMIC ENERGY COMMISSION CHICAGO OPERATIONS OFFICE P. O. Box 59

LEMONT, ILLINOIS

March 30, 1960

Mr. Robert Keith Sharp, Patent Attorney Hanford Atomic Products Operation General Electric Company 3760 Building, 300 Area Richland, Washington

Subject: CASE S-19530 (HWIR-1057) - EARL J. WHEELWRIGHT

Dear Mr. Sharp:

We are enclosing the specification and claims for review by the inventor.

You no doubt know that trade names or trade marks mostly are objected to in patent applications. We have the basic information for Dowex 1 but do not have any background for Dowex 21K and Permutit SK also used in some examples. If this information is available to you or to the inventor, we would appreciate your sending it to us so that we can define also the other two resins used.

Further, it would be advantageous if we could have ranges for the acidities of feed solution, wash solution and eluant and also the concentration range for the aluminum nitrate. Likewise we would like to have the plutonium content of the feed solutions used in Example II.

We would very much like to add one other example, based on pages 98-102 of HW-55893, for two reasons: these pages seem to deal with the application of the process of this invention to an unprocessed dissolver solution and the resin there is Dowex 1 for which we have all the information necessary for sufficiency of disclosure. In order to prepare an example from these pages of HW-55893, we would have to know a little bit more about the fate of the uranium and the concentration in which it is present in the various solutions obtained in the process. Perhaps the inventor can give us some supplemental data so that we can add a third example.

We are sending you this application without formal papers because it seems that some additional information has to be incorporated. After the changes have been made we will resubmit the application to you with the formal papers. This application and S-19,539 (HWIR-1051) should be filed on the same day and not later than June 15, 1960. Therefore kindly return this application together with application S-19,539 also submitted to you today.

Very truly yours,

George H. Lee, Chief Chicago Patent Group

orge X. Lee

Enclosure: Specification and Claims Mr. George H. Lee, Chief Chicago Patent Group U. S. Atomic Energy Commission P. C. Rox 59 Lemont, Illinois

Dear Mr. Lee:

HWIR-1037 - AEC CASE S-13,959 HWIR-1057 - AEC CASE S-19,530 HWIR-1072 - AEC CASE S-19,577 HWIR-1091 - AEC CASE S-20,447 HWIR-1112 - AEC CASE S-20,486 HWIR-1123 - AEC CASE S-21,311

We have a request for approval of publication of the attached Unclassified Document RW-62000, entitled "Plutonium Recycle Program Armsal Report Fiscal Year 1959", to release for distribution.

We would appreciate receiving your approval of publication of the enclosed report as soon as possible.

Very truly yours,

Contract Engineer

C Poetes

nc.

ec: Wr. Roland Anderson w/ inc.

MR. WEBB: We would also appreciate receiving your approval as soon as possible. Our folders HWIR-1057, 1091 and 1123 are enclosed and should be returned to us when you have finished your review. WCP



January 27, 1960

W. C. Poe
703 Bldg.
700 Area

HW-62000
Plutonium Recycle Program Annual Report - FY 1959
by J. M. Atwood & W. A. Snyder (Unclassified)

The subject matter in the above document discloses patent information as follows:

HWIR - 1057 - 1037 HWIR - 1091 HWIR - 1123 HWIR - 1112 HWIR - 1072

Please notify us when there is no objection from a patent standpoint to release of the subject document.

As Mr. Snyder has mentioned to you, we are anxious to release this document as soon as possible.

Supervisor

DOCUMENT DISTRIBUTION & FILES

MF Puckett/vbl

#### ATOMIC PRODUCTS DIVINGE 1 Manufactorisation (ICC).

Cincinnati 15, Ohio

September 1, 1959

dre place her and

Mr. W. C. Poe Contract Engineer Hanford Atomic Products Operation RICHLAND, WASHINGTON

Subject: HWIR-1051 HWIR-1057

Dear Mr. Poe:

I am returning the above-identified files and the copy of Mr. E. J. Wheelwright's paper for presentation at Gatlinburg on September 7-9, 1959. My telegram to you, dated September 1, 1959, set forth that there appeared to be no Company patent objection to the publication of the attached speech in view of the above invention report folders.

Invention report folder HWIR-1057 indicates a request from your office that I recommend to Mr. R. A. Anderson filing of domestic and foreign patent applications by the Commission. Mr. Anderson advised you, on July 17, 1959, that a domestic patent application was being prepared on this invention report. On August 24, 1959, you requested Mr. G. H. Lee's office to approve the publication of a paper and speech by Mr. E.J. Wheelwright for presentation at Gatlinburg on September 7-9, 1959. You might wish to consider the relationship of these requests in connection with future recommendations for patent application filing by the Commission and publication of the material by HAPO inventors. As you know, publication of the material in Mr. Wheelwright's paper will bar patent coverage on Invention Report Folders HWIR-1051 and HWIR-1057 in most foreign countries, effective upon publication. However, the Commission does have a maximum period of one year after the publication to file an application in this country. Thus, it appears inconsistent for the Company to recommend domestic and foreign filing to the Commission and request, within a period of months afterwards, Commission approval to publish an article or present a speech on the same subject matter.

Since the disclosure in HWIR-1057 was initially classified, a recommendation for domestic and foreign filing was reduced to

KKEL

Cincinnati 15, Ohio

Mr. W. C. Poe

pur est contrade programmes on the character of the contrade o

9/1/59

domestic filing. It does not appear from the file that this modified recommendation was furnished to Mr. Anderson's office.

You might wish to point out in response to inquiries from inventors who plan to publish material based on invention reports that such publication bars substantially all foreign patent protection and creates a bar in this country one year after the publication. It would appear desirable for the members of the HAPO Patent Panel to consider domestic and foreign filing, and possible publication of the material. this manner, a recommendation to the Commission requesting filing would include by implication that no publication would be made of the subject matter within the immediate future. Unless this factor is taken into consideration, it is very possible that the Commission might tend to look unfavorably upon our requests for domestic and foreign filing.

I would be interested in discussing this matter further with you on the telephone at your convenience.

Very truly yours,

PAUL R. WEBB, II

Department Patent Counsel

PRW.II:dyh

Enclosures

### JN'T SAY IT --- b cite It!

Sivi bill in which it.	DATE <u>September 3, 1959</u>
TO PJ Wheelver girt	FROM
JE Brown	

#### HNIR-1051 - AND CASE 8-19,539 HNIR-1057 - AND CASE 8-19,530

There is no objection from a patent point of view to the publication of paper entitled "Application of Ion-Exchange to the Recovery, Purification, and Concentration of Plutonium and of Meptunium", written by E.J. Weelwright for a presentation at the Catlinburg Ion Exchange Conference, Gatlinburg, Tennessee, September 7-9, 1959.

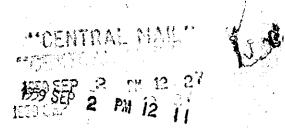
**WCPies** 

OT 16-58 FARCIGE RICH, AND WASH

oe: Mr (5)

"BE SAFE - BE WISE - ENJOY ANOTHER SAFETY PRIZE"

BUSH



TUX NR 91

/PRIORITY/

FROM ROLAND A ANDERSON USAEC WASHDC SEPT 021955Z

TO W C POE GEN ELEC CO RIGHLAND WASH

INFO GEORGE H LEE USAEC COO LEMONT ILL

TWX-4

NO OBJECTION TO PUBLICATION OF PAPER QUOTE APPLICATION OF ION-EXCHANGE TO THE RECOVERY CMM PURIFICATION AND CONCENTRATION OF PLUTONIUM AND NEPTUNIUM UNQUOTE BY E J WHEELWRIGHT END/REF GCP CLN RAA AEC 91

ANO HA

B ....

F 001 -705A PDT 9/2/59-WSA016-RLND ENDA D7 9/2

MR. W. C. POE - CONTRACT ENGINEER

GE/HAPO

· CENTRAL BURN

150 CEP (2 14 7 24

THERE APPEARS TO BE NO COMPANY PATENT OBJECTION TO THE PUBLICATION OF WHEELWRIGHT'S PROPOSED SPEECH BASED ON HWIR-1051 AND HWIR-1057.

P R WEBB, II ANPD

August 27, 1959

Roland A. Anderson, Assistant G neral Counsel for Patents

George H. Lee, Chief Chicago Patent Group

PATENT REVIEW PAPER APPLICATION OF ION-EXCHANGE TO THE RECOVERY, PURIFICATION AND CONCENTRATION OF PLUTONIUM AND OF HAPNIUM; CASES \$-19,539 AND \$-19,530

SYMBOL: PD: JWF

We have completed a patent review of the above paper which was forwarded to us by Mr. Poe for patent review. As stated in the General Electric release form, this paper discloses material relevant to Case S-19,530 authorized but not prepared. Material relevant to this case was reported in the declassification review of Quarterly Literature Review Reactor Fuel Processing, November 1950 to January, 1959", memorandum of March 30, 1959, and in HW-55893 reviewed in our memorandum of May 29, 1959. The first paper was released by your memorandum of April 3, 1959. The declassification was authorized with deletions on June 25, 1959. There is also material relevant to Case S-19,539. A Preliminary Disclosure was forwarded to you on January 25, 1959, with a generally unfavorable recommendation. The material of this case was shown in the Quarterly Literature Review Reactor Fuel Processing, February to April, 1959, covered by our memorandum of June 5, 1959. This was declassified with deletions on June 17, 1959. Our discussion of the novelty in this case did not take into consideration the severity of the nitric acid concentration environment on the reducing agents. This is discussed on page 24 of the presently considered report. This should modify our recommendation to a more favorable one.

We feel that there should be no objection from a patent standpoint to the release of the present document in view of the material released so far. We also feel that foreign filing would not be warranted in any case. Please note the urgency of Mr. Poe's request for clearance.

CC: W. C. Poe, Hanford

P. S. Please note that the deletions made in declassifying the above report did not remove the material pertinent to the above cases.

Mr. George H. Lee, Chief Chicago Patent Group U. S. Atomic Emergy Commission P. G. Box 59 Lement, Illinois

Dear Mr. Loe:

#### HVIR-1051 - AEC-CASE 5-19,539 HVIR-1057 \* AEC CASE 5-19,530

We have a request for approval of publication of the attached paper, entitled Application of Ion-Exchange to the Recovery, Purification, and Concentration of Plutonium and of Reptunium, written by E. J. Wheelwright for a presentation at the Gatlinburg Ion Exchange Conference, Gatlinburg, Termessee, September 7-9, 1959.

We would appreciate receiving your approval, if possible, of publication of the enclosed paper by not later than September 1, 1959.

Very truly yours,

Contract Engineer

WC Pueses

813C .

cc: Mr. Roland A. Anderson v/ enc.

bcc: Paul R Webb

File LB T

MR. WEBB: We would also appreciate receiving your approval. Our folder on Cases HWIR-1051 and HWIR-1057 are enclosed for your review and should be returned to us. WCP

Sarl J. Wheelwright 325 Building 300 Area

#### HWIR-1057 - AEC CASE 6-19, 30

I thought you would like to know that the Atomic Energy Commission has advised me that they are going to prepare a patent application based on your invention report, entitled "The use of fluoride to increase the separation of plutonium from fission products by nitric acid anion exchange process.

I vill advise you as soon as the application has been prepared and is ready for your approval and signature.

CRIGINAL SIGNED BY

W.C. POE Contract Engineer

WC Poetes

## UNITED STATES ATOMIC ENERGY COMMISSION



WASHINGTON 25, D. C.

July 17, 1959

GCP:RAA

Mr. W. C. Poe, Contract Engineer Contract Unit, Manufacturing Department General Electric Company Richland, Washington

Subject: AEC CASE S-19,530 - HWIR NO. 1057

Dear Mr. Poe:

This is to advise that this office is undertaking the preparation of an application on the above-identified disclosure which was received with Mr. Lee's memorandum of December 29, 1958, bearing the above-identified HWIR number.

Very truly yours,

Roland A. Anderson Assistant General Counsel

Roland a. Candenna

for Patents

cc: Mr. George H. Lee



1

## Cincinnati, Chic February 25, 1959

Mr. Roland A. Andersen Chief, Patent Branch U. S. Atomic Energy Commission Washington 25, D. C.

SUBJECT: General Electric Company, Hanford Atomic Products Operation, Invention Reports

Dear Mr. Anderson :

As a result of a recent meeting at which Hanford Atomic Products Operation invention reports were reviewed, the following suggestions are advanced.

The invention reports listed below are believed to disclose developments of sufficient significance in the field of atomic energy, if novel, to warrant a review by the Commission as to the advisability of filing domestic and foreign patent applications based thereon.

HWIR NO.	AEC CASE NO.	TITLE	INVENTOR
<b>\_1057</b>	8-19,530	Use of Fluoride to Increase Separation of Flutenium from Fission Products by the Nitric Acid Exchange Process	E. J. Wheelwright
1068	8-19,534	Fuel Element for Neutronic Reactors	Bates et al

Very truly yours,

Original Signed By P. R. WEBB, II PAUL R. WEBB, II

PRW, II idyh

ec: Mr. W. C. Poe

Paul R. Webb, II
Patent Counsel
Aircraft Nuclear Propulsion Dept.
Cincinnati 15, Ohio

## HWIR-1057 - AEC CASE S-19,530

Reference is made to your memo of January 21, 1959 concerning subject case.

It is noted that you are of the opinion that this case should be reviewed at an invention review meeting before you write to Mr. R. A. Anderson. Under our present system of reviewing HAPO submits, the Patent Panel does not review cases until after the AEC has advised us that the Commission is not interested in filing an application.

Mr. Rohrmann is a member of the HAPO Patent Panel, representing Hanford Laboratories, and has recommended domestic and foreign filing of the above-mentioned case by the AEC.

We would appreciate it if you would submit your recommendations to Mr. R. A. Anderson.

Contract Engineer

WC Poe:gs
Enc.--Folder HWIR-1057
cc: File
LB
T

ATOMIC PRODUCTS DIVISION
1 River Rd., Schenectady 5, N. Y.

January 21, 1959

Mr. W. C. Poe, Contract Engineer Hanford Atomic Products Operation General Electric Company Richland, Washington

SUBJECT: HWIR-1057 - AEC Case S-19,530

Dear Mr. Poe:

Reference is made to Mr. D. S. Cameron's letter of December 30, 1958 to Mr. L. B. Mackey requesting concurrence in Mr. Charles R. Rohrmann's recommendation of domestic and foreign filing by the Atomic Energy Commission of the above-identified invention report.

After this invention report has been considered at an invention review meeting, I shall be happy to suggest to Mr. R. A. Anderson that consideration be given to the preparation of domestic and foreign patent applications based on the above invention report.

Very truly yours,

and R. Well, II

PAUL R. WEBB, II

PRW:hma

Attachment

December 30, 1958

L. B. Mackey
Patent Counsel
Atomic Products Division
General Electric Company
1 River Road
Schenectady 5, New York

## HWIR-1057 - AEC CASE S-19,530

As suggested by Charles R. Rohrmann, Patent Panel Representative of HIO, we are enclosing the above-mentioned folder for your review relative to recommended domestic and foreign filing by the AEC.

Please return the folder to us when you have completed your review.

Specialist, Contracts

Douglas S. Cameron:gs

Enc.

cc: File

LB

T

# UNITED STATES ATOMIC ENERGY COMMISSION

CHICAGO OPERATIONS OFFICE
P. O. Box 59
Lemont, Illinois

December 17, 1958

Mr. W. C. Poe, Contract Engineer Relations & Utilities Operation Jeneral Electric Company Richland, Washington

Subject: HwIR-1055 AEC-S-19,528 HwIR-1056 AEC-S-19,529 HwIR-1057 AEC-S-19,530

Dear Mr. Poe:

We are in receipt of your letters and Invention Reports in triplicate relative to HWIR-1055, 1056, and 1057 and we have assigned AEC-S-19,528, S-19,529, S-19,530 thereto.

We will notify Mr. Anderson's office in Washington of these cases by sending him two copies each of your letters and invention Reports together with our recommendations at a later date.

Very truly yours,

Jeorge d. Lee, Chief Chicago Patent Group Mr. George H. Lee, Chief Chicago Patent Group U. S. Atomic Energy Commission P. O. Box 59 Lemont, Illinois

Dear Mr. Lee:

Enclosed are three copies of Hanford Report of Invention, G. E. Case HVIR-1057. (UNCLASSIFIED HW-58327).

Very truly yours,

Contract Engineer

WC Poetga

Locs.

ce: HOO - ABC

Attn: Process Eng. & Mrg. Div.

bcc: Records Center

File LB

MW-58327 Dec.2,1958

54-3000-039 (9-56) AEC-GE RICHLAND, WASH.

7 - W.H.Reas 8 - M.T.Walling,Jr.

HANFORD ATOMIC PRODUCTS OPERATION 9 - E.J. Wheelwright GENERAL ELECTRIC COMPANY 10 - 300 File

RICHLAND, WASHINGTON

11 - Records Center

#### REPORT OF INVENTION

A.E.C.CASE NO. - 19,530 G. E. CASE NO.

TO:

1 : ATTACHED HERETO IS A DESCRIPTION OF WHAT MAY BE AN INVENTION IN:

The Use of Fluoride to Increase the Separation of Plutonium from Fission Products by the Nitric Acid Anion Exchange Process.

II: THE NAME, TITLE OR POSITION, WORKS LOCATION, AND PERMANENT ADDRESS OF THE INVENTOR(S) IS:

Earl J. Wheelwright

Chemist I

325 Building, 300 Area

Area

613 Catskill

Richland, Washington

III: EVIDENCE AS TO WHEN AND WHERE THE INVENTION WAS MADE CAN BE FOUND IN THE FOLLOWING LISTED WRITTEN OR PICTORIAL MATERIAL (NOTEBOOK, FILE REPORTS OR DRAWINGS, ETC. ):

HWN-1899, pages 96 to 135, inclusive. (Secret)

[V: THE APPROXIMATE DATE OF THE FIRST ENTRY IN SAID WRITTEN OR PICTORIAL MATERIAL DESCRIBING OR SHOWING SAID INVENTION IS:

June 12, 1958.

V. PERSONS WHO COULD TESTIFY AS TO WHEN AND WHERE THE INVENTION WAS MADE INCLUDE THE FOLLOWING:

Jack L. Ryan

Ward L. Lyon

M.T. (Day Quic V. HIDILY HI

## DESCRIPTION OF INVENTION

#### Introduction

A process wherein plutonium(IV) is concentrated and separated from various metallic and non-metallic impurities, including fission products, is described in the literature<sup>1,2,3</sup>. This process consists of three steps:

- (1) Plutonium(IV), as an anionic nitrate complex ion, is absorbed by a strong base anion exchange resin from a strong nitric acid or nitric acid metal nitrate solution.
- (2) The plutonium laden resin is then washed with a strong nitric acid or nitric acid metal nitrate washing solution to remove the unabsorbed or weakly absorbed impurities from the resin bed.
- (3) The plutonium(IV) is eluted from the resin by passage of a dilute nitric acid solution through the resin bed.

In the event that the plutonium solution charged to the anion exchange unit contains appreciable quantities of the long-lived fission products such as zirconium-95 and niobium-95, the resin becomes highly contaminated with these fission products during the absorption cycle. The fission products are not completely removed even by a very extensive nitric acid washing cycle. A major fraction of those remaining on the resin at the end of the washing cycle are removed with the plutonium during the elution cycle. The mechanism by which the fission products are retained on the resin throughout the washing cycle is

3. Aiken, A. M., Chem. Eng. Prog., 53, No. 2, 82F (1957).

Carl Mulwright 12/9/50 Date

Read, and understood by me this \_\_\_\_\_\_\_, 1958.

Withess Y. Styan

Ward & Lyon Witness

<sup>1.</sup> Ryan, J. L., and Wheelwright, E. J., Application of Anion Exchange to the Reprocessing of Plutonium, Proceedings, Second International Conference on the Peaceful Uses of Atomic Energy, A/CONF. 15/P/1915 (1958).

<sup>2.</sup> Ryan, J. L., and Wheelwright, E. J., The Recovery, Purification and Concentration of Plutonium by Anion Exchange in Nitric Acid, HW-55893, April 25, 1958 (Secret).

not completely understood, but may be physical entrapment by the resin as it contracts during the absorption cycle or polymer formation within the resin beads.

The claim of this invention is that the addition of small quantities of fluoride to the nitric acid wash solution and, or, to the plutonium feed solution increases the rate at which the fission products can be removed from the plutonium laden resin and therefore significantly lowers the fission product activity in the plutonium product eluted from an anion exchange resin.

## The Effect of Adding Fluoride to the Wash

The data of Table I show the effect on the separation of plutonium from fission products produced by the addition of various concentrations of fluoride to part of the wash solution. The fission product activity in the feed for each of the six runs was approximately constant (Zr-Nb =  $10^{\circ}$  6/m, ml; Ru-Rh =  $10^{\circ}$  6/m, ml). The plutonium concentration varied from 0.4 to 0.6 grams per liter. The addition of fluoride to the wash solution decreased the fission product contamination of the product by a factor of 100.

Some plutonium will be eluted from the resin when a resin column which has been loaded to breakthrough is washed with a nitric acid solution containing fluoride. This slight plutonium elution and any corrosion which might be caused by low level fluoride ( $\leq 0.05$  M HF) can be minimized by the addition of moderate amounts of aluminum nitrate to the solutions which contain fluoride. Plutonium elution can be eliminated by operating the absorption cycle such that a band of unloaded resin remains downstream from the loaded resin at the completion of the absorption cycle.

## The Addition of Fluoride to Anion Exchange Feed Solution

The essential difference between runs 1 and 2 of Table II is that the feed for run 2 contained 0.0056 M HF while the feed for run 1 contained no fluoride. The zirconium-niobium activity,  $3 \times 10^7$  M/m, ml, of these feed solutions was a factor of two higher than the activity of the feed solutions for the runs shown

Jaco

Ward J. Jyon Witness in Table I. The zirconium-niobium activity in the product of run 2 is lower than that for run 1 by a factor of almost two. Even more significant is the fact that the zirconium-niobium activity of the wash solution after the first 14 column volumes was lower for run 2 by a factor of 5.6 compared to run 1. This means that the same product purity could be achieved with significantly less wash solution when some fluoride is added to the feed solution.

Earl Whelwright 13/9/58
Inventor Date

Witness
Ward L. Lyon

EJW/ffs

TABLE I. FLUORIDE WASH CYCLE

		Wash Cycle		Elu	Elution Cycle	a	,		Product Purity	Purity
	Column		Average Flow	Average Flow	Pu	ė	Fission Product Decontamination	Product ination	micro cur Ruapha di	micro curies impurity) Ruspha disintegrations
Run No.	Volumes of Wash	Wash Solution Composition	kate min.,cm2)	$\frac{m1}{(\pi i n., cm^2)}$	Froduct Conc. (gms/1)	ru Conc. Factor	Zr-Nb	Ru-Rh	Zr-Nb (x10-11)	Ru-Rh (x10-11)
4	32	7.2 M HNO3	5.5	1.1	50	120	186 186	6,200	12	0.45
Q	₹	7.2 M HNO3	5.3	1.2	50	120	1,210	13,000	10	0.20
$\circ$	28	7.2 M ENO3	8.4	0.5	20	85	604	336	16	0.25
<b>†</b>	7 7 8	7.2 M HNO3 7.2 M HNO3; 0.056 M HF 7.2 M HNO3	9.4	6.0	<b>ग्</b> ष	98	190,000	10,000	0.05	<0.05
<u>r</u>	17 18 6	7.2 M HNO3 7.2 M HNO3; 0.01 M HF 7.2 M HNO3	5.2	η.ς	64	77	25,700 38,500	38,500	0.05	<0.05
9	14 22 6	7.2 M HNO3 7.2 M HNO3; 0.0056 M HF 7.2 M HNO3	£.	9°0	35	62	27,000	27,000 10,000	0.25	<0.05
Rest	n: Perm ating Ter	Resin: Permutit SK 20-50 mesh Operating Temperature: 60 C						•	,	

2 Operating Temperature: day of Ber

Read and understood by me this

, 1958 /

Witness

THE EFFECT OF FLUORIDE ADDED TO FEED TABLE II.

Purito	micro curies impurity) (Pu alpha disintegrations) Zr-Nb Ru-Rh (x10-11) (x10-11)	<0.05	<0.05
Product Parity	micro cur Fu alpha d Zr-Nb (x10-11)	0.35	0.20
	Fission Product Decontamination Factor Zr-Nb Ru-Rh	1	!
	Fission Decontured Factor	112 70,000	000 <b>'</b> 98 †6
•	Pu Conc. Factor	112	46
Elution Cycle	Pu Product Pu Conc. Cor (gms/1) Fac	44	24
	Average Flow Rate min.,cm <sup>2</sup> )	3.1	ج 2•
	Average Flow Rate min.,cm <sup>2</sup> )	5.2	4.5
Wash Cycle	Wash Solution Composition	7.2 M HNO3 7.2 M HNO3; 0.01 M HF 7.2 M HNO3	7.2 M HNO3 7.2 M HNO3; 0.01 M HF 7.2 M HNO3
	Column Volumes of Wash	15 18 6	17 6 6
	Run No.	-	Q

Resin: Permutit SK 20-50 mesh Operating Temperature: 60 C

. day of 194 Read and understood by me this



RICHLAND, WASHINGTON . . . . . . . . . . . . . . . HANFORD ATOMIC PRODUCTS OPERATION

December 12, 1958

Mr. George H. Lee, Chief Chicago Patent Group U. S. Atomic Energy Commission P. O. Box 59 Lemont, Illinois

Dear Mr. Lee:

Enclosed are three copies of Hanford Report of Invention, G. E. Case HWIR-1057. (UNCLASSIFIED HW-58327).

Very truly yours,

Contract Engineer

WC Poetgs

Encs.

cc: HOC - AEC

Attn: Process Eng. & Mfg. Div.

54-3000-039 (9-56) AEC-GR RICHLAND, WASH.

7 - W.H.Reas

Dec.2,1958

8 - M.T.Walling, Jr.

## HANFORD ATOMIC PRODUCTS OPERATION 9 - 15.J. Wheelwright GENERAL ELECTRIC COMPANY 10 - 300 File

RICHLAND, WASHINGTON

11 - Records Center

## REPORT OF INVENTION

A.E.C.CASE NO.

G. E. CASE NO.

49118

TO:

#### I : ATTACHED HERETO IS A DESCRIPTION OF WHAT MAY BE AN INVENTION IN:

The Use of Fluoride to Increase the Separation of Plutonium from Fission Products by the Nitric Acid Anion Exchange Process.

II: THE NAME, TITLE OR POSITION, WORKS LOCATION, AND PERMANENT ADDRESS OF THE INVENTOR(S) IS:

Earl J. Wheelwright Chemist I 325 Building, 300 Area

613 Catskill Richland, Washington

III. EVIDENCE AS TO WHEN AND WHERE THE INVENTION WAS MADE CAN BE FOUND IN THE FOLLOWING LISTED WRITTEN OR PICTORIAL MATERIAL (NOTEBOOK, FILE REPORTS OR DRAWINGS, ETC.):

HWN-1899, pages 96 to 135, inclusive. (Secret)

IV: THE APPROXIMATE DATE OF THE FIRST ENTRY IN SAID WRITTEN OR PICTORIAL MATERIAL DESCRIBING OR SHOWING SAID INVENTION IS:

June 12, 1958.

Y. PERSONS WHO COULD TESTIFY AS TO WHEN AND WHERE THE INVENTION WAS MADE INCLUDE THE FOLLOWING:

Jack L. Ryan Ward L. Lyon

ward L. Lyon

M. T. Walling, Y.

DATE

DEPARTMENT

14/0/41

HLC

## DESCRIPTION OF INVENTION

#### Introduction

A process wherein plutonium(IV) is concentrated and separated from various metallic and non-metallic impurities, including fission products, is described in the literature<sup>1,2,3</sup>. This process consists of three steps:

- (1) Plutonium(IV), as an anionic nitrate complex ion, is absorbed by a strong base anion exchange resin from a strong nitric acid or nitric acid metal nitrate solution.
- (2) The plutonium laden resin is then washed with a strong nitric acid or nitric acid metal nitrate washing solution to remove the unabsorbed or weakly absorbed impurities from the resin bed.
- (3) The plutonium(IV) is eluted from the resin by passage of a dilute nitric acid solution through the resin bed.

In the event that the plutonium solution charged to the anion exchange unit contains appreciable quantities of the long-lived fission products such as zirconium-95 and niobium-95, the resin becomes highly contaminated with these fission products during the absorption cycle. The fission products are not completely removed even by a very extensive nitric acid washing cycle. A major fraction of those remaining on the resin at the end of the washing cycle are removed with the plutonium during the elution cycle. The mechanism by which the fission products are retained on the resin throughout the washing cycle is

3. Aiken, A. M., Chem. Eng. Prog., 53, No. 2, 82F (1957).

Earl Meelwright 12/8/5

Read and understood by me this 9 day of December , 1958

Witness T. Ryan

Ward L. Xyou
Witness

Ryan, J. L., and Wheelwright, E. J., <u>Application of Anion Exchange to the Reprocessing of Plutonium</u>, Proceedings, Second International Conference on the Peaceful Uses of Atomic Energy, A/CONF. 15/P/1915 (1958).

<sup>2.</sup> Ryan, J. L., and Wheelwright, E. J., The Recovery, Purification and Concentration of Plutonium by Anion Exchange in Nitric Acid, HW-55893, April 25, 1958 (Secret).

not completely understood, but may be physical entrapment by the resin as it contracts during the absorption cycle or polymer formation within the resin beads.

The claim of this invention is that the addition of small quantities of fluoride to the nitric acid wash solution and, or, to the plutonium feed solution increases the rate at which the fission products can be removed from the plutonium laden resin and therefore significantly lowers the fission product activity in the plutonium product eluted from an anion exchange resin.

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The data of Table I show the effect on the separation of plutonium from fission products produced by the addition of various concentrations of fluoride to part of the wash solution. The fission product activity in the feed for each of the six runs was approximately constant (Zr-Nb =  $10^{-6}$  /m, ml; Ru-Rh =  $10^{-6}$  /m, ml). The plutonium concentration varied from 0.4 to 0.6 grams per liter. The addition of fluoride to the wash solution decreased the fission product contamination of the product by a factor of 100.

Some plutonium will be eluted from the resin when a resin column which has been loaded to breakthrough is washed with a nitric acid solution containing fluoride. This slight plutonium elution and any corrosion which might be caused by low level fluoride ( $\leq 0.05$  M HF) can be minimized by the addition of moderate amounts of aluminum nitrate to the solutions which contain fluoride. Plutonium elution can be eliminated by operating the absorption cycle such that a band of unloaded resin remains downstream from the loaded resin at the completion of the absorption cycle.

## The Addition of Fluoride to Anion Exchange Feed Solution

The essential difference between runs 1 and 2 of Table II is that the feed for run 2 contained 0.0056 M HF while the feed for run 1 contained no fluoride. The zirconium-niobium activity,  $3 \times 10^7$  M/m, ml, of these feed solutions was a factor of two higher than the activity of the feed solutions for the runs shown

Read and understood by me this 9 day of December, 1958.

lack L. Rus

Ward L. Lyon
Witness

-4-

in Table I. The zirconium-niobium activity in the product of run 2 is lower than that for run 1 by a factor of almost two. Even more significant is the fact that the zirconium-niobium activity of the wash solution after the first 14 column volumes was lower for run 2 by a factor of 5.6 compared to run 1. This means that the same product purity could be achieved with significantly less wash solution when some fluoride is added to the feed solution.

Read and understood by me this 2 day of December, 1958.

EJW/ffs

TABLE I. FIUORIDE WASH CYCLE

Run No. 2	Column Volumes of Wesh 32 34 34 14 14 14 18 6	Wash Solution  Wash Solution  7.2 M HN03  7.2 M HN03  7.2 M HN03  7.2 M HN03; 0.056 M HF  7.2 M HN03; 0.01 M HF  7.2 M HN03; 7	Average Flow Rate (min., cm2) 5.5 5.3 4.8	Average Flow Rate ml (min., cm2) 1.2 0.5 0.9	Elution Cycle Pu Product Conc. 50 50 50 50 44	Pu Conc. Factor 120 120 85	Fission Product Decontamination Factor  Zr-Nb Ru-Rh 984 6,200 1,210 13,000 409 336 190,000 10,000	on Product tamination Factor Ru-Rh 10 13,000 09 336 00 10,000	Product Purity  micro curies in  Ru alpha disintegr  Zr-Nb (x10-11) (x10- 12 0.0 16 0.0 0.05 (0.0	Product Purity micro curies impurity) Ruapha disintegrations Zr-Nb (x10-11) (x10-11) 12 0.45 10 0.25 16 0.25 0.05 <0.05
	14 8 6	7.2 M HNO3 7.2 M HNO3; 0.0056 M HF 7.2 M HNO3	4.2	9°0	35	62	27,000	10,000	0.25	<0.05
Resin:	: Permu	Resin: Permutit SK 20-50 mesh								

Resin: Permutit SK 20-50 mes Operating Temperature: 60 C

Q Read and understood by me this

Witness

, 1958

day of

THE EFFECT OF FLUORIDE ADDED TO FEED TABLE II.

D.174 + 17	micro curies impurity).  (Fu alpha disintegrations).  Zr-Nb Ru-Rh  (x10-11) (x10-11)	<0.05	<0.05
Product Buritar	micro cui Pu alpha d Zr-Nb (x10-11)	0,35	0.20
	Fission Product Decontamination Factor Zr-Nb Ru-Rh	;	;
		70,000	94 86,000
	Pu Conc. Factor	112	76
Elution Cycle	Pu Product Conc. (gms/1)	71	24
	Average Flow Rate ml (min.,cm <sup>2</sup> )	3.1	ج. د.
	Average Flow Rate (mln.,cm2)	5.2	4,5
Wash Cycle	Wash Solution Composition	7.2 M HNO3 7.2 M HNO3; 0.01 M HF 7.2 M HNO3	7.2 M HNO3 7.2 M HNO3; 0.01 M HF 7.2 M HNO3
	Column Volumes of Wash	15 18 6	14 16 6
	Run	H	N

Resin: Permutit SK 20-50 mesh Operating Temperature: 60 C

, 1958. day of Read and understood by me this