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Hugh Kinsch
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Approved By: *Malcolm M. Haring*
Malcolm M. Haring
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ACTINIUM RESEARCH

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Problem Title - Separation of Radium from the Uranium Residues

Report By - J. G. Stites

Work Done By - J. G. Stites and A. W. Martin

INTRODUCTION

The extraction of radium from uranium residues was proposed either by the use of the old (Curie) process or by the development and use of a new process. Qualitative procedures for the extraction of radium from pitchblende are found recorded in the literature¹ but little quantitative data are available.

In order to become acquainted with the process and techniques involved, an attempt was made to use the method developed by the Curies, which essentially is being used by the Eldorado Gold Mines, Limited, Port Hope, Ontario.

DETAILED REPORT

A spectrographic analysis of the residues was run and the following elements were found present:

1. In large amounts; lead, calcium, silicon, copper, molybdenum, nickel, aluminum, and magnesium.
2. In moderate amounts; iron, vanadium, titanium, and potassium.
3. In trace amounts; zinc, sodium, tin, silver, barium, and uranium.

Two processes to remove these elements which have been studied are given here in detail. Figures 1 and 2 are flow sheets for these processes.

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Uranium is removed from pitchblende by leaching with nitric acid. The residue from this leach is radiferous and, apparently, is the sludge on which the following work was done using the Curie procedure.

Canadian pitchblende contains from one to seven per cent silver, which should be removed along with the uranium in the nitric acid treatment. However, in order to learn the process for removing silver, a sample of the cake was heated for 1.5 hours with sodium chloride, at least 100 per cent in excess of that amount necessary to react with the silver present. A temperature of 500° was used for this treatment. This temperature was arbitrarily chosen to prevent loss of silver chloride by volatilization.

This chloridized residue was extracted for three hours with a solution of sodium thiosulfate containing an amount of this salt which is ten per cent in excess of that required to react with the silver present.

The mixture after the hypo leaching was filtered. The filtrate was treated with sodium sulfide to precipitate silver sulfide.

The residue from the leaching was mixed with an equal weight of sodium hydroxide, 0.5 per cent by weight of barium sulfate, and sufficient water to cover the mixture. This very alkaline conglomerate was heated to boiling (120°) and digested for 1.5 hours. The purpose of this treatment was to dissolve the lead as sodium plumbite and the aluminum as sodium aluminate.

After digestion an attempt was made to filter the mixture but with no success. Buchner funnels with coarse and fine papers, vacuum, pressure, vacuum and pressure, and fritted glass funnels - all were used in an attempt to filter the slimy, gelatinous, and viscous mixture, but no filtration worked.

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Finally, the insoluble material was separated from the liquid by centrifugation. Even this procedure was slow and tedious and, if possible, should be avoided. The centrifugate was removed by decantation; the residue was washed with 10 per cent sodium hydroxide and recentrifuged.

The residue from this final centrifugation was placed in an iron reactor along with a twofold excess, by weight, of sodium carbonate and sufficient water to form a thick sludge. The reactor was heated until steam issued from the port. Then the vessel was closed and a pressure of 15 to 25 pounds maintained for six hours.

The carbonated product was then extracted for four hours with hydrochloric acid at a slightly elevated temperature after which time no further effervescence was noted.

The soluble chlorides of radium, barium, calcium, copper, nickel, iron, magnesium, and some lead are all in the supernatant liquid, while the residue contains the remaining lead, silica, and any residual silver. The mixture was difficult to filter because of the silica present.

After filtration some activity was still noted in the siliceous residues. Thus, these residues should be subjected to further carbonation to remove the remaining radiferous material.

The filtrate from the chloride leach was heated to boiling and treated with sulfuric acid until no more precipitate formed upon the addition of more acid. The mixture was filtered, and the residue containing the radium sulfate was retained.

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This residue is again subjected to a carbonation. The residue is then leached with hydrobromic acid. Traces of lead and other heavy metals were removed by the addition of a small amount of barium sulfide and barium hydroxide to the bromide solution.

The mixed bromides of barium and radium are then fractionally crystallized. In as much as this process was carried out using relatively small amounts of starting material, no attempt was made to take the radium through the second carbonation.

Since great difficulty was experienced in handling and filtering the alkaline, siliceous residues, and since there were no clear-cut separations, a procedure was developed which eliminated the silica in the first step and which made it possible to obtain clear-cut separations of other contaminating elements in the following steps.

In this modified procedure a two-pound sample of uranium residues was treated in 0.2-pound lots with sufficient concentrated sulfuric acid to cover the mixture and sufficient 48-per cent hydrofluoric acid to remove all silicon dioxide as silicon tetrafluoride. This reaction was carried out in Tygon bottles, and the silicon tetrafluoride was absorbed in sodium carbonate solution. Hydrofluoric acid was added to the mixture until no further fumes of silicon tetrafluoride were observed when a current of air was blown through the mixture.

When the sulfuric acid was added to the residues, fumes of nitrogen dioxide were evolved, which indicates that the cake had received a nitric acid treatment to remove the uranium. Silica represents approximately one-half of the bulk of the original material. The fluoride treatment removed this silica.

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The residue, after the removal of the silica, was agitated with two liters of water and neutralized with sodium hydroxide. By dilution and neutralization, the insoluble sulfates of lead, barium, and radium formed and were separated by filtration. The radiferous cake was digested with sodium carbonate for six hours at 15 to 25 pounds of pressure. Afterwards, the precipitate was washed thoroughly with water to remove soluble carbonates and sulfates and then treated with nitric acid. This treatment dissolves the insoluble carbonates such as barium, radium, and lead. Hydrochloric acid perhaps would be better than nitric acid for this purpose.

The acidified mixture was filtered and the filtrate set aside for further purification, while the residue was subjected to a further carbonation.

The filtrates from the two carbonations were combined, neutralized with sodium hydroxide, and made slightly acid with hydrochloric acid.

Hydrogen sulfide was bubbled into the acidified solution until no further precipitate formed. The mixture was filtered, the residue discarded, and the filtrate made slightly alkaline with ammonium hydroxide.

Again hydrogen sulfide was bubbled into the mixture and again a heavy black precipitate formed.

The precipitate from the acid treatment consists mainly of the sulfides of lead, copper, molybdenum, and bismuth, while the precipitate from the alkaline treatment consists mainly of the sulfides of nickel and iron and the hydroxides of aluminum and iron.

The filtration of the sulfides is a rather slow process because of the gelatinous nature of the third group hydroxides. However, the filtration is possible to carry out using ordinary techniques, i.e., with a Buchner funnel and filter paper.

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The filtrates from the acid and alkaline sulfide precipitations were combined and boiled. Then sodium sulfate is added to precipitate the barium and radium as the insoluble sulfates. This procedure yields a highly gamma-active precipitate which can be further carbonated, dissolved in hydrobromic acid, and fractionally crystallized.

Carbonate digestions for converting insoluble sulfates to carbonates are not complete reactions and must be repeated several times before all of the radioactivity is removed from the residues. However, carbonate fusions at temperatures up to 1100° have been tried with success. There is virtually a complete reaction with just one fusion. The fused mixture is quenched and washed with water to cool and remove soluble sulfates and carbonates, and then the acid leach is used as described previously. By being quenched while still very hot, the material breaks up readily, and solution of the soluble sulfates and carbonates is attained quite easily.

The handling of the uranium cake residues presents a rather serious health problem. It has been determined that there is 0.67 grams of radium per ton of residues; thus, in the normal course of events a large amount of radon is formed. This radon must be handled in such a way as not to be a health hazard.

The drums in which the uranium residues were shipped are virtually air-tight, so that when they are opened, a larger-than-usual amount of radon escapes. The air samples in the storage room show a tremendous increase in activity when the drums are opened. Although the cake is in the form of a fine, dry powder and could dust, it is surmised that the increase in activity is due to the radon rather than to the dust.

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The half life of the activity on the air sample paper has been determined to be of the order of 30 minutes. Radon, itself, would not remain on the paper, but, if any were to decay in passing through the paper its daughters would remain.

Radon-222 has a half life of 3.825 days and decays, with the loss of an alpha particle, to RaA^{218} , half life of 3.05 minutes, which in turn decays to RaB^{214} , half life of 26.8 minutes. If the radon were entrained for a time sufficient to decompose on the air sample paper and leave behind a residue of RaA^{218} , and if, for example, an hour were allowed between sampling and the first reading, only a very small amount of RaA^{218} would remain, and the bulk of the activity would be due to RaB^{214} . Since the half life of the air sample has been determined at approximately 30 minutes this hypothesis would apply.

Radon can be removed from radiferous mixtures by heating. This gas can also be adsorbed on charcoal at low temperatures.

Since large quantities of radon constitute a serious health hazard, the gas must be removed and trapped so as not to contaminate the air either in the working space or in the stack.

It is believed that radon-222 has a sufficiently long half life (3.825 days), so that if it were released into the stack gases, it would contaminate the air in the vicinity of the plant.

SUMMARY

1. The Curie method for the recovery of radium from uranium ores has been tested and proved.

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2. A new method for the recovery of radium has been devised and proved.
3. The problem of the handling of radon must be studied and remedies found before large scale production can begin.

REFERENCE

1. Pochon, M., Chem. and Met. 44, 7, 362 (1937).

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RADIUM RECOVERY PROCESS "CURIE PROCEDURE"

FIGURE 1

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FIGURE 2

Problem Title - Analysis of Uranium Residues
Report By - R. W. Moshier
Work Done By - N. E. Rogers

INTRODUCTION

The analytical procedures are being investigated and analyses are being run as a service for the work on the separation of radium from uranium residues.¹

DETAILED REPORT

At the present time analyses are required on the silica, lead, barium, and silver content of the uranium residues.

The method of analysis being used for silica is found in Scott² subject to the following modification.

After the sodium carbonate fusion, the mixture is leached with cold water to dissolve most of the silica. If hydrochloric acid is added immediately, both lead chloride and silica are precipitated. The residue and leachings are then processed separately with hydrochloric acid for separation of silica. The filtrates are then combined for subsequent determinations.

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1. Stites, J. G., Quarterly Report for General Research, MLM-405-3.
2. Scott's Standard Methods of Chemical Analysis, 5th Ed., N. Howell Furman, D., Van Nostrand Company, New York, pp. 860, 2164-66, and 2168 (1939).

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Problem Title - Power Growth and Decay of Actinium
 Report By - S. R. Orr and J. R. Parks
 Work Done By - S. R. Orr and J. R. Parks

INTRODUCTION

A study of the growth of power output in the decay of actinium seemed advisable in that growth was recognized but its magnitude unknown. Data from the solution of this problem are useful in our case to determine whether or not our present calorimeters can be used. If a different calorimeter is needed, the data would give an indication as to the specifications in the design and construction of the calorimeter.

The power output per curie of actinium at time of isolation is equal to 32.72×10^{-5} watts. This power is equivalent to 10.2 millicuries of polonium. Our most sensitive calorimeter at present has a sensitivity of 10 microvolts per millicurie. The precision of a single run on this size sample would be in the order of one per cent, if maximum stability could be achieved.

DETAILED REPORT

A Study of the Power Output of Actinium

As can be seen from Tables I and II, a rigorous calculation of the total power output of actinium would be quite involved.

Reduction of C_T , the total power output, to include fewer terms as expressed in Table II, can be made by inspection of the different half lives of the products. Since the computations were performed for

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five-day intervals of time, it is a good approximation to consider the short half-life products (37 minutes or less) as disintegrating almost immediately following the formation of a daughter product from a longer half-life parent. This approximation was used in the chain following actinon to actinium D. Another approximation was made in the consideration of the double branches of actinium A and actinium C by disregarding the lower per cent branches (5×10^{-4} per cent and 0.32 per cent) since their contribution to the total power output would be negligible.

The rise and decay with time of the activity of the disintegration products can be computed from the following differential equations.¹ Assuming that at time $t = 0$, the amount present in substance number is N_0 and that no disintegrations products are present:

$$dN_1/dt = -\lambda_1 N_1$$

$$dN_2/dt = \lambda_1 N_1 - \lambda_2 N_2$$

$$dN_3/dt = \lambda_2 N_2 - \lambda_3 N_3$$

The solution of this system of rate of growth differential equations, with the given initial conditions, is as follows:

$$N_1 = N_0 e^{-\lambda_1 t}$$

$$N_2 = \frac{N_0 \lambda_1}{\lambda_2 - \lambda_1} \left[e^{-\lambda_1 t} - e^{-\lambda_2 t} \right]$$

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$$N_3 = \frac{N_0 \lambda_1 \lambda_2}{(\lambda_1 - \lambda_2)(\lambda_2 - \lambda_3)(\lambda_3 - \lambda_1)} \left[(\lambda_3 - \lambda_2) e^{-\lambda_1 t} + (\lambda_1 - \lambda_3) e^{-\lambda_2 t} + (\lambda_2 - \lambda_1) e^{-\lambda_3 t} \right]$$

The corresponding activities or disintegrations per second, D, are obtained by multiplying by the decay constants. Therefore:

$$D_1 = -\lambda_1 N_1$$

$$D_2 = -\lambda_2 N_2$$

$$D_3 = -\lambda_3 N_3$$

The power developed is then the activity or number of disintegrations per second multiplies by the energy, E, of each particle emitted, i.e.;

$$C_1 = E_1 \cdot D_1 = E_1 \lambda_1 N_1$$

$$C_2 = E_2 \lambda_2 N_2 \text{ etc.}$$

With actinium the power developed is as follows, taking into account the branching of actinium and the approximations made previously. See decay scheme in Figure 1.

$$C_{\text{Total}} = C_{1B} + C_{2B} + C_3^B + C_{1A} + C_3^A$$

Where:

$$C_{1B} = 0.99 E_{1B} \lambda_1 N_0 e^{-\lambda_1 t}$$

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$$\begin{aligned}
C_{2B} &= \frac{0.99 E_{2B} \lambda_1 \lambda_{2B} N_0}{\lambda_{2B} - \lambda_1} \left[e^{-\lambda_1 t} - e^{-\lambda_{2B} t} \right] \\
C_3^B &= \frac{0.99 \lambda_1 \lambda_{2B} \lambda_3 N_0}{(\lambda_1 - \lambda_{2B})(\lambda_{2B} - \lambda_3)(\lambda_3 - \lambda_1)} \\
&\quad \left[(\lambda_3 - \lambda_{2B}) e^{-\lambda_1 t} + (\lambda_1 - \lambda_3) e^{-\lambda_{2B} t} + (\lambda_{2B} - \lambda_1) e^{-\lambda_3 t} \right] \cdot E \\
E &= \left[E_3 + E_4 + E_{5A} + E_{6B} + E_{7A} + E_{8B} \right] \\
C_{1A} &= 0.01 E_{1A} \lambda_1 N_0 e^{-\lambda_1 t} \\
C_3^A &= \frac{0.01 \lambda_1 \lambda_3 N_0}{(\lambda_3 - \lambda_1)} \left[e^{-\lambda_1 t} - e^{-\lambda_3 t} \right] \cdot E
\end{aligned}$$

The β^- -decay in the one per cent branch was neglected because of its low per cent, low energy, and short half life. C_3^A is another approximation but its validity can be easily shown.

Table III gives the numerical results of the constants appearing in previous equations. Values of the half lives and energies of decay were taken from Seaborg and Perlman's Table of Isotopes.² The energy of the α -decay was computed as the sum of the alpha energy and the recoil energy of the nucleus. The weighted mean value of the α -decay energy was taken when more than one energy level appeared. In β^- -decay it was taken to be equal to one-half the upper limit of the energy of the decay electrons. Calculations were done for five-day intervals for six months, and then for one-year intervals for ten years. The results for the relative power output at any time are given in units of the power output of one curie of actinium at the time of isolation.

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The results of the calculations as shown in Figures 2 and 3 show a marked increase in the power output with the maximum occurring at about 185 days after isolation.

Figure 2 gives the growth of power output of one curie of actinium, the time of isolation taken as the zero time, in units of the power output of one curie of actinium at the time of isolation. The curve is plotted on a five day interval up to the time of maximum power output. The maximum is seen to be reached in about 185 days.

In this time the power output of one curie has grown from the equivalent of 10 millicuries of polonium to the equivalent of six curies of polonium. In a matter of 10 days after isolation the power has grown from 10 millicuries of polonium to 0.75 curies of polonium equivalent. A sample of one curie of actinium would be adequate to obtain a growth curve in the early period, say up to 10 to 15 days after the time of isolation, with a microcalorimeter similar to 38 or 44. A study of the early period of growth should be of great interest to the chemists. A problem that will present itself will be putting a curie of actinium in a can sufficiently small to fit the microcalorimeters. A curie of actinium weighs approximately 13 milligrams.

After 15 to 20 days the power output would be such that a macrocalorimeter could be used to follow the growth of one curie and then after approximately 200 days to follow the decay. After the maximum has been reached a calorimetric study could begin leading to a good value of the half life of actinium. Using the curve obtained by a microcalorimeter it would

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TABLE I

RATES OF FORMATION OF THE PRODUCTS
IN THE ACTINIUM SERIES

1. $dN_1/dt = -\lambda_1 N_1$
2. $dN_{2A}/dt = 0.01 \lambda_1 N_1 - \lambda_{2A} N_{2A}$
3. $dN_{2B}/dt = 0.99 \lambda_1 N_1 - \lambda_{2B} N_{2B}$
4. $dN_3/dt = \lambda_{2A} N_{2A} + \lambda_{2B} N_{2B} - \lambda_3 N_3$
5. $dN_4/dt = \lambda_3 N_3 - \lambda_4 N_4$
6. $dN_5/dt = \lambda_4 N_4 - \lambda_5 N_5$
7. $dN_{6A}/dt = \sim 1.00 \lambda_5 N_5 - \lambda_{6A} N_{6A}$
8. $dN_{6B}/dt = 5 \times 10^{-6} \lambda_5 N_5 - \lambda_{6B} N_{6B}$
9. $dN_7/dt = \lambda_{6A} N_{6A} + \lambda_{6B} N_{6B} - \lambda_7 N_7$
10. $dN_{8A}/dt = 0.9968 \lambda_7 N_7 - \lambda_{8A} N_{8A}$
11. $dN_{8B}/dt = 0.0032 \lambda_7 N_7 - \lambda_{8B} N_{8B}$

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TABLE II

POWER OUTPUTS IN WATTS

1. C_{1A}	$= 0.01 E_{1A} \lambda_{1N_1}$
2. C_{1B}	$= 0.99 E_{1B} \lambda_{1N_1}$
3. C_{2A}	$= E_{2A} \lambda_{2A} N_{2A}$
4. C_{2B}	$= E_{2B} \lambda_{2B} N_{2B}$
5. C_3	$= E_3 \lambda_{3N_3}$
6. C_4	$= E_4 \lambda_{4N_4}$
7. C_{5A}	$= 1.00 E_{5A} \lambda_{5N_5}$
8. C_{5B}	$= 5 \times 10^{-6} E_{5B} \lambda_{5N_5}$
9. C_{6A}	$= E_{6A} \lambda_{6A} N_{6A}$
10. C_{6B}	$= E_{6B} \lambda_{6B} N_{6B}$
11. C_{7A}	$= 0.9968 E_{7A} \lambda_{7N_7}$
12. C_{7B}	$= 0.0032 E_{7B} \lambda_{7N_7}$
13. C_{8A}	$= E_{8A} \lambda_{8A} N_{8A}$
14. C_{8B}	$= E_{8B} \lambda_{8B} N_{8B}$

$$\text{Total Power } C_T = \sum_{n=1}^{8B} C_n$$

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TABLE III

VALUES OF CONSTANTS USED IN CALCULATIONS
LATEST VALUES AS GIVEN IN "TABLE OF ISOTOPES"
BY SEABORG AND PERLMAN

λ_1	$= 1.012 \times 10^{-9} \text{ sec}^{-1}$
λ_{2B}	$= 4.313 \times 10^{-7} \text{ sec}^{-1}$
λ_3	$= 7.163 \times 10^{-7} \text{ sec}^{-1}$
N_0	$= 3.656 \times 10^{19} \text{ Atoms}$
E_{1B}	$= 8.0 \times 10^{-16} \text{ Joules/Atom}$
E_{2B}	$= 9.621 \times 10^{-13} \text{ Joules/Atom}$
$\sum_{i=3,4,5A,6A,7A,8A} E_i$	$= 45.45 \times 10^{-13} \text{ Joules/Atom}$
E_{1A}	$= 8.061 \times 10^{-13} \text{ Joules/Atom}$

Watts per Curie of Actinium at

Time of Isolation $= 32.75 \times 10^{-5}$

Watts per Milli-curie of Polonium

Time of Isolation $= 3.201 \times 10^{-5}$

Weight of One Curie of Actinium

$= 0.0138 \text{ g}$

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FIGURE 1

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FIGURE 2

FIGURE 3

GROWTH AND DECAY OF ONE CURIE OF ACTINIUM

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be possible to obtain approximate values of the half lives of actinium and its first two-daughter products whose half lives are measured in days, provided the branching of actinium and the energies of the ejected particles in the whole chain are well known.

Figure 3 presents the long range view of the growth and decay. This curve is plotted in one-year intervals for 10 years. It shows that after equilibrium is reached, the decay is controlled by the long half-lived isotope actinium-227.

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2. Seaborg, G. T. and Perlman, I., Table of Isotopes, Rev. of Mod. Phys., 20, 4, 585-667 (1948).

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Problem Title - Separation of Actinium from Radium
Report By - P. M. Engle
Work Done By - P. M. Engle

INTRODUCTION

The process now being developed at Argonne National Laboratory for the separation of actinium from radium makes use of an ion exchange method for one step in the procedure. It is planned here to investigate the possible use of ion exchange techniques to accomplish the entire separation. An additional reason for starting the problem is to gain general information on the use of ion exchange resins and methods for the separation of other materials, such as the separation of radium from barium.

DETAILED REPORT

The work on the problem has not yet progressed to the point where experimental data have been obtained. A partial literature survey on ion exchange methods has been completed with particular attention given to the work done on difficult separations at Oak Ridge National Laboratory and Ames, Iowa.

Column-type operations are planned and one experimental apparatus has been completed. Four Amberlite synthetic resin cation exchangers are on hand, and a quantity of Dowex-50 is being obtained. The initial work will be done with barium and lanthanum solutions because of the similarity of these elements to radium and actinium, respectively. An effort has been made to analyze barium solutions turbidometrically, but this has been only

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partially successful. Further work on developing quick, reliable analytical methods for these elements must be done before any real progress can be made on the major phases of this problem.

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Problem Title - Process Development on Radium Recovery
Report By - J. H. Payne, Jr.
Work Done By - J. A. Rauscher

INTRODUCTION

The purpose of this problem is to develop a process for the recovery of radium from uranium residues. The chemistry involved in carrying out the process is described in a separate section of the Quarterly Report.¹

At present it is planned to study the process with equipment capable of handling 10 to 25 pounds of residue per day and to carry the process up to the final barium-radium separation. Definite plans for the small-scale equipment are contingent upon the working space which can be assigned to this program and upon the engineering information which may already be available. The Mallinckrodt Company, which is the current source of the residues, undoubtedly has solved some of the engineering and health problems which are certain to be encountered.

It is difficult to decide the extent to which operations should be carried out on a small scale. The costs of small equipment is out of proportion with its size and sometimes may even cost more than full sized equipment when single units must be constructed by hand. Also the cost of control equipment is nearly independent of size. On the other hand, considerable savings are realized on space requirements, shielding, and materials by the

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use of small equipment. Furthermore, if the cost of standardized types of engineering equipment are capitalized as a general development laboratory for research on problems involving radioactive materials, the cost eventually may be spread over a large number of problems.

DETAILED REPORT

Drawings have been completed for the construction of a digester for the hydrogen fluoride treatment for removal of silica. Stress-relieved, low-carbon steel appears to be a suitable material for handling the hydro-fluoric-sulfuric acid mixture provided soluble nitrates are first leached from the residue.

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1. Stites, J. G., The Separation of Radium from Uranium Residues, Quarterly Report for General Research, MLM-405-3.

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