HW-SA-2290

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54-3000-405 (9-59) AEC-GE RICHLAND, WASH.

CONTINUOUS ANION EXCHANGE PROCESSING OF PLUTONIUM*

G. C. Oberg and W. H. Swift

October 3, 1961

HANFORD ATOMIC PRODUCTS OPERATION

RICHLAND, WASHINGTON

GENERAL ELECTRIC

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

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.

(1) 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.

(2) 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 M HNO_3 and then decreases gradually at higher acidities. Temperature also affects the distribution with lower coefficients

(3) 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 Commerical 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 10^5 roentgens per hour. Since most elastomeric organic materials begin to show considerable radiation damage at levels of 10^8 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 3.4×10^8 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

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

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

(6) 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.

(7) 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 2×10^{-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

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

(9) "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

(10) 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 recylced via the slip-water stream (XSW) from the bottom of the elution column. In both cases, these streams are recylced 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

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.

DISTRIBUTION COEFFICIENTS OF Pu (IV)
ON DOWEX-1, X-4 (50-100 mesh) FROM
NITRIC ACID AND CALCIUM NITRATE

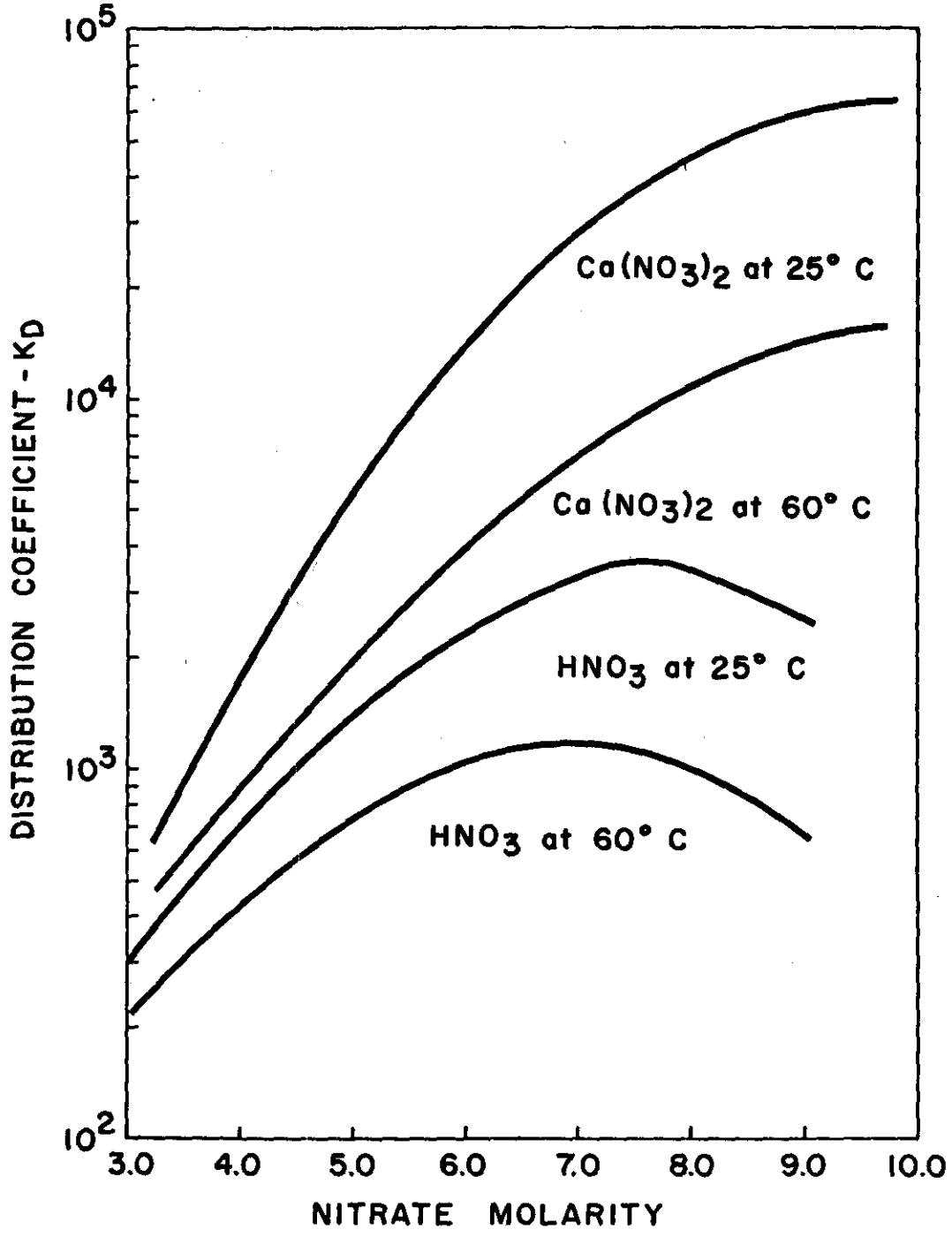


FIGURE 1

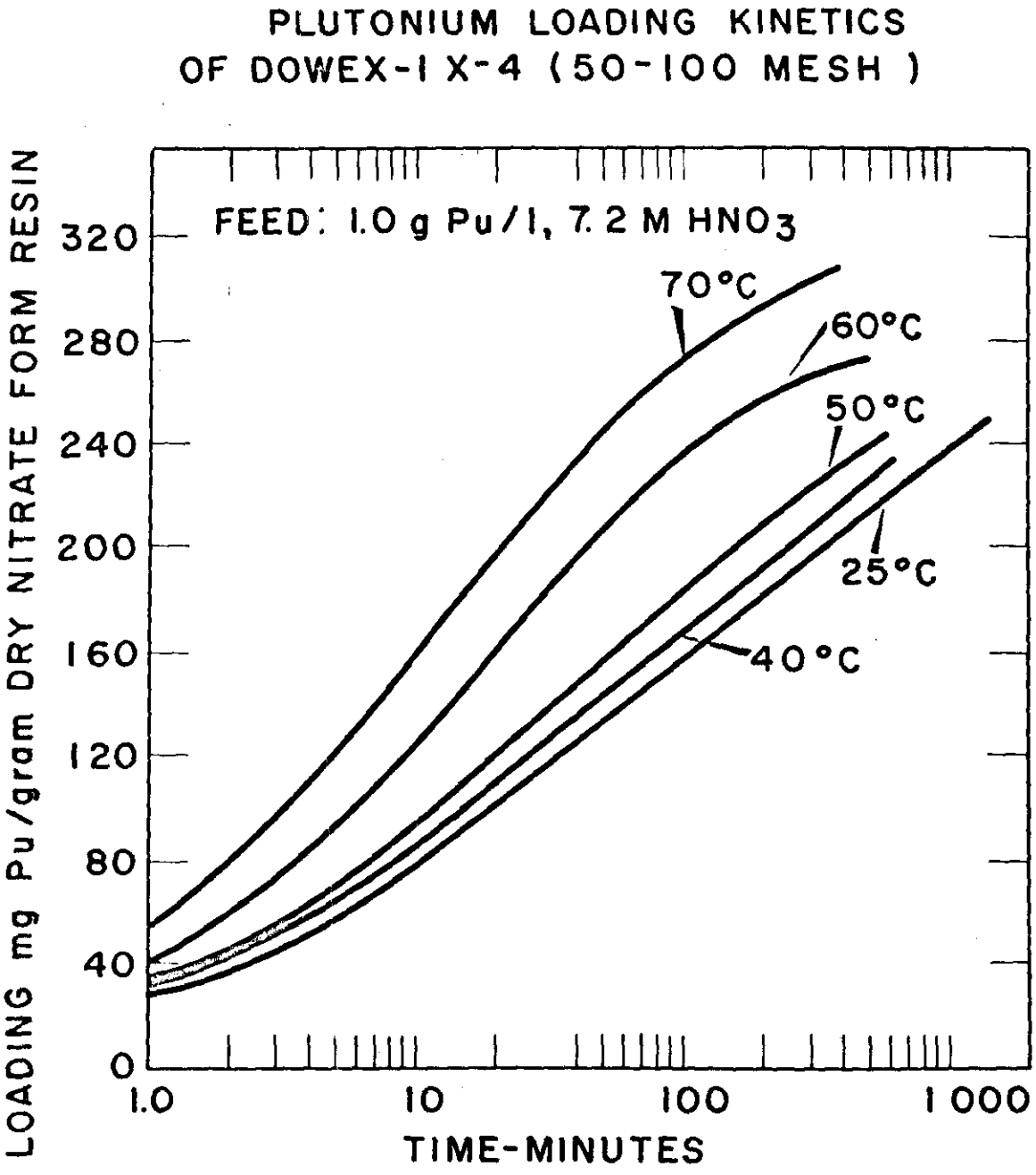

PLUTONIUM LOADING KINETICS
OF DOWEX-1 X-4 (50-100 MESH)
FEED: 1.0 g Pu/l, 7.2 M HNO3
70°C
60°C
50°C
25°C
40°C
LOADING mg Pu/gram DRY NITRATE FORM RESIN
320
280
240
200
160
120
80
40
0
1.0
10
100
1 000
TIME-MINUTES

FIGURE 2

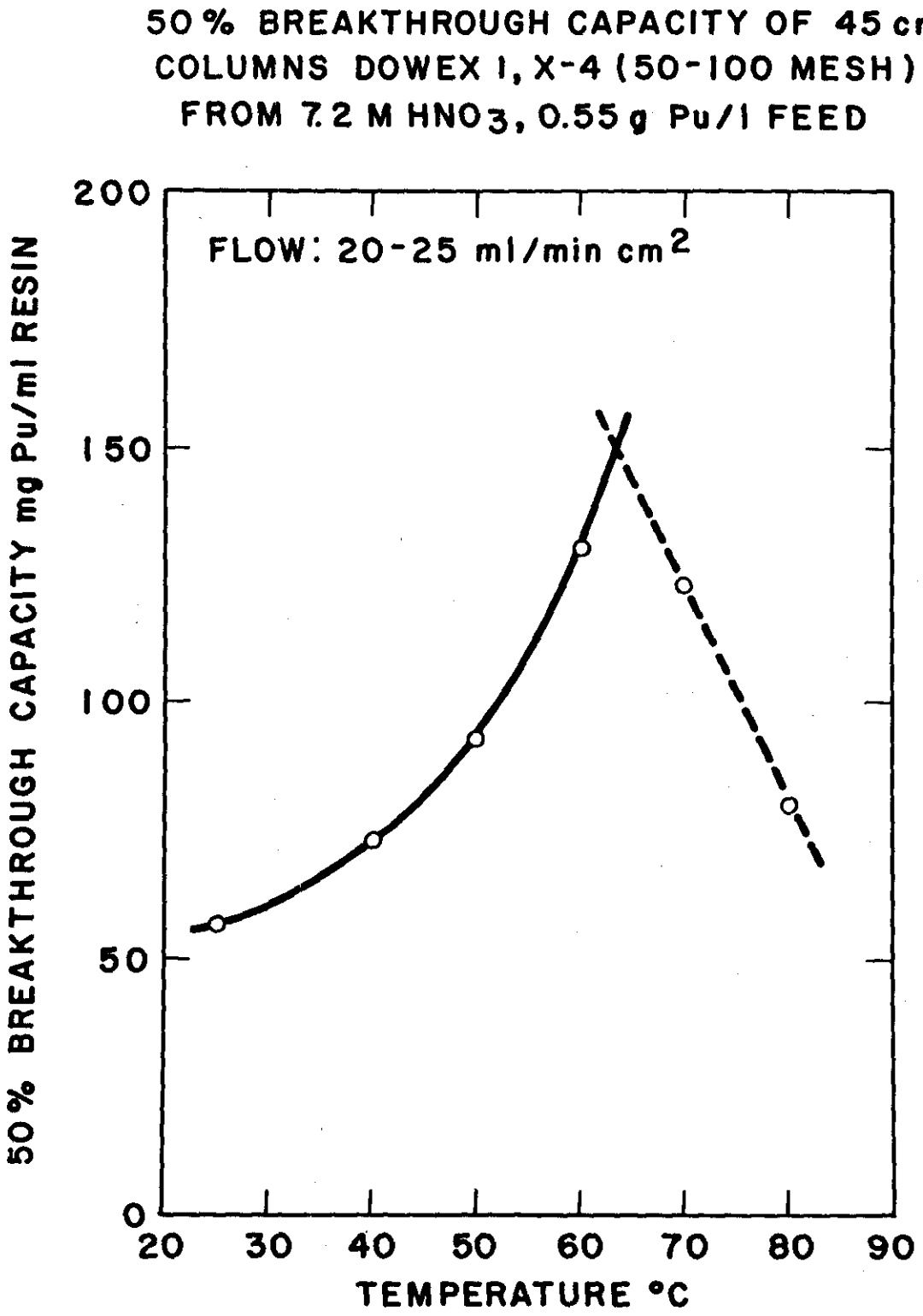

50% BREAKTHROUGH CAPACITY OF 45 cm
COLUMNS DOWEX 1, X-4 (50-100 MESH)
FROM 7.2 M HNO3, 0.55 g Pu/l FEED
FLOW: 20-25 ml/min cm2
50% BREAKTHROUGH CAPACITY mg Pu/ml RESIN
200
150
100
50
0
20
30
40
50
60
70
80
90
TEMPERATURE °C

FIGURE 3

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

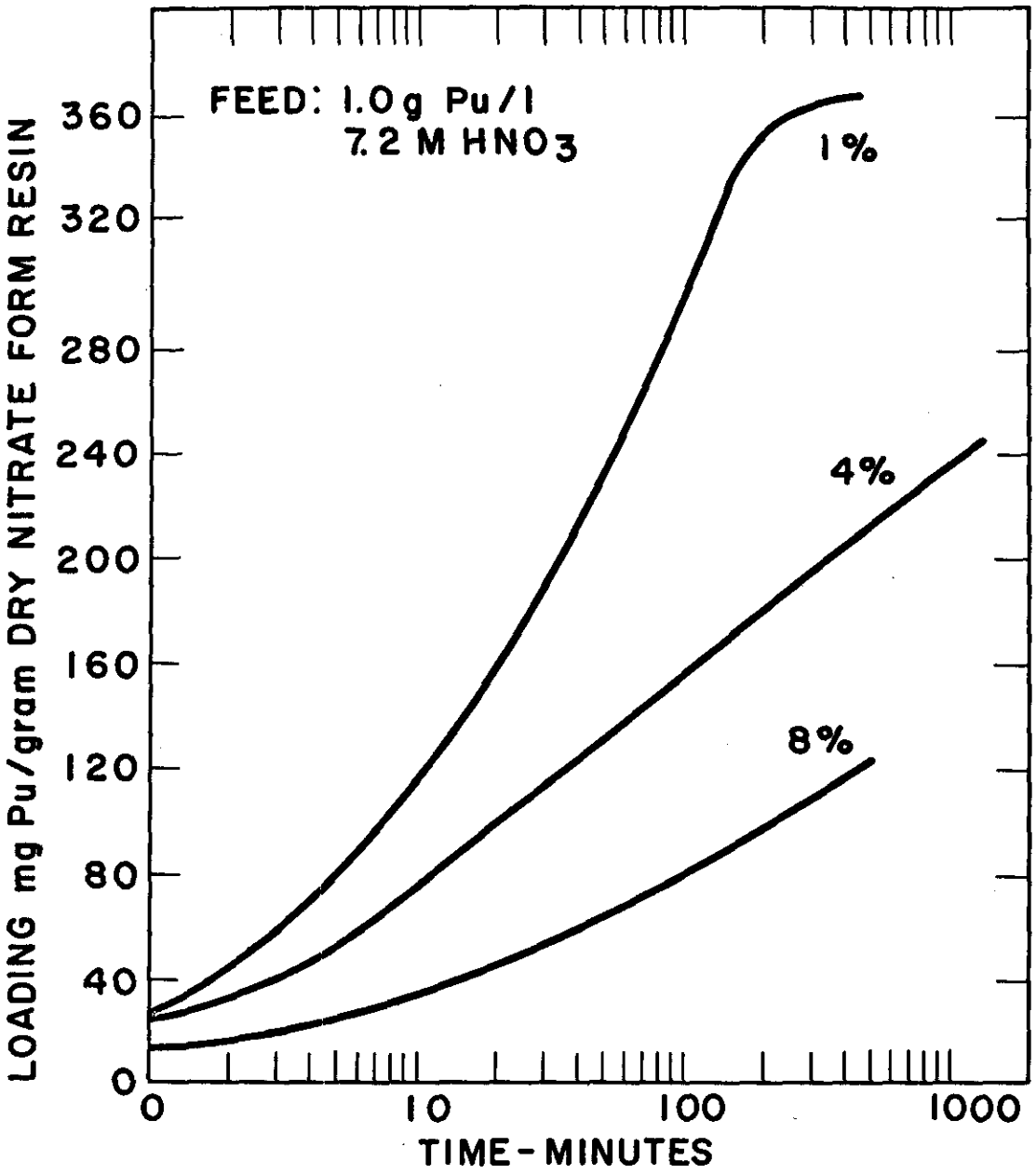


FIGURE 4

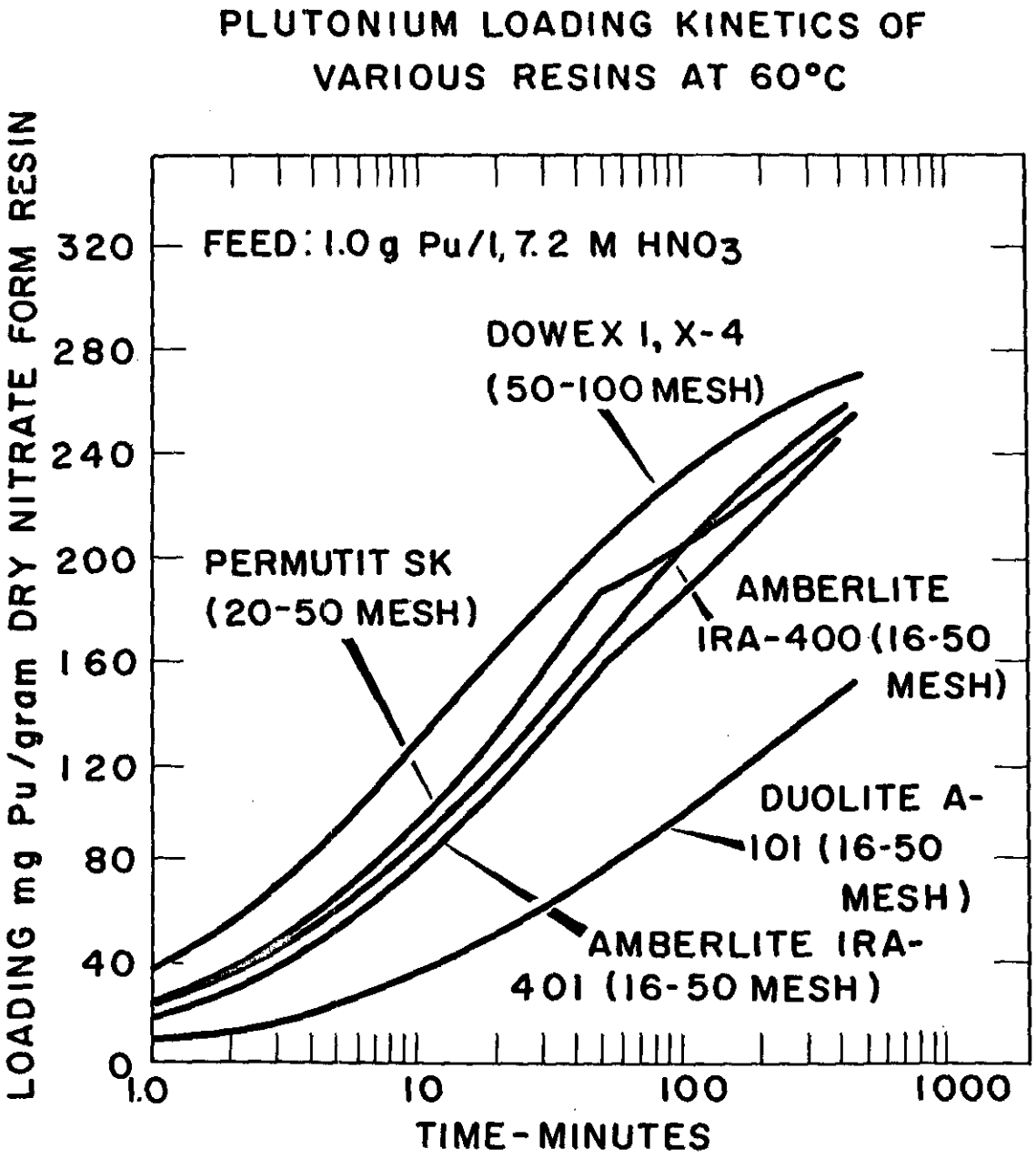

PLUTONIUM LOADING KINETICS OF
VARIOUS RESINS AT 60°C
FEED: 1.0 g Pu/l, 7.2 M HNO3
DOWEX 1, X-4
(50-100 MESH)
PERMUTIT SK
(20-50 MESH)
AMBERLITE
IRA-400 (16-50
MESH)
DUOLITE A-
101 (16-50
MESH)
AMBERLITE IRA-
401 (16-50 MESH)
LOADING mg Pu/gram DRY NITRATE FORM RESIN
0
40
80
120
160
200
240
280
320
1.0
10
100
1000
TIME-MINUTES

FIGURE 5

CONTINUOUS PLUTONIUM ANION EXCHANGE FLOW DIAGRAM

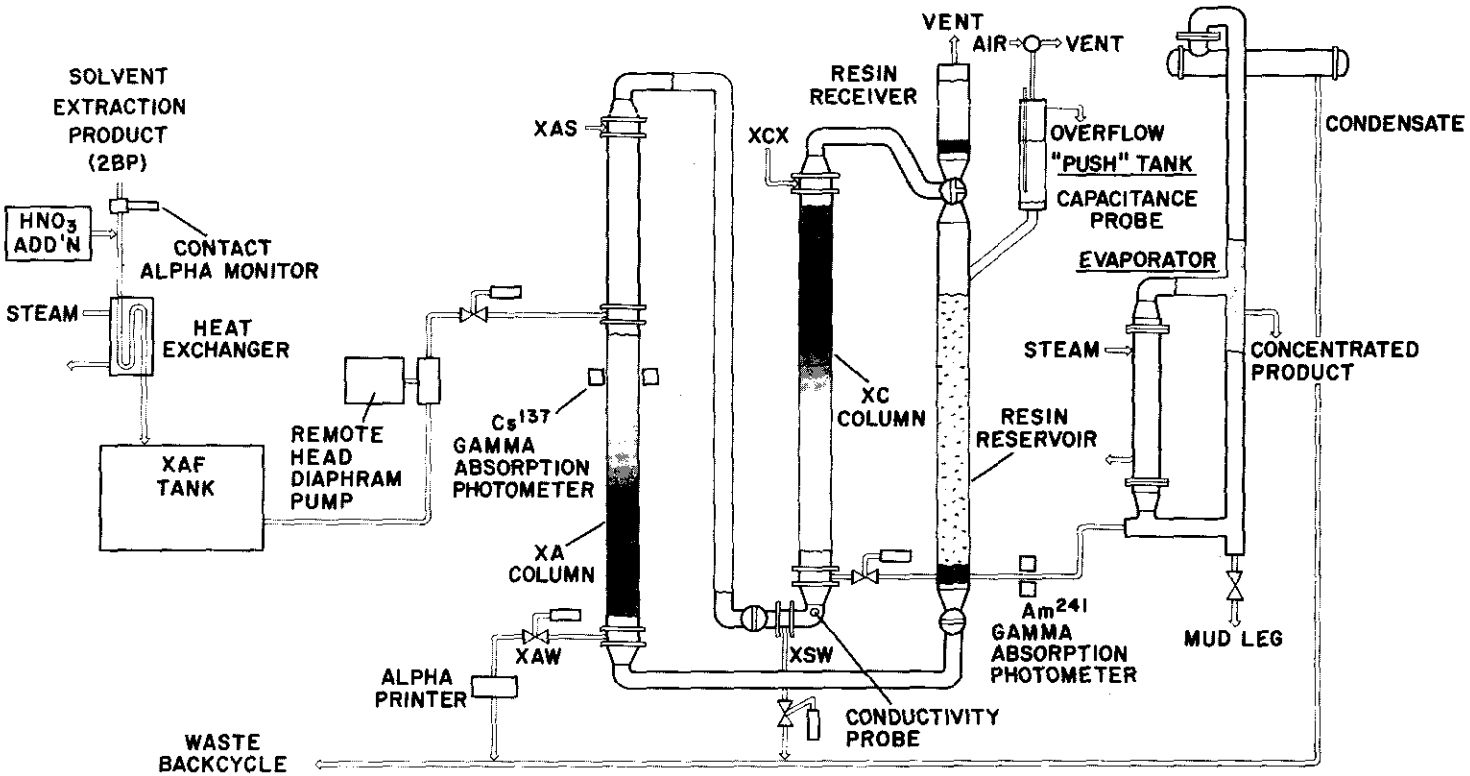
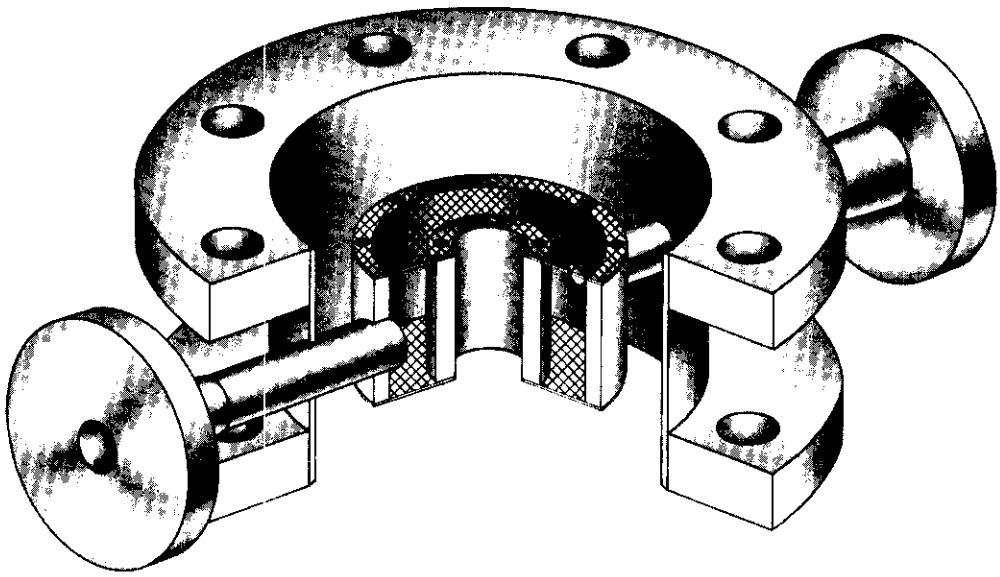


FIGURE 6

HW-SA-2290

ION EXCHANGE SOLUTION DISTRIBUTOR



ION EXCHANGE SOLUTION COLLECTOR

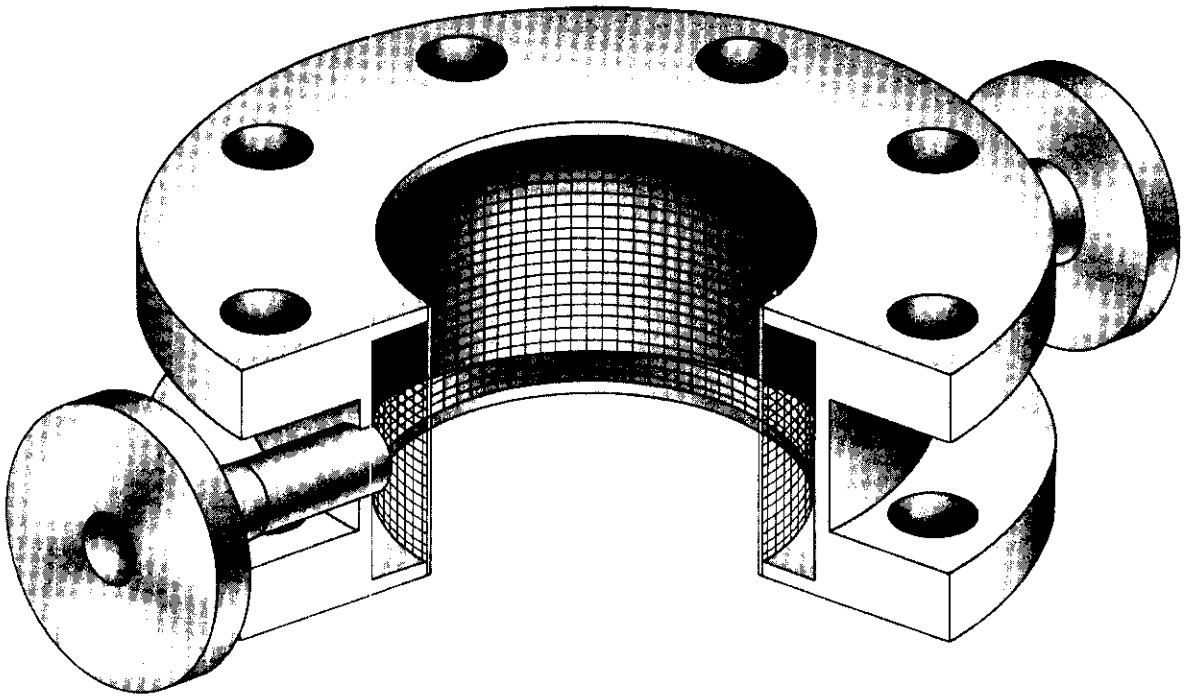


FIGURE 6A

AEC-GE RICHLAND, WASH.

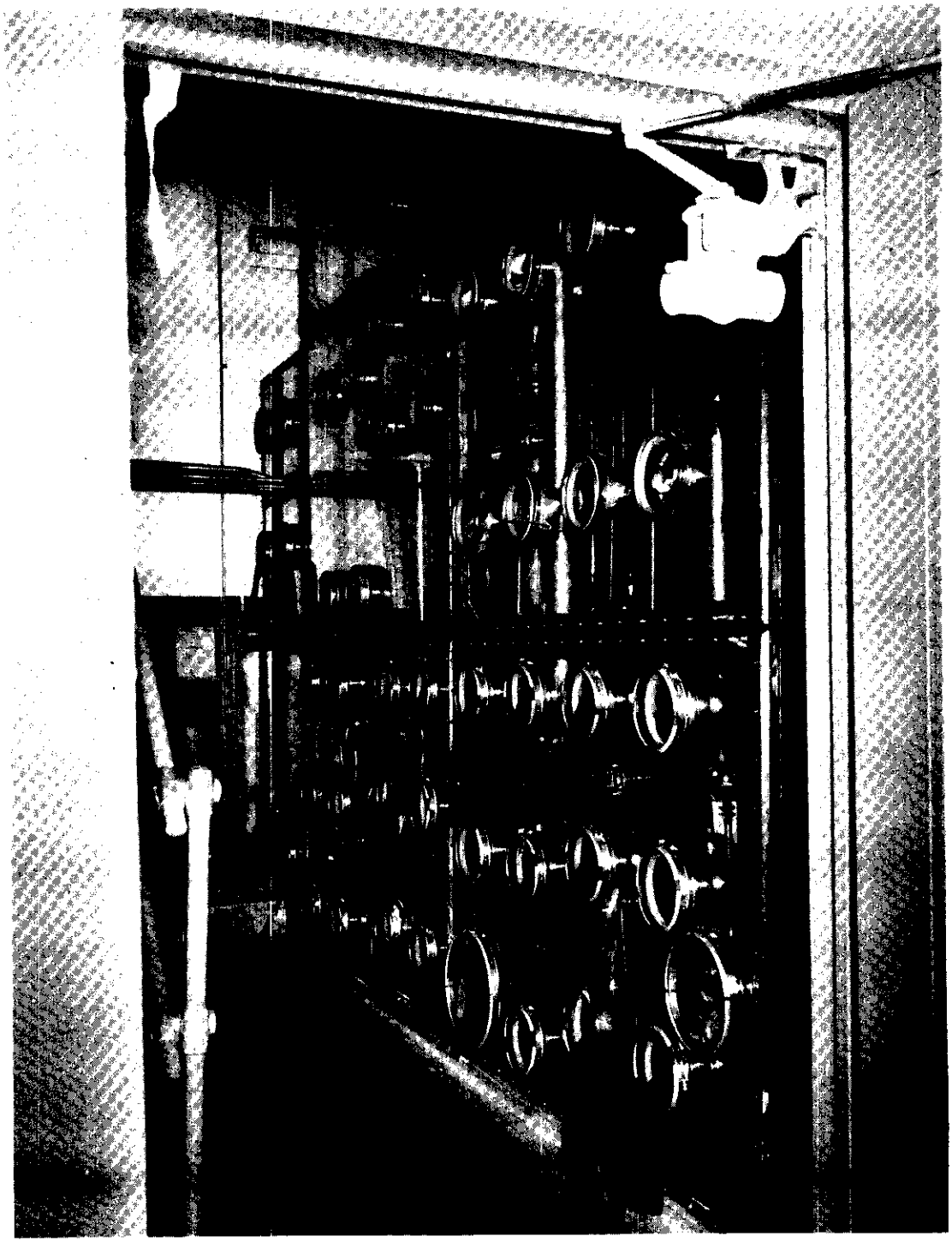


FIGURE 7

Lengthwise View of Anion Exchange Hood

AEC-GE RICHLAND, WASH.

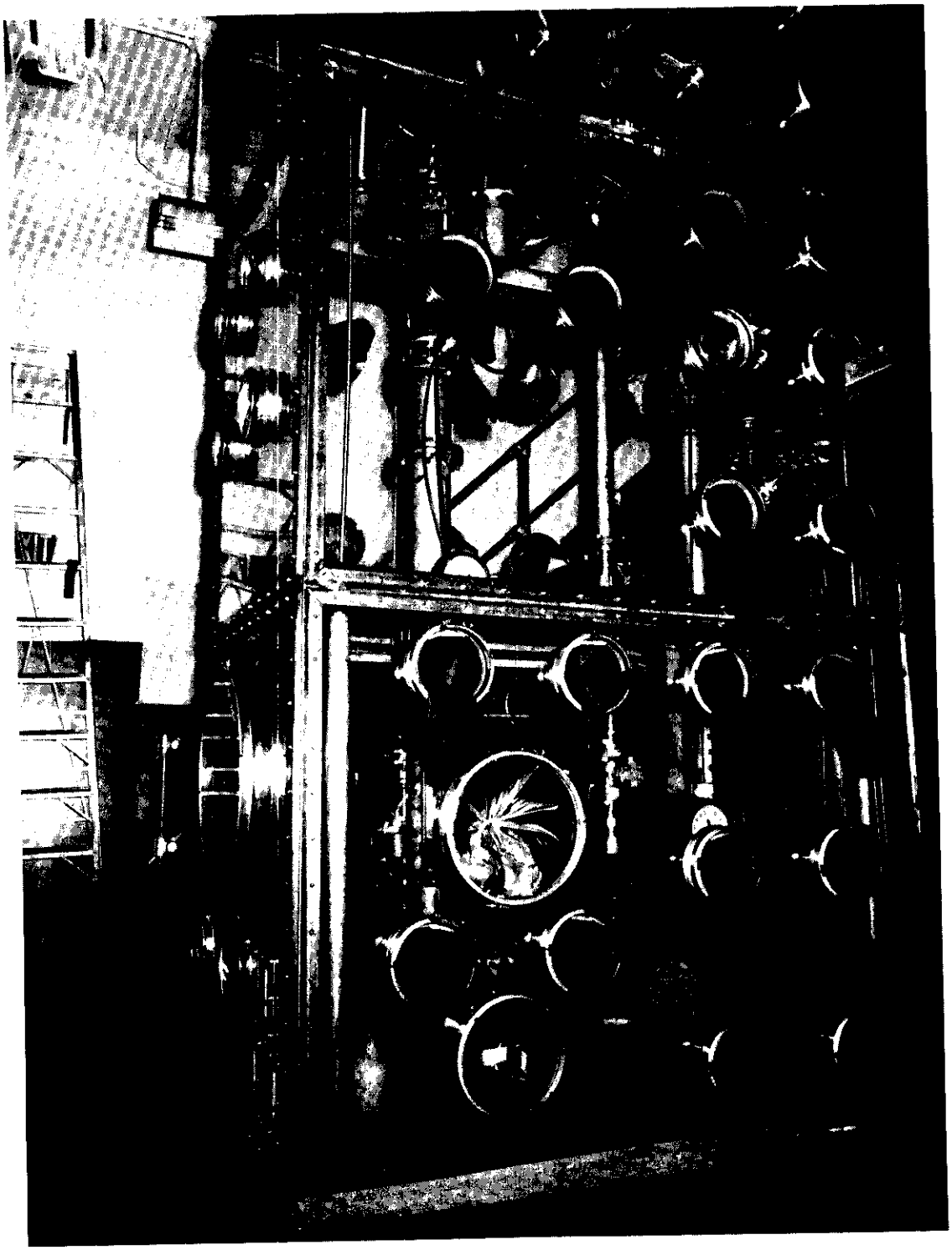


FIGURE 8

Transverse View of Anion Exchange Hood

AEC-GE RICHLAND, WASH.

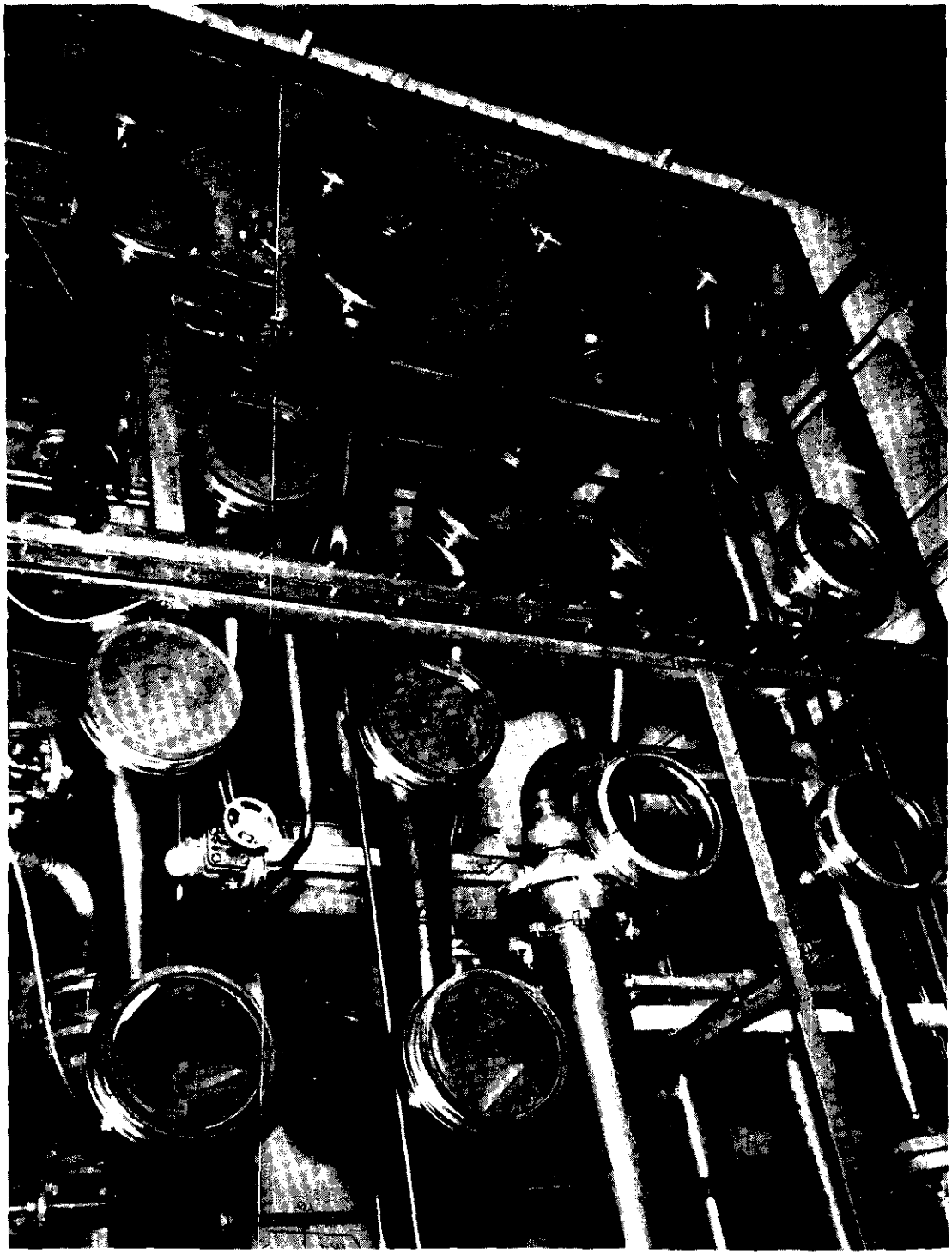


FIGURE 9

Anion Exchange Hood Showing Concentrator and Condenser

AEC-GE RICHLAND, WASH.

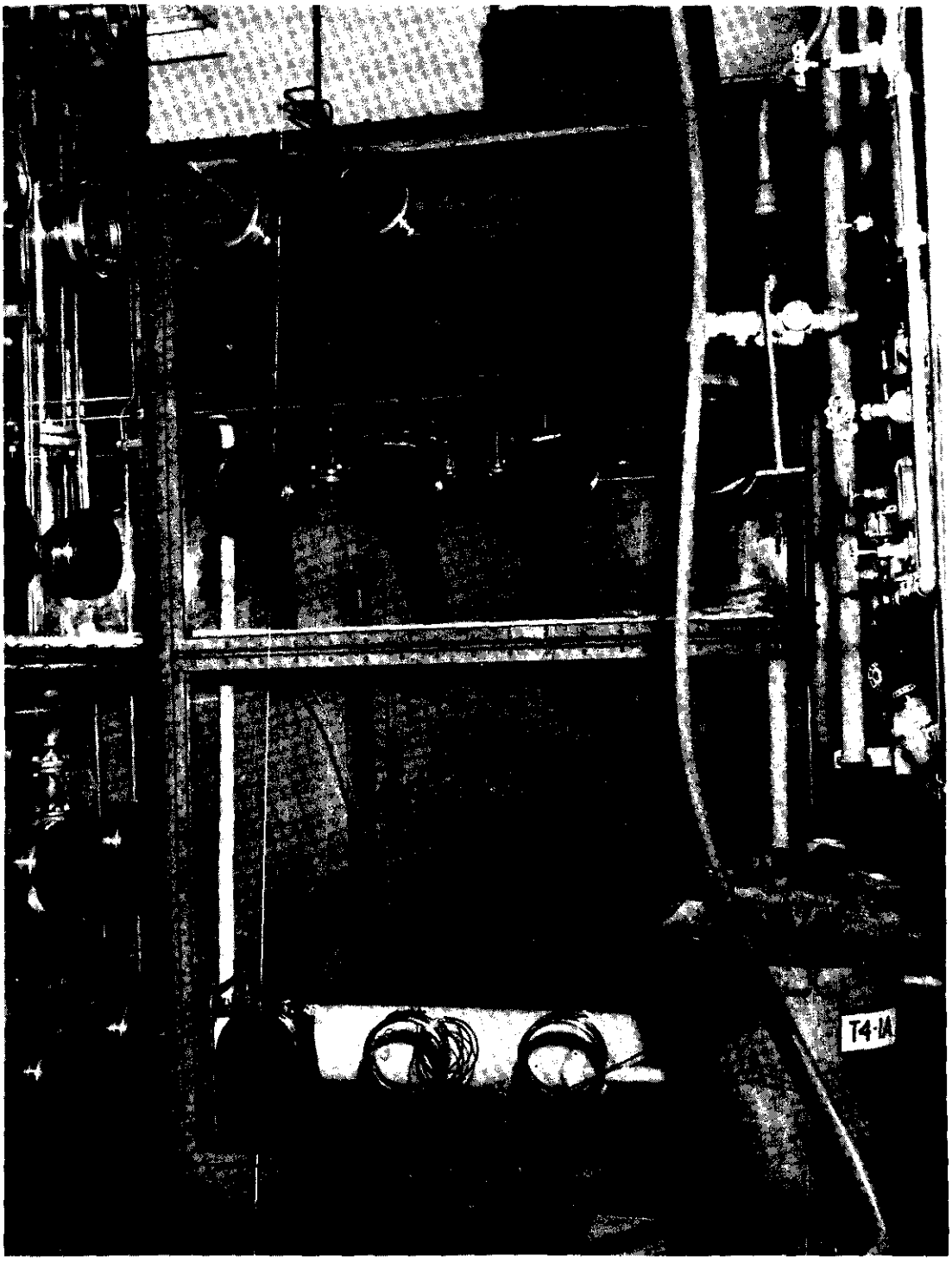
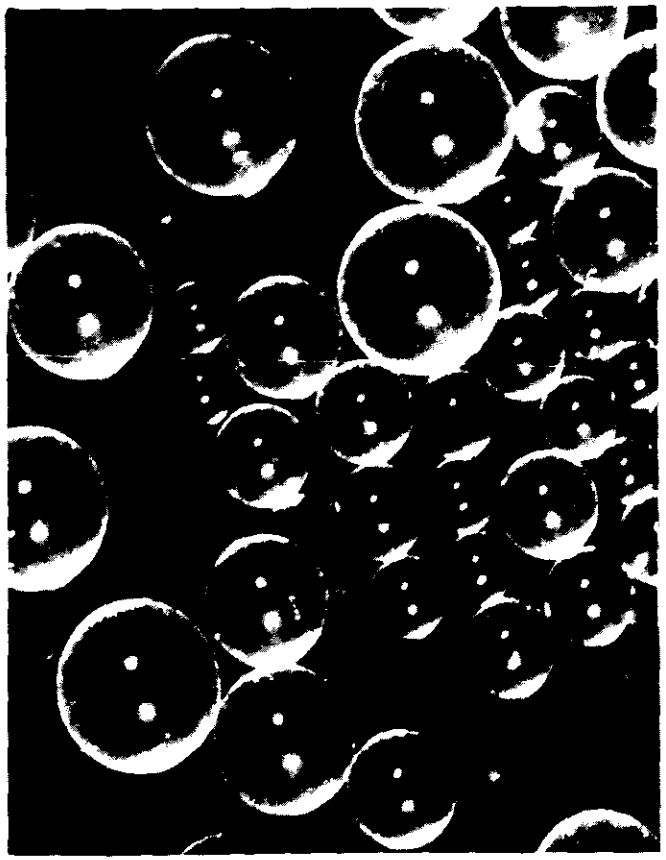


FIGURE 10

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

G. E. Lee, Chief
Chicago Patent Group

March 15, 1965

R. K. Sharp, Patent Attorney
Richland Operations Office

PATENT CLEARANCE

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

Number	Title
HW-83601	Progress in Plutonium Utilization

Remarks

Discloses the subject matter of the following invention reports:

HWCI-	AEC Case No.	
347	S-16,710	Patent application filed
394	S-16,754	
1209	S-22,961	
1512	S-27,330	
1595	S-23,253	
1025	S-17,995	
1130	S-22,421	
1210	S-22,904	
1535	S-23,206	
1615	S-23,267	
1765	S-31,323	Inactivated
931	S-17,320	Patent application filed
1057	S-19,530	
1227	S-22,934	Inactivated
1557	S-23,203	Inactivated
1003	S-17,975	Patent application filed
1132	S-22,423	
1234	S-24,060	
1414	S-26,352	Inactivated
1415	S-26,355	Patent application filed
1503	S-27,313	
1507	S-28,252	
1469	S-23,251	Inactivated
1513	S-27,337	Patent application filed

RKS:eg

TO: Jack Brown

December 17, 1964

PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
PROGRESS IN PLUTONIUM UTILIZATION	HW-63601
AUTHOR: O. F. Hill	
PROPOSED DISTRIBUTION: TID-4500	

PATENT REVIEW

[X] NO OBJECTION TO PROPOSED DISTRIBUTION

[ ] DOES NOT DISCLOSE AN INVENTION.

[X] DISCLOSES THE SUBJECT MATTER OF HWIR- 877, 894, 1209, 1512, 1995, 1995, 1130, 1210, 1585, 1616, 1745, 936, 1057, 1227, 1507, 1003 1192, 1234, 1414, 1415, 1503, 1607, 1605, 1513

[ ] DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION.

[ ] DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HWIR HAS BEEN FILED.

[ ] DISCLOSES SUBJECT MATTER OF HWIR-

REMARKS: HWIR-1745, 1227, 1414, 1605, and 1507 have been dropped. Patent applications have been filed on all the other cases.

GENERAL LEGAL REVIEW

[X] NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.

[ ] LEGAL CLEARANCE WITHHELD OR QUALIFIED.

REMARKS:

cc: To Be Reported File
HWIR Files
Publications File

Robert Keith Sharp
PATENT ATTORNEY
COUNSEL'S OFFICE

54-3000-290 (8-62)

GENERAL ELECTRIC COMPANY

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

HANFORD ATOMIC PRODUCTS OPERATION

99352

Extension 3601 3760 Bldg., 300 Area

JAN 17 1964

bcc: HWIR Files
LJ 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
Argonne, Illinois

Subject: RELEASE OF DOCUMENT

HWIR-	AEC CASE NO.
1051	[illegible]-19,539
1057	[illegible]-19,539

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-3282

Title: "New Neptunium 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
HWIR-1057 - "The Use of Fluoride to Increase the Separation of Plutonium from Fission Products by Nitric Acid Anion Exchange Process" by E. J. Wheelwright.

Very truly yours,

Robert Keith Sharp
Patent Attorney

RKS:eg
Enclosure

cc: Roland A. Anderson - USAEC-HDQ (w/encl.)

TO: Maurine Sheridan

DEC 23 1963

PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
NEW NEPTUNIUM PURIFICATION FACILITY AT THE PUREX PLANT	HW-SA-3202

AUTHOR: J. P. Duckworth and J. R. LaFiviere

PROPOSED DISTRIBUTION: American Chemical Society Meeting - Division of Nuclear Chemistry and Technology, Denver, Colorado, January 20-21, 1964

PATENT REVIEW

[X] NO OBJECTION TO PROPOSED DISTRIBUTION

[ ] DOES NOT DISCLOSE AN INVENTION.

[X] DISCLOSES THE SUBJECT MATTER OF HWIR- 1051, 1057

[ ] DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION.

[ ] DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HWIR HAS BEEN FILED.

[ ] DISCLOSES SUBJECT MATTER OF HWIR- _____

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

GENERAL LEGAL REVIEW

[X] NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.

[ ] LEGAL CLEARANCE WITHHELD OR QUALIFIED.

REMARKS: _____

cc: JP Duckworth
JR LaFiviere
Publications File
HWIR Files

Robert Keith Shary
PATENT ATTORNEY
COUNSEL'S OFFICE

Note Copies mailed to Messrs Lee and Anderson 1/17/64

54-3000-290 (8-62)

GENERAL ELECTRIC 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
LB

Subject: RELEASE OF DOCUMENT

HWIR-	AEC CASE NO.
1017	S-18,905
1051	S-19,539
1057	S-19,530

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) ~~has~~ (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

cc: Roland A. Anderson - USAEC-HDQ (w/encl.)

TO: Maurice Sheridan December 17, 1963

PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
NEPTUNIUM RECOVERY AND PURIFICATION AT HANFORD	HW-6A-3083

AUTHOR B. F. Judson

PROPOSED DISTRIBUTION American Chemical Society National Meeting - January 21, 1964 at Denver, Colorado.

PATENT REVIEW

[X] NO OBJECTION TO PROPOSED DISTRIBUTION

[ ] DOES NOT DISCLOSE AN INVENTION.

[X] DISCLOSES THE SUBJECT MATTER OF HWIR- 1017, 1051, 1057

[ ] DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION.

[ ] DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HWIR HAS BEEN FILED.

[ ] DISCLOSES SUBJECT MATTER OF HWIR-

REMARKS: HWIR-1017 has been dropped. Patent applications have been filed on the other two cases.

GENERAL LEGAL REVIEW

[X] NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.

[ ] LEGAL CLEARANCE WITHHELD OR QUALIFIED.

REMARKS:

cc: HWIR Files
Publications File

Robert Edith Sharp
PATENT ATTORNEY
COUNSEL'S OFFICE

TO: Maurice Sheridan December 16, 1963

PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
[redacted]	HW-34-3203
AUTHOR: B. F. Judson	
PROPOSED DISTRIBUTION: American Chemical Society National Meeting - January 21, 1964 at Denver, Colorado.	

PATENT REVIEW

- [x] NO OBJECTION TO PROPOSED DISTRIBUTION
  - [ ] DOES NOT DISCLOSE AN INVENTION.
  - [x] DISCLOSES THE SUBJECT MATTER OF HWIR- 1017, 1051, 1057
- [ ] DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION.
  - [ ] DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HWIR HAS BEEN FILED.
  - [ ] DISCLOSES SUBJECT MATTER OF HWIR-

REMARKS: HWIR-1017 has been dropped. Patent applications have been filed on the other two cases.

GENERAL LEGAL REVIEW

- [x] NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.
- [ ] LEGAL CLEARANCE WITHHELD OR QUALIFIED.

REMARKS:

cc: BF Judson
Neptunium Recovery File
HWIR Files
Publications File

Robert Keith Sharp
PATENT ATTORNEY
COUNSEL'S OFFICE

October 27, 1961

W. E. Swift
326 Building
300 Area

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

PATENT AND LEGAL REVIEW OF HW-SA-2290
CONTINUOUS ANION EXCHANGE PROCESSING OF PLUTONIUM

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." None 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 HMIR-1057 (HW-58327) we were able to obtain sufficient identification of only Dowex-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

Patent Attorney

RK Sharp:lb
3760 Bldg., 300 Area

cc: M. Sheridan
HMIR-1057
Publications File
LB

TO: M. Sheridan 10-27-61

PATENT AND LEGAL REVIEWS ON DOCUMENTS

TITLE	DOCUMENT NUMBER
Continuous Anion Exchange Processing of Plutonium	HW-SA-2290

AUTHOR: W. H. Swift and G. C. Oberg

PROPOSED DISTRIBUTION: Symposium on Ion Exchange at Feb. 1962 AICHE Meeting in Los Angeles and for possible publication in an AICHE periodical such as Chemical Engineering Progress.

PATENT REVIEW

[XX] NO OBJECTION TO PROPOSED DISTRIBUTION

[ ] DOES NOT DISCLOSE AN INVENTION.

[X] DISCLOSES THE SUBJECT MATTER OF HWIR- 1057

[ ] DO NOT RELEASE FOR UNRESTRICTED EXTERNAL DISTRIBUTION

[ ] DISCLOSES POSSIBLE INVENTION(S) ON WHICH NO HWIR HAS BEEN FILED.

[ ] DISCLOSES SUBJECT MATTER OF HWIR-

REMARKS:

GENERAL LEGAL REVIEW

[XX] NO LEGAL OBJECTION TO PROPOSED DISTRIBUTION.

[ ] LEGAL CLEARANCE WITHHELD OR QUALIFIED.

REMARKS: See attached letter.

cc: Publications File
HWIR-1057

R. K. Sharp ORIGINAL SIGNED BY ROBERT KEITH SHARP
PATENT ATTORNEY
COUNSEL'S OFFICE

A-7000-012 (9-61)

54-3000-290(11-60)

GENERAL ELECTRIC COMPANY

RICHLAND, WASHINGTON . . . TELEPHONE WHitehall 2-1111

HANFORD ATOMIC PRODUCTS OPERATION

Extension 3601 3760 Bldg., 300 Area

October 27, 1961

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

HWIR-1057 File
Publications File
Record Copy
LB

Subject: RELEASE OF DOCUMENT

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

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: HW-SA-2290

Title: Continuous Anion Exchange Processing of Plutonium.

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

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

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

Remarks:

Very truly yours,

Robert Keith Sharp

Robert Keith Sharp
Patent Attorney

RKS:lb

Enclosure

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,

E. J. Merlich, Acting

George H. Lee, Chief
Chicago Patent Group

July 14, 1960

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

Dear Mr. Lee:

BMIR-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.

Very truly yours,

SIGNED BY
ROBERT KEITH SHARP

Robert Keith Sharp
Patent Attorney
3760 Building, 300 Area

RKS:pmg

Encl.

bcc: Records Management
File
LB

July 8, 1960

W. Sale, Manager
Financial Operation
Hanford Laboratories
Hanford Atomic Products Operation

PATENT AWARD - EARL J. WHEELWRIGHT

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 proceed with the processing of request for issuance of award for the period June 1, 1960 through July 8, 1960.

Robert Keith Sharp
Patent Attorney

RKS:

Enclosures

cc: EJ Wheelwright personnel folder

bcc: PB Lamphere
MT Walling
File - Patent Award
File - HWIR-1057
Records Management
LB

July 8, 1960

Transfer List No. _____

DEPT. LIST NO. 99

L. L. Weiss, Manager
Consolidated Payroll Reports and Statistics
General Electric Company
Schenectady, New York

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

The following employee has signed a patent application and has been granted an award of $125, payable in part in shares of General Electric Company common stock and remainder in cash:

GE CASE NO.	AEC CASE NO.	DATE OF INVENTION REPORT	DATE PATENT APPLICATION SIGNED	FULL NAME AND ADDRESS	ACCOUNT TO BE CHARGED
1057	S-19,530	12-10-59	5-26-60	Earl J. Wheelwright 613 Catskill Richland, Washington	

Mail certificate to: W. Sale, Manager
Financial Operation
Hanford Laboratories Operation
Hanford Atomic Products Operation
Richland, Washington

Award Authorized by: _____ Certified: _____

July 3, 1950

Mr. Earl J. Wheelwright
613 Catskill
Richland, Washington

Dear Mr. Wheelwright:

NOTICE OF STOCK AWARD
HW18-1077 - ASC CASE 3-12,590

In connection with your execution of a patent application on the subject matter of the above case and in token of the Company's appreciation for the contribution which your invention represents, you are being awarded $12[illegible], payable in part in shares of General Electric Company common stock and remainder in cash.

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 near future.

Very truly yours,

W. E. Johnson

General Manager

WE Johnson:RKS:pmg

cc: EJ Wheelwright personnel folder

bcc: PE Lamphere
W. Sale
MT Walling
WE Johnson
RK Sharp (3)
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

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)

AEC-CH-271 (9-57)

ASSIGNMENT

WHEREAS, I, Earl J. Wheelwright, 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 FROM FISSION PRODUCTS, for 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 thereon;

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 Atomic 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 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 me 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 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 may 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 10 day of June, 1960

Earl J. Wheelwright (SEAL)

STATE OF ~~Washington~~ Idaho
COUNTY OF ~~Benton~~ Bannock } SS:

Personally appeared before me the above-named Earl J. Wheelwright to me well known, this 10th day of June, 1960, who signed the foregoing in my presence and acknowledged the same to be his voluntary act and deed.

Notary Public
Residing at Pocatello, Idaho
My Com. Expires: Oct. 13, 1963

(NOTARIAL SEAL)

Approved and consented to this 12th day of July, 1960

ATTEST:

GENERAL ELECTRIC COMPANY
HANFORD ATOMIC PRODUCTS OPERATION

(SEAL)

July 6, 1960

Marion Powell
3760 Building, 300 Area

HWIRs 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 Only" 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) HW-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: HWIR Files (8)
LB

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. 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 (HWIR-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

George H. Lee, Chief
Chicago Patent Group

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

AIR MAIL

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. 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 (HWIR-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)

AIR MAIL

cc: Mr. George H. Lee

June 9, 1960

Mr. Roland A. Anderson
Assistant General Counsel for Patents
U. S. Atomic Energy Commission
Washington 25, D. C.

Dear Mr. Anderson:

[redacted]

This is a companion case to S-19,539 ([redacted]). As indicated by Mr. Lee's letter of June 3, 1960, these two cases are to be filed on the same day and not later than June 17, 1960.

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

Due to these circumstances, the assignments do not bear the approval and consent on behalf of General Electric Company. Mr. Lee has an assignment dated May 26, 1960 which does bear this approval. If you consider the new assignment necessary, please return both copies to me, together with a confirmed copy of the application so that I may present them to Mr. Johnson for his signature.

Very truly yours,

ORIGINAL SIGNED BY
ROBERT KEITH SHARP

Robert Keith Sharp
Patent Attorney
3760 Building, 300 Area

RKS

Encl. Specification
Oath, Power of Attorney, and Petition
Assignment (2)

bcc: Records Management
File
LB

June 9, 1960

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

Dear Mr. Wheelwright:

RE-EXECUTION OF PATENT APPLICATION
HWDR-1057 - AEC CASE S-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 Oath, 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 stamped 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

RKS:pmg

Encl.: Specification
Oath, Power of Attorney and Petition
Assignment (2)
Letter to Roland A. Anderson
Envelope

bcc: File
LD

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 H. Lee

George H. Lee, Chief
Chicago Patent Group

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

May 31, 1960

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

HWIR-816 - AEC CASE S-15,473
HWIR-1051 - AEC CASE S-19,539
HWIR-1057 - AEC CASE S-19,538

ATTENTION - HUNK CARRE

Dear Mr. Lee:

Enclosed are the executed formal papers for the above three cases.

Very truly yours,

[redacted]

Robert Keith Sharp
Patent Attorney
3760 Building, 300 Area

RKS:p

Enclosures:
Specifications (3)
Print of Drawings (1)
Oath, Power of Attorney and Petition forms (3)
Assignments (4)

bcc: Records Management
HWIR Files (3)
LB

AEC-CH-271 (9-57)

ASSIGNMENT

WHEREAS, I, Earl J. Wheelwright, 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 FROM FISSION PRODUCTS, for 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 thereon;

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 Atomic 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 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 me 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 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 may 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 26th day of May, 1960.

Earl J. Wheelwright (SEAL)

STATE OF Washington
COUNTY OF Benton } SS:

Personally appeared before me the above-named Earl J. Wheelwright to me well known, this 26th day of May, 1960, who signed the foregoing in my presence and acknowledged the same to be his voluntary act and deed.

Leah R. Klymper
Notary Public

(NOTARIAL SEAL)

Approved and consented to this 26th day of May, 1960

ATTEST:

R. W. Watkins

GENERAL ELECTRIC COMPANY
HANFORD ATOMIC PRODUCTS OPERATION

bcc: Records Management
File
LB

April 28, 1960

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

Dear Mr. Lee:

IMTR-1057 - AEC CASE S-19,538

This is in response to your letter of March 30, 1960. The draft of the specification has been reviewed in detail by the inventor.

The product Dowex 21K is stated by the inventor to be chemically identical with Dowex 1. Apparently there is some physical difference, but he does not even know what that is. He knew nothing about the chemistry of Permutit SK beyond that already given. The most that I have been able to find is that it is a quaternary ammonium resin (Kunin, Ion Exchange Resins, Second Edition, page 93).

In my own past experience I found that more information regarding the chemical identity of synthetic resins could be obtained by going to Div. 90 of the Patent Office than by any other means. They have an extensive collection of manufacturer's literature. Perhaps someone in your Washington office could help us out. I fear, however, that Permutit may be one of the "close-mouthed" organizations.

I am returning the carbon copy of your letter, on which the inventor has given some of the data requested by you. He has also entered proposed amendments in pencil.

I should perhaps explain that the figures on page 6, lines 17 and 18 mean "gamma-disintegrations (or gamma photons) per minute per milliliter". The "gamma count" has been corrected to give these fundamental values.

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

Page 2, cancel the entire page and substitute:

--This invention deals with the recovery of plutonium values from an aqueous nitric acid solution by anion exchange and in particular with the separation of plutonium values from fission product values.

The anion exchange process is readily adaptable to a wide variety of process applications. The process is basically a three step operation----an absorption step wherein anionic 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 decontaminated from all objectionable impurities, no decontamination is necessary and the washing cycle can be eliminated from the process. If, however, anion exchange is used as a process where decontamination from uranium, fission products, or other ionic or nonionic 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.

Nitric acid solutions containing plutonium and fission product values are obtained during the processing of neutron-irradiated uranium, for instance by extracting the uranium and the bulk of the plutonium with tributyl or other trialkyl phosphates and subsequently back-extracting the plutonium with a solution of a reducing agent. The recovery of plutonium from such an aqueous nitric acid solutions with strong-base anion exchange resins 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 resin is washed with strong nitric acid or metal nitrate solutions to remove coabsorbed fission products, some fission products are not removed; in particular, zirconium and niobium are held rather strongly by the resin. Consequently, these fission products, or at least part of them, are eluted together with the plutonium when the resin is treated with dilute nitric acid, which means that the plutonium product solution is contaminated 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 widely, depending on the specific application; however, since the resin capacity begins to decrease when the concentration 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 waste solutions which are waste solutions containing unusually high concentrations of plutonium, plutonium feed concentrations as low as 0.010

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

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

--Single cycle zirconium-niobium decontamination factors in the range of 2×10^4 to 5×10^4 are readily obtained by the process of this invention and with special care zirconium-niobium decontamination factors as high as 5×10^5 can be achieved in one cycle. (The decontamination factor is the amount of fission 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 fission products in the eluted product solution).--

As regards the requested example based on pages 98-102, the inventor states that this was a highly theoretical proposal as to which he cannot give the necessary data. He has proposed the following Example XII to show separation of uranium. It does not involve the invention feature, however, and you may feel that it would confuse the issue 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.

Example XII

The process of this invention has application in the processing of plutonium reactor fuels, more particularly the recovery of purified plutonium from plutonium recycle reactor fuels. Three runs were made to determine the effect the high uranium content of such dissolved fuels would have on the anion exchange process. The feed solution for these three runs contained uranium and plutonium at a mole ratio of 300 to 1; corresponding to natural uranium irradiated to about 3000 megawatt days per ton of uranium. 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 moderate amount of resin washing when the uranium and plutonium are present in the 7 M HNO_3 feed solution at a mole ratio of 300 to 1. The operating conditions and the decontamination results are compiled in Table XII.

RUN XXI

Run Number	1	2	3
Feed Solution			
Plutonium Concentration (grams per liter)	0.405	0.084	0.686
Uranium Concentration (grams per liter)	120	60.6	184
Ratio Uranium/Plutonium	296	298	294
Product Solution			
Plutonium Concentration (grams per liter)	38.4	34.8	46.5
Uranium Concentration (grams per liter)	0.006	0.010	0.002
Uranium Decontamination Factor	1.9×10^6	1.0×10^6	6.8×10^6

Resin: Dowex-1, X-4 (50-100 mesh)
Temperature: 60 C for all operations
Wash Solution: 16 Column Volume of 7 M HNO_3

The results of these three runs clearly show that plutonium can be almost completely decontaminated from uranium in one cycle. A second cycle would reduce the uranium content of the final plutonium product solution to an undetected amount. No fluoride was present in the feed or wash solutions of the runs of example III. However, the presence of fluoride in the amounts indicated in the runs of examples I and II would result in equal or greater separation of uranium from plutonium. The data in the runs of examples I, II, and III clearly show that two cycles of the anion exchange process of this invention will give complete decontamination from uranium and fission products of plutonium from Plutonium Recycle Reactor irradiated fuels.

I have discussed the separation cycle with the inventor thereof but he has not completed his detailed review. I will forward it to you as soon as I receive it.

Very truly yours,

Robert Keith Sharp
Patent Attorney
3760 Building, 300 Area

RKS:yng

Enclosures:
copy of letter
copy of specifications

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

George H. Lee, Chief
Chicago Patent Group

Enclosure:
Specification and Claims

January 29, 1960

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

Dear Mr. Lee:

HWIR-1037 - AEC CASE S-18,959
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 HW-62000, entitled "Plutonium Recycle Program Annual 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

WC Poe:es

Enc.

cc: Mr. Roland Anderson w/ Enc.

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

54—7000—002

HANFORD LABORATORIES OPERATION
GENERAL ELECTRIC
RICHLAND, WASHINGTON

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
HWIR - 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.

M F Puckett

Supervisor
DOCUMENT DISTRIBUTION & FILES

MF Puckett/vbl

GENERAL ELECTRIC

ATOMIC PRODUCTS DIVISION
[redacted]
Cincinnati 15, Ohio

file
Yes I think it's a good idea.
Doug

September 1, 1959

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

GENERAL ELECTRIC

ATOMIC PRODUCTS DIVISION
[redacted]
Cincinnati 15, Ohio

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. In 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

PAUL R. WEBB, II
Department Patent Counsel

PRW.II:dyh

Enclosures

DON'T SAY IT --- *Write It!*

DATE September 3, 1959

TO EJ Wheelwright
JE Brown

FROM WC Poe

HNIR-1051 - AEC CASE S-19,539
HNIR-1057 - AEC CASE S-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 Neptunium", written by E.J. Wheelwright for a presentation at the Gatlinburg Ion Exchange Conference, Gatlinburg, Tennessee, September 7-9, 1959.

WCP:es

cc: File (2)
LB

RUSH

"CENTRAL MAIL"
1959 SEP 2 PM 12 27
1959 SEP 2 PM 12 11

TWX NR 91 /P R I O R I T Y/

TWX-4

FROM ROLAND A ANDERSON USAEC WASHDC SEPT 021955Z
TO W C POE GEN ELEC CO RICHLAND WASH
INFO GEORGE H LEE USAEC COO LEMONT ILL

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

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

Roland A. Anderson,
Assistant General Counsel for Patents

August 27, 1959

George H. Lee, Chief
Chicago Patent Group

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

SYMBOL: PD:JWP

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 1958 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 29, 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.

August 24, 1959

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

Dear Mr. Lee:

HWIR-1051 - AEC CASE S-19,539
HWIR-1057 - AEC CASE S-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 Neptunium", written by E. J. Wheelwright for a presentation at the Gatlinburg Ion Exchange Conference, Gatlinburg, Tennessee, 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 Poe:cc

Enc.

cc: Mr. Roland A. Anderson w/ 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

July 21, 1959

Earl J. Wheelwright
325 Building
300 Area

HWIR-1057 - AEC CASE S-19,030

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

ORIGINAL SIGNED BY
W. C. POE
Contract Engineer

WC Poe:es

cc: MF Walling
CA Rohrmann
Records Center
File
LB

UNITED STATES
ATOMIC ENERGY COMMISSION
WASHINGTON 25, D. C.

July 17, 1959

IN REPLY REFER TO:

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
for Patents

cc: Mr. George H. Lee

Cincinnati, Ohio
February 25, 1959

Mr. Roland A. Anderson
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
1057	S-19,530	Use of Fluoride to Increase Separation of Plutonium from Fission Products by the Nitric Acid Exchange Process	E. J. Wheelwright
1068	S-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:dyh

cc: Mr. W. C. Poe

January 26, 1959

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

GENERAL ELECTRIC

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,

Paul R. Webb, 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 HLO, 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
General 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,

George H. Lee, Chief
Chicago Patent Group

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 Poe:gs

Encs.

cc: HOO - AEC
Attn: Process Eng. & Mfg. Div.

bcc: Records Center
File
LB

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

1 - 6 - W.C.Poe
7 - W.H.Reas
8 - M.T.Walling,Jr.
9 - E.J.Wheelwright
10 - 300 File
11 - Records Center

HW-58327
Dec.2,1958

HANFORD ATOMIC PRODUCTS OPERATION
GENERAL ELECTRIC COMPANY
RICHLAND, WASHINGTON

REPORT OF INVENTION

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

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.

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

Jack L. Ryan
Ward L. Lyon

SIGNED (SUPERVISOR)	DATE	DEPARTMENT
M.T. Walling, Jr.	12/10/58	HLO

NOTE: SUGGESTIONS FOR PREPARING THE INVENTION DESCRIPTION ARE CONTAINED ON THE REVERSE SIDE OF THIS FORM.

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[1,2,3]. 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

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1. 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).

2. 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).

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

Earl J. Wheelwright — 12/9/58
Inventor — Date

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

Jack L. Ryan
Witness

Ward L. Lyon
Witness

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 = 10^7 γ/m, ml; Ru-Rh = 10^6 γ/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 (≤ 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 x 10^7 γ/m, ml, of these feed solutions was a factor of two higher than the activity of the feed solutions for the runs shown

Earl J. Wheelwright 12/9/58
Inventor Date

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

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Ward L. Tyson
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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.

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EJW/ffs

TABLE I. FLUORIDE WASH CYCLE

Run No.	Wash Cycle: Column Volumes of Wash	Wash Cycle: Wash Solution Composition	Wash Cycle: Average Flow Rate (ml/min., cm²)	Elution Cycle: Average Flow Rate (ml/min., cm²)	Elution Cycle: Pu Product Conc. (gms/l)	Elution Cycle: Pu Conc. Factor	Fission Product Decontamination Factor: Zr-Nb	Fission Product Decontamination Factor: Ru-Rh	Product Purity (micro curies impurity / Pu alpha disintegrations): Zr-Nb (x10⁻¹¹)	Product Purity (micro curies impurity / Pu alpha disintegrations): Ru-Rh (x10⁻¹¹)
1	32	7.2 M HNO_3	5.5	1.1	50	120	984	6,200	12	0.45
2	34	7.2 M HNO_3	5.3	1.2	50	120	1,210	13,000	10	0.20
3	28	7.2 M HNO_3	4.8	0.5	50	85	409	336	16	0.25
4	14 14 8	7.2 M HNO_3 7.2 M HNO_3; 0.056 M HF 7.2 M HNO_3	4.6	0.9	44	86	190,000	10,000	0.05	<0.05
5	14 18 6	7.2 M HNO_3 7.2 M HNO_3; 0.01 M HF 7.2 M HNO_3	5.2	2.4	49	77	25,700	38,500	0.05	<0.05
6	14 22 6	7.2 M HNO_3 7.2 M HNO_3; 0.0056 M HF 7.2 M HNO_3	4.2	0.6	35	62	27,000	10,000	0.25	<0.05

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

Carl J. Wheelwright
Inventor

12/9/58
Date

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

Jack L. Ryan
Witness

Howard F. Tyson

TABLE II. THE EFFECT OF FLUORIDE ADDED TO FEED

Run No.	Wash Cycle: Column Volumes of Wash	Wash Cycle: Wash Solution Composition	Wash Cycle: Average Flow Rate ($\frac{ml}{min., cm^2}$)	Elution Cycle: Average Flow Rate ($\frac{ml}{min., cm^2}$)	Elution Cycle: Pu Product Conc. (gms/l)	Elution Cycle: Pu Conc. Factor	Fission Product Decontamination Factor: Zr-Nb	Fission Product Decontamination Factor: Ru-Rh	Product Purity ($\frac{\text{micro curies impurity}}{\text{Pu alpha disintegrations}}$): Zr-Nb ($\times 10^{-11}$)	Product Purity ($\frac{\text{micro curies impurity}}{\text{Pu alpha disintegrations}}$): Ru-Rh ($\times 10^{-11}$)
1	15	7.2 M HNO_3								
	18	7.2 M HNO_3; 0.01 M HF								
	6	7.2 M HNO_3	5.2	3.1	44	112	70,000	--	0.35	<0.05
2	14	7.2 M HNO_3								
	16	7.2 M HNO_3; 0.01 M HF								
	6	7.2 M HNO_3	4.5	3.2	47	94	86,000	--	0.20	<0.05

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Earl J. Wheelwright 12/9/58
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-3000-036 (9-56)
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GENERAL ELECTRIC
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RICHLAND, WASHINGTON . . . . . HANFORD ATOMIC PRODUCTS OPERATION

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HANFORD ATOMIC PRODUCTS OPERATION
GENERAL ELECTRIC COMPANY
RICHLAND, WASHINGTON

REPORT OF INVENTION

A.E.C.CASE NO.

G. E. CASE NO. HW[illegible]7

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Ward L. Lyon

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M. T. Walling, Jr.	12/10/58	HLO

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Earl J. Wheelwright — Inventor — 12/9/58 — Date

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