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THE ADAPTATION OF AN ION EXCHANGE PROCESS TO REMOTE OPERATION IN A RADIOCHEMICAL SEPARATIONS PLANT

by
W. J. Mottel and J. F. Proctor
Separations Engineering Division

September 1961



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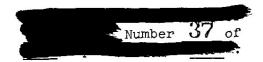
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William J. Mottel and John F. Proctor

September 1961



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ABSTRACT

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Equipment and techniques are described for the adaptation of a small-scale ion exchange process to remote operation in a large radiochemical separations plant.

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CONTENTS

	Page
Introduction	4
Summary	4
Discussion	5
Simplified Process Description	5
The Equipment Frame	6
The Ion Exchange Column	12
The Prototype Unit	14
Appendix - Description of Auxiliary Equipment	15
1. Service Piping	15
2. Process Vessels	15
3. Air Lifts	18
4. Vessel Venting and Entrainment	19
Bibliography	20
LIST OF FIGURES	
Figure	
1 Process Flow Diagram	7
2 Equipment Frame Structure	9
3 Plan View of a Complete Equipment Frame	10
4 Elevation View of a Complete Equipment Frame	11
5 A Typical Ion Exchange Column	13
Appendix	
A-1 Filter	16
A-2 Target Dissolver	17
A-3 Typical Air Lift and Separator	18

THE ADAPTATION OF AN ION EXCHANGE PROCESS TO REMOTE OPERATION IN A RADIOCHEMICAL SEPARATIONS PLANT

INTRODUCTION

In general, ion exchange processes have advantages over solvent extraction processes when batch operations are indicated; that is, when the quantities of material to be handled are small and the desired product concentrations are high. Conversely, solvent extraction is usually preferred when large quantities of material are to be processed because it is readily adaptable to continuous operations. Ion exchange, although extensively used in laboratory radiochemical separations, has not often been applied in large production facilities that handle highly radioactive solutions. The limited application of ion exchange in this field can be attributed to the difficulties associated with the adaptation of these processes to remote operations, principally the problems of handling the solid ion exchange resins. This report describes ion exchange equipment and techniques that have been developed for a specific remote process application, namely, the use of an anion exchange process to recover Pu238 and Np237 from irradiated neptunium oxide target elements.

SUMMARY

A compact, remotely operated unit was designed for the processing of irradiated NpO₂ target elements to recover both Pu²³⁸ and unburned Np²³⁷. The equipment needed to separate and decontaminate these isotopes included a dissolver, five ion exchange columns, and eleven valence adjustment and collection tanks complete with instrumentation and solution transfer devices. All of this equipment was mounted within two steel frames, each designed to occupy one shielded module of an existing fuel processing plant. Each frame, complete with its complement of tanks, columns, and piping, was capable of being installed in, or removed from, the module as a single unit. Only those few items subject to periodic failure could be removed individually from the frame, while the majority of the equipment, including tankage and piping, was permanently fastened to the frame structure.

Although a departure from the traditional separations plant practice of individual disassembly and removal of each process vessel and service pipe, this technique of treating complete processing units as individual equipment pieces accomplished several objectives. It permitted the processing of small volumes in small equipment without excessive liquid holdup, utilized a minimum of shielded space, and precluded any interference between the ion exchange process and the existing operations of fuel processing.

DISCUSSION

The production of small quantities of Pu²³⁸ by the neutron irradiation of Np²³⁷ target elements was begun at Savannah River in 1958, and an ion exchange process for the separation of these isotopes from the irradiated targets was demonstrated in a minature pilot plant during the following year. Concurrent with the pilot plant operations, the development of equipment was begun for a plant-scale facility to separate these isotopes. This report describes the equipment that was developed to adapt the small-scale ion exchange process to remote operation in the large shielded facilities of the Savannah River fuel processing plants.

Heavily shielded facilities and equipment that was adaptable to remote operation were necessary because the formation of fission products during irradiation of the neptunium targets results in gamma radiation levels comparable to those encountered in processing spent reactor fuels. Because some shielded space was available in the existing plant facilities, it was economically preferable to use these facilities rather than to erect a separate processing plant. However, before this space could be effectively utilized, means had to be developed to adapt the small equipment needed by the new process, particularly the ion exchange columns, to operation in a building originally designed for the large equipment associated with reactor fuel processing. At the same time, the new process had to be installed in a manner which ensured complete chemical and operational independence from the existing fuel processing operations.

A brief description of this separation process is presented here to provide the background for the discussion of the equipment.

SIMPLIFIED PROCESS DESCRIPTION

Pu²³⁸ is produced by the neutron irradiation of Np²³⁷; an (n,γ) reaction on Np²³⁷ initially forms Np²³⁸, which subsequently β -decays to Pu²³⁸. The irradiated target elements, consisting of NpO₂-Al compacts containing Np²³⁷, Pu²³⁸, and fission products, are "cooled" for several months after removal from a nuclear reactor. These elements are then transferred to a separations plant, where, in shielded facilities, they are dissolved in nitric acid and processed through ion exchange equipment to recover the Pu²³⁸ and the unburned Np²³⁷. The waste fission products are retained in underground storage tanks.

Plutonium and neptunium in the (IV) valence state are two of the few metal ions which form stable anionic nitrate complexes that can be strongly absorbed on anion resins from solutions of moderate nitrate concentration. (1) This characteristic permits neptunium and plutonium to be separated from most contaminants, including fission products. Decontamination factors as high as 1,000 are attainable for each cycle

of anion exchange. The maximum absorption of Pu(IV) and Np(IV) on anion resin occurs in solutions having a total nitrate concentration of about 8M, and, because the anionic nitrate complex ionizes at low nitrate concentrations, these elements can readily be eluted from the resin by solutions containing about 0.3 molar nitrate ion. Hydrolysis and polymerization are prevented during processing by maintaining a minimum acid concentration of 0.25M. The selection of a specific anion resin for the process is governed by the need for reasonably rapid absorption and desorption kinetics and the need for resins that resist degradation by ionizing radiation. Both "Permutit" SK and "Dowex"-l have been satisfactory in this respect. The details of these processes and the reaction kinetics of these resins have been discussed in detail by Tober, et al. (1), Burney and Prohaska(2), and others. (3)

Figure 1 shows the paths of plutonium and neptunium through a process designed to separate them from each other with a cross-contamination of less than 1 per cent while reducing the concentration of fission products by factors greater than 5 x 10⁵ in each product. Five cycles of anion exchange are required. In the first cycle, both the neptunium and the plutonium are absorbed and thus are separated from aluminum and the bulk of the fission products. In the second cycle, both elements are again absorbed, while again fission products pass through; the plutonium is then selectively eluted. The plutonium next undergoes two successive cycles of anion exchange which constitute the final decontamination and concentration of this product. The neptunium undergoes one additional cycle of decontamination before it is transferred to other facilities to be concentrated, converted to the oxide, and fabricated into new target elements.

THE EQUIPMENT FRAME

Shielded space was available within the existing Savannah River "canyon"* buildings for the installation of this new process. However, the throughput of the process was to be quite small compared to that of existing "canyon" operations, and small equipment was necessary to minimize inventory and maintain acceptable product concentrations. The remote operation of this small equipment in shielded facilities designed for a process of much larger scale was one of the governing considerations in the design of the processing unit. It was not feasible to supply all of the services required by the new process and

^{*}The term "canyon" as applied locally refers to the heavily shielded building used to house, and supply services to, the process equipment needed for the recovery of plutonium and uranium from spent reactor fuels. A detailed description of this building, the design philosophy used in its development, details of its modular construction, and the manner in which services are supplied to process equipment has been published by Colven, et al. (4)

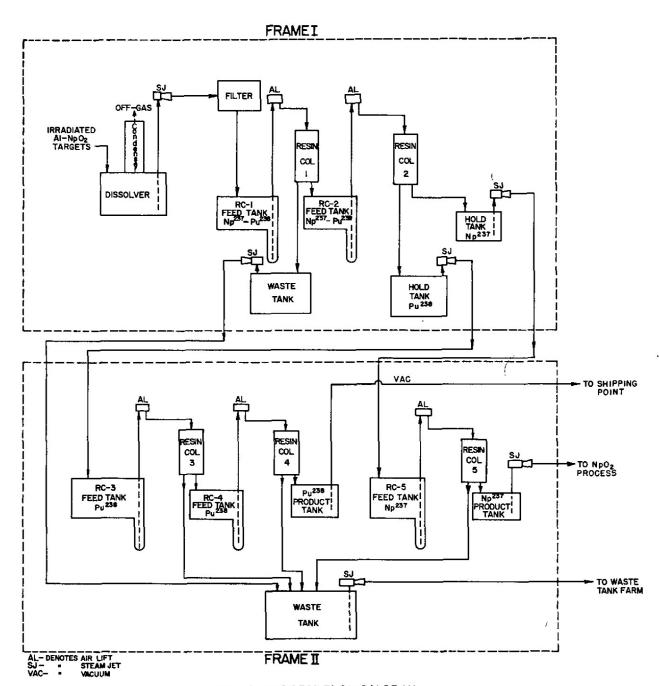


FIG. 1 PROCESS FLOW DIAGRAM

still retain the usual practice of individually assembling each equipment piece without holding up excessive volumes of solutions in the process piping and without utilizing a prohibitive amount of shielded space.

A solution to this problem was the development of a "unitized" design in which all of the individual equipment pieces required by the process were mounted in two equipment frames of structural steel, each capable of being installed in or removed from the canyon as a unit. All of the process piping and tankage was permanently mounted within these frames, and only those few critical equipment pieces subject to periodic failure were designed to be individually removed from the frames and the canyon. This technique represented the minimum departure from the accepted practices of a remotely maintained plant and was equivalent to the installation of completely integrated processing units in space originally designed for a single process vessel.

Each of the two equipment frames required by the process was designed to occupy one module of the canyon, (4) a space about 10 feet square by 17 feet high. As illustrated in Figure 1, one frame contained the equipment for the dissolving of the target elements, the initial decontamination cycle, and the partitioning of plutonium and neptunium. The second frame contained the equipment for additional decontamination of the neptunium and plutonium, product collection, and waste handling.

The first frame contains a dissolver, filter, two ion exchange columns, and five solution adjustment tanks. The process vessels, excepting the ion exchange columns, are equipped with agitators and probes for the measurement of liquid level and specific gravity; most are also equipped with heating coils, samplers, and temperature instrumentation. Solutions are transferred between vessels either by steam jets or by air lifts. The steam jets, air lifts, ion exchange columns, motordriven agitators, several of the resistance thermometers, and the filter are individually removable from this equipment frame.

The second frame contains three ion exchange columns and six tanks, all of which are equipped with auxiliaries similar to those in the first frame. The ion exchange columns, steam jets, air lifts, and some thermometers in this frame can also be removed individually.

Each of the equipment frames is a rugged, box-like steel structure containing internal support members for each piece of equipment. Panels are located on the top of the frame for mounting the removable piping and connectors that span the gap between the frame and the supply piping embedded in the shielding walls of the canyon. Figure 2 is a photograph of a typical frame before its equipment is installed; it shows the heavy structural members used to provide maximum rigidity and to minimize stresses on the permanently mounted piping and tankage.

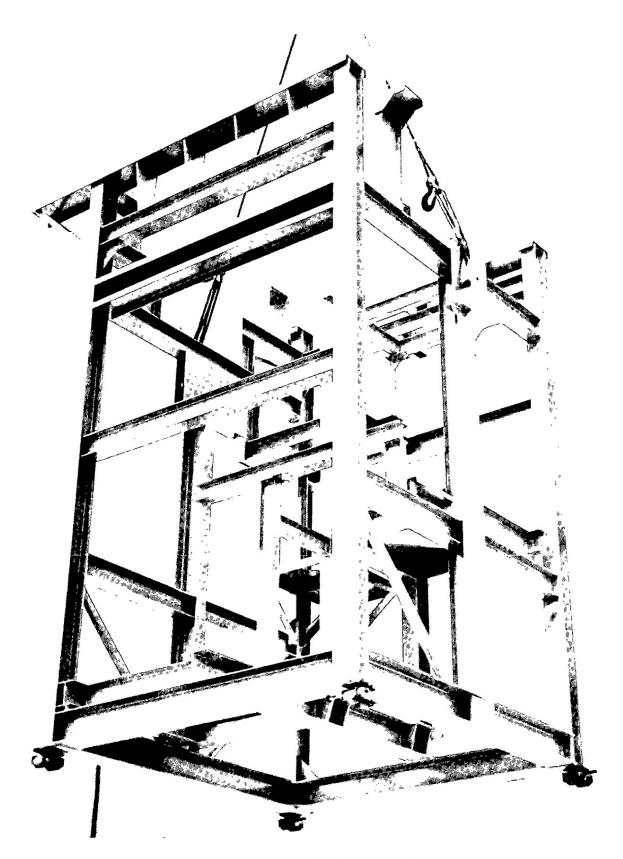


FIG. 2 EQUIPMENT FRAME STRUCTURE

All of the structural steel has been protected from corrosion by "Amercoat" protective paint, and the bottom of each frame has been completely enclosed with stainless steel to form a sump to collect tank overflow and incidental spillage. This sump completes the chemical isolation of the ion exchange process from the remainder of the canyon operation by ensuring that all spills are reprocessed within the equipment frame.

Figures 3 and 4 show the plan and elevation views of a completed frame and illustrate the manner in which the permanent piping and tankage were installed. Pipe sizes were chosen to provide minimum process holdup consistent with reasonable head requirements for solution transfers. Where feasible, lines were sloped to ensure complete drainage. With the exception of the ion exchange columns, the equipment installed in these frames was typical of that normally used for remotely maintained installations in the canyon. Descriptions of this auxiliary equipment are summarized in the Appendix.

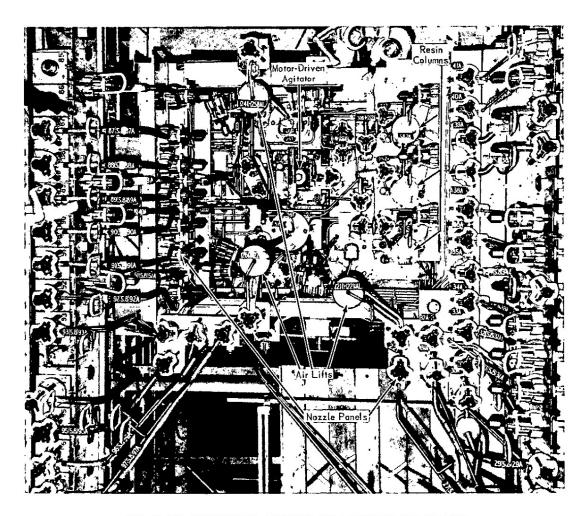


FIG. 3 PLAN VIEW OF A COMPLETE EQUIPMENT FRAME

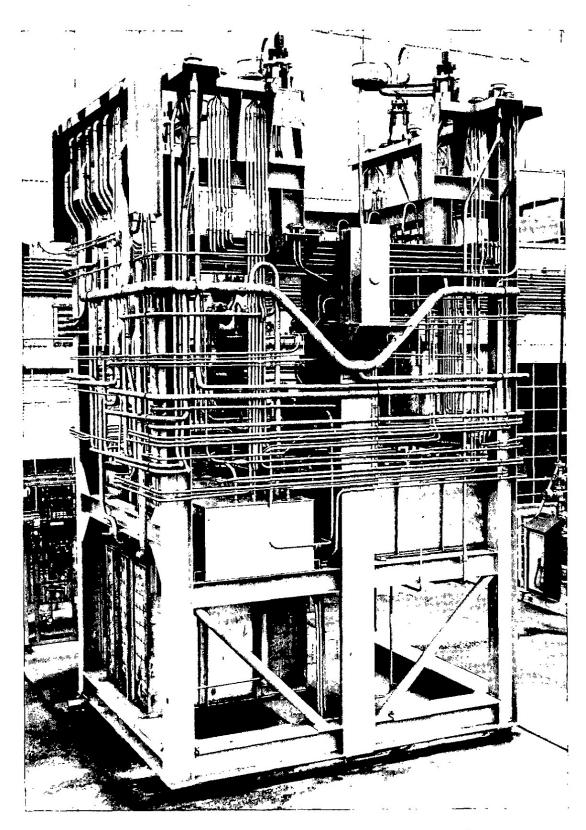


FIG. 4 ELEVATION VIEW OF A COMPLETE EQUIPMENT FRAME

THE ION EXCHANGE COLUMN

The principal new equipment piece required to adapt this process to the existing facilities was an ion exchange column in which all operations including replacement of resin could be performed remotely. In this process, relatively frequent resin replacement was required because of the cumulative effects of chemical degradation of the resin in the concentrated nitric acid solutions and the degradation induced by the relatively high radiation fields. In addition, the normal cyclic operations of absorption, washing, elution, and regeneration had to be performed without recourse to complicated valving mechanisms.

Figure 5 shows the ion exchange column developed for this process. Resin is charged and discharged from this column by suspending it as a free-flowing slurry in a solution having a specific gravity comparable to that of the resin. The valving operations are accomplished by a set of two weirs; air pressure can be applied preferentially to either or both weirs, causing them to act as a three-way valve for the feed and wash, elution, and resin removal operations.

As shown in Figure 5, the ion exchange column is a right vertical cylinder in which the resin is normally contained as a fixed bed submerged in a pool of the process solution. The resin bed is supported a short distance above the bottom of the column by a stainless steel screen. Process liquid enters the column at the top and is distributed by a splash plate before falling into the liquid pool above the resin. After passing downward through the bed and the support screen, the process liquid leaves the column through one of the two weirs. The first weir, the absorption and wash effluent weir, is positioned at an elevation far enough above the top of the settled resin to ensure that (1) the resin is always submerged in liquid, and (2) enough solution is present for good agitation of the resin during the resin loading and unloading operations. The second weir, the elution weir, is positioned slightly above the first. The application of air pressure to either weir governs weir overflow so that the two weirs function as a diversion valve. The inlet and outlet lines from the weir boxes take the form of "U" tubes to contain the air pressure supplied to actuate the weirs.

The resin column is surmounted by a long neck through which passes the shaft of a multiple-paddle agitator. The agitator consists of two sets of paddles and a set of splines. The upper paddle agitates and suspends the resin in the column so that resin can be unloaded as a free-flowing slurry. The lower paddle sweeps the support screen during the bed regeneration portion of the ion exchange cycle to clear the screen of any solids which may have accumulated during the prior absorption operations. The splines attached to the agitator shaft in the neck of the column aid in maintaining a free-flowing slurry during unloading and also serve to break up any resin

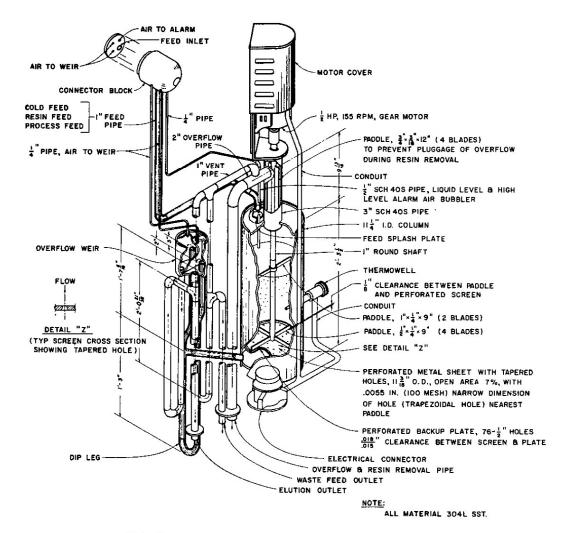


FIG. 5 A TYPICAL ION EXCHANGE COLUMN

agglomerates that may have formed under the exposure of the resin to radiation and chemical degradation.

Resin is fed to the column as a slurry in a solution having a specific gravity equal to that of the resin. The "loading solution" passes through the screen and overflows the effluent weir while the resin is retained in the column by the screen. The agitator is in operation during part of the loading operation to minimize pressure drop through the bed.

Resin is unloaded from the column by applying air pressure to both weirs and feeding to the column a solution having a specific gravity the same as that of the resin. With the agitator running, the resin slurry rises in the neck of the column and passes out of the column through an overflow pipe in the neck. During the operations of absorption, washing, and elution the column acts as a fixed but unrestrained bed, while during regeneration the resin is agitated to remove accumulated solids and to redistribute the resin so that all resin receives approximately equal radiation exposure.

The three liquid effluent lines from the column (feed effluent, elution effluent, and overflow) mate with funnels and pipe which are mounted within the frame to direct the solutions to the proper collection tank. The resin slurries that are removed from each column enter a common header that terminates in a waste collection tank from which the resin is eventually transferred to the waste handling system of the building.

The ability of this column to clear itself of accumulated solids makes it useful in applications where filtration is impractical; in these instances the agitator can be operated during the feed cycle. The bed performance no longer follows the typical breakthrough characteristic of fixed bed ion exchange; however, it can be used in an efficient manner by procedures described by Prout⁽⁵⁾ and Caracciolo⁽⁶⁾. Caracciolo has published a description of the operation of this column as an "agitated bed" in which the effects of channeling, resin attrition, loading and unloading techniques, permissible flow rates, and specific gravity of the process solutions are discussed in detail.

THE PROTOTYPE UNIT

A completely equipped prototype of one frame was constructed to demonstrate the feasibility of the concept and the reliability of the individual piece of equipment when assembled in a complete unit. This prototype duplicated all features necessary to the remote operation in the Plant process. All of the transmitting and recording instrumentation was included to ensure that no instrument difficulties would be encountered during Plant operation. The prototype unit was subjected to extensive operability tests, determinations of the sensitivity of instruments to process changes, and flow and volumetric calibrations. In terms of over-all equipment utilization, the equivalent of several months of operation was accumulated during this demonstration. Some of the information developed during the demonstration has been included in the Appendix.

This prototype frame was installed as part of an ion exchange recovery process for the decontamination of the Np²³⁷ that is recovered from the waste streams of the natural uranium fuel processing plant. As such, it represented the third frame to be installed in the plant. The prototype frame was essentially a duplicate of one of the frames required for the neptunium target process; i.e., it contained three ion exchange columns and essentially the same process tankage and transfer equipment as the frame required for final decontamination of Np²³⁷ and Pu²³⁸. To ensure that all equipment was operable and reliable, each of the target processing frames was similarly tested before it was installed in the plant.

W. 4. Mottel

J. F. Proctor Separations Engineering Division

APPENDIX

DESCRIPTION OF AUXILIARY EQUIPMENT

Descriptive information is presented below on the design of individual equipment pieces developed for the process. The descriptions are typical for each frame that has been constructed.

SERVICE PIPING

The services required included water, steam, electric power, chemical solutions, service air and instrument air. All of these services were supplied to the frames through existing lines embedded in the canyon shielding wall⁽⁵⁾. Connections to the frames were made by removable pipe and electrical "jumpers" that connected nozzles on the embedded pipe with corresponding nozzles mounted on panels at the top of the frame (see Figure 3). From the nozzle panels the services were distributed by permanently installed piping and electrical conduit.

Initially, there were not enough embedded lines to supply all of the services required by the frames; however, the number of individual service lines was increased by drawing bundles of as many as six small (1/2-inch) tubes through the existing 3-inch pipes embedded in the canyon shielding walls. A total of fifteen of these "tubing pulls" were made. Some were through embedded pipe sections up to 15 feet long that contained several irregular bends. The force required to draw a bundle of six tubes was of the order of 2,000 pounds, while a maximum of 5,000 pounds was required to form a tight seal between the new nozzle of the bundle and the existing nozzle on the embedded pipe. It is expected that this technique will be applicable to other canyon applications.

2. PROCESS VESSELS

All of the solution adjustment and hold tanks were of a flat-bottomed rectangular design to provide the maximum tankage volume. Consistent with canyon practice, none of the tanks were equipped with bottom drains and therefore each operates with a small nonremovable liquid heel. All service lines enter through the top of the vessel to minimize the number of welded junctions continuously exposed to process solution. A complete X-ray examination was made of each tank and pipe weld, and all tanks were pickled for 24 hours in hot nitric acid to ensure that a welded joint would not fail prematurely.

With a few exceptions, tanks were equipped with agitators, probes to measure liquid level and specific gravity, sample lifts, temperature measuring devices, and coils for cooling and heating the vessel contents. Air spargers were used for agitation in all tanks except those handling resin slurries; electrically driven propeller agitators were provided in those tanks which handled resin discharge from the columns.

The neptunium target dissolver was a scaled-down version of a dissolver used in the processing of natural uranium reactor fuels⁽⁴⁾. It was also equipped with liquid level and specific gravity probes, pressuresensing taps, and an off-gas condenser. A filter, which was individually removable from the frame, was provided to remove solids, principally silica, from the dissolver solution. Figure A-1 shows the filter and Figure A-2 shows the dissolver.

Solution transfers between tanks were made with both air lifts and steam jets. In those tanks containing air lifts, a 5-foot-long, 4-inch-diameter well was mounted in the bottom of the tank to provide the necessary submergence for operation of the lift. Steam jets were used for transfers between frames and for recycle of material within a frame, while air lifts were used to feed all columns. Steam jets were used to transfer waste solutions and neptunium product; vacuum lifts were used for transfers of plutonium product and samples.

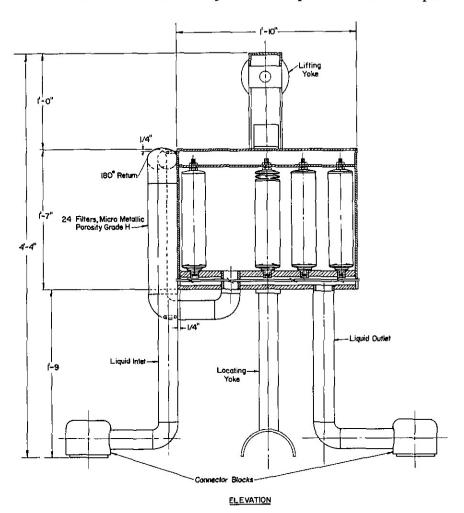


FIG. A-1 FILTER

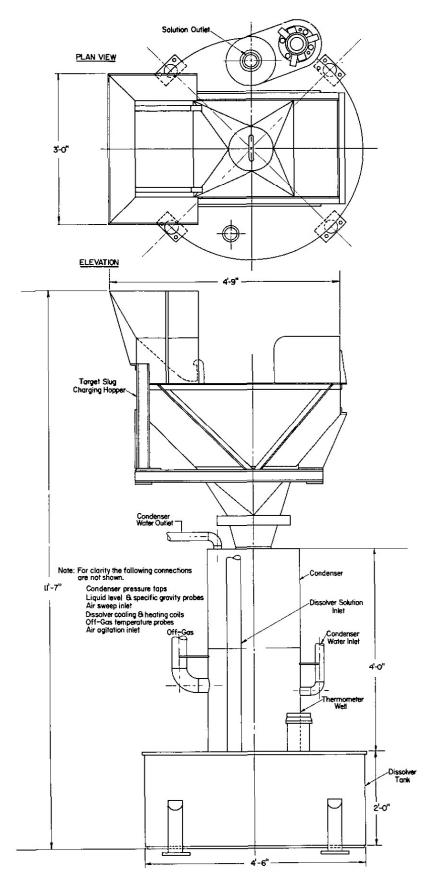


FIG. A-2 TARGET DISSOLVER

3. AIR LIFTS

The feed rate of solution to an ion exchange column is governed principally by the kinetics of the resin, the size of the column, and the concentration of the absorbable species. In this process these factors place a range on feed rate of between one and three liters per minute. Air lifts were chosen for this service because of their simplicity (no moving parts) and their demonstrated ability to handle resin slurries without pluggage⁽⁷⁾. Steam or air jets of a suitable capacity would be susceptible to pluggage of the nozzle throat, while mechanical pumps would be subject to wear and leakage.

Figure A-3 show a typical air lift. The air flows required to produce a range of liquid flows from one to three liters per minute range from 0.5 to 2 scfm, and changes in liquid level and specific gravity of the feed solution produce no more than a 10% change in liquid flow at any given air flow. Monitoring of the liquid flow rate in the frames is based upon the tank depletion indicated by the liquid level instrumentation.

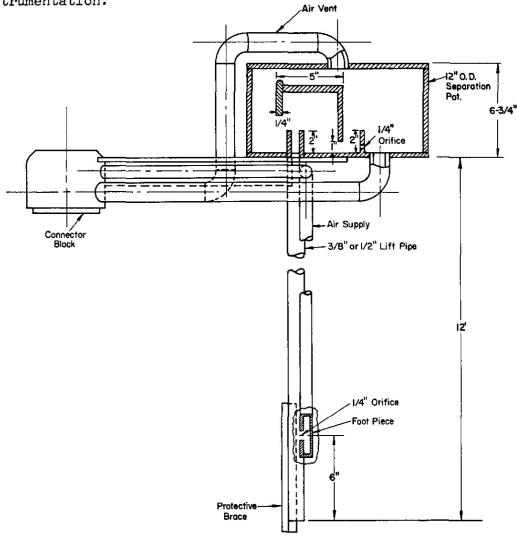


FIG. A-3 TYPICAL AIR LIFT AND SEPARATOR

The receiving chamber of the top of the lift shown in Figure A-3 was required to provide a disengaging space for liquid entrained in the air stream and to provide a small liquid holdup to damp the inherent surging action of these lifts.

4. VESSEL VENTING AND ENTRAINMENT

In compliance with accepted separations plant practice, all of the process equipment within each frame was connected to the vessel vent system of the canyon. This system maintains a one- to two-inch negative pressure differential between process equipment and the canyon atmosphere to minimize the release of airborne activity within the canyon. To reduce the number of individual connections required, each process vessel within the frame was connected to a common vessel vent header mounted permanently within the frame. This header then made a single connection with the common vessel vent system of the canyon. The ion exchange columns, however, were not connected directly to this vent system. Instead, in order to simplify removal of the columns from the frame, air sweeps from the canyon into the frame equipment were provided across each of the openings in the column. Also in accordance with established practice liquid seals were included in all process lines that entered the frame from a service area of the canyon building. This technique minimizes the possibility of active materials being released in areas occupied by personnel and is considered mandatory for operation of the separations plants.

Tests were made on the prototype equipment frame to establish the level of entrained activity to be expected in the vessel vent system during the operation of air lifts and air spargers. The measured entrainment levels were consistently below 2 ppm at air flows of 1 to 1.5 pounds per minute (15 cfm) in the vent header on the frame. These results indicated that the air lift separators (Figure A-3) and the vent header were efficient entrainment separators and that no difficulties would be encountered with excessive airborne radioactivity.

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- 7. Kearsley, G. W. T. <u>Use of an Air Lift as a Metering Pump for Radioactive Solutions</u>. Oak Ridge National Laboratory, Tenn.

 AEC Research and Development Report ORNL-2175, 35 pp. (October 1956).

REPORT DATA SHEET

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References:

- (1) V. P. Caraccielo, Anion Exchange in a Large Scale, Agitated Bod, DP-524 (1961)
- (2) Prepublication review of (1) dated June 26, 1961

No items were noted that, in my opinion, should be called to the attention of the ANC for patent consideration.

The report describes the design and operation of agitated ion exchange beds mounted on movable racks. The facility is remotely operable including the steps of loading and unloading the resin in the beds. Similar agitated ion exchange beds are described in the references.

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Attached for review are three copies of the following classified report:

The Adaptation of an Ion Exchange Process to Remote Operation in a Radiochemical Separations Plant by W. J. Mottel and J. F. Proctor

Please contact my office if you have any comments. The report will be released when approval is received, but not until after 14 days from the date shown above.

Yours very truly,

Original signed by W. P. Overbeck

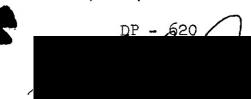
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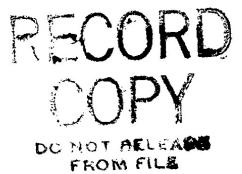
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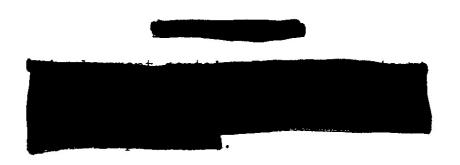
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September 21, 1961

Mr. W. P. Overbeek, Director Savannah River Laboratory E. I. du Pont de Nemours and Company Aiken, South Caroling

Attention: J.W. Morris, Director Separations and Services Section

Dear Mr. Overbeck:

We have reviewed the following document transmitted by your letter of September 19, 1961, and this office has no objection to its proposed release:

DP-620 - "The Adaptation of an Ion Exchange Process to Remote Operation in a Radiochemical Separations Plant" - by W. J. Mottel and J. F. Proctor

Your office was so notified by telephone this date.

Very truly yours,

Paul J. Hagelston, Director Technical & Production Division

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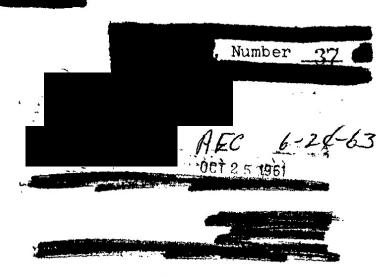
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DP-620, THE ADAPTATION OF AN ION EXCHANGE PROCESS TO REMOTE OPERATION IN A RADIOCHEMICAL SEPARATIONS PLANT by W. J. Mottel and J. F. Proctor

This report describes the successful application of the "frame" concept to the equipment for processing Np²³⁷ and Pu²³⁸ by ion exchange with anion resin. When we were requested by the Atomic Energy Commission to provide facilities for this processing, it was realized that anion exchange could be used as the basic process but that the service conditions were entirely different from previous experience. Up to this time ion exchange had been used only in gloved-box cabinets in low radiation fields. In this new application, remote operation would be required because of the high gamma radiation and the resin life would be very short because of the high alpha radiation.

A "frame", which consists of a structural unit containing several vessels and interconnecting piping, fits into the canyon space originally provided for a single vessel. Tanks are permanently installed within the frame, but resin beds and similar equipment can be individually replaced by remote techniques. Similarly, sampler lines and bubblers are permanently installed, but the airlifts used for liquid transfers can be replaced remotely. The achievement of the proper compromises between increased complexity and versatility and the selection of mutually compatible means of agitation, liquid transfer, control, and the like are described in the attached report.

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It appeared that the "frame" concept should be feasible and that, if successful, it would be a low cost system that could be put in operation earlier than the alternatives, all of which required building additions. Accordingly, the Technical Division was requested to design and build two of these frames and demonstrate the feasibility of this concept. When this work was successfully completed, the Plant system using frames was accepted as the basis of design. The Plant units, which include the two built on the Technical Division project, were exhaustively tested at the Semiworks before being installed in the Plant. The degree of success of these units in Plant operation will indicate the extent to which this concept should be applied to other processing requirements.

W. E. Winsche, Research Manager Separations Engineering Division



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