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MOUND LABORATORY
Operated By
MONSANTO CHEMICAL COMPANY
MIAMISBURG, OHIO

MONTHLY TECHNICAL ACTIVITIES REPORT THROUGH OCTOBER 15, 1955

BY

D. L. Scott, J. F. Eichelberger, and F. C. Mead, Jr

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PRODUCTION PROGRAM

These projects are concerned with the production of alpha and neutron sources, and with the disposal of radioactive wastes. Work is being done to improve present methods and processes.

One polonium mock-fission neutron source, nineteen polonium-beryllium neutron sources and two alpha sources were shipped in September. The alpha sources and six of the polonium-beryllium sources were for non-AEC sites.

Work is in progress on the determination of small percentages of hydrogen in inert materials. The gamma ray of 2.2 Mev emitted when a slow neutron is absorbed by a hydrogen atom is detected and counted. A sodium iodide crystal four inches in diameter has been placed in service. The greater sensitivity of the larger crystal enables the use of a neutron source at least ten times smaller than was used with a sodium iodide crystal one and one-half inches in diameter. A hydrogen peak has been found with the large crystal. The equipment is being realigned in an attempt to determine differences in hydrogen concentration.

Work was done on a laboratory scale of disposing of protactinium waste from the HH-building. Tests were conducted on water containing HH-building filtrates and protactinium sludge filtrate. Tests were also run on water containing protactinium sludge. No additional treatment was necessary to reduce the activity to normal levels. Data for the Waste Disposal Operations are given in Table I.

TABLE I

DISCHARGE VOLUME	352,800	GALLONS
TOTAL ALPHA ACTIVITY	5.6	MILLICURIES
TOTAL BETA ACTIVITY	34.0	MILLICURIES
ACTIVITY DENSITY		
ALPHA	5	CT/MIN/ML
BETA	28	CT/MIN/ML

DEVELOPMENT PROGRAM

IONIUM PROJECT

The ionium project is directed towards the development and operation of a process for the extraction of ionium from partially processed raffinates from the Mallinckrodt uranium refinery. The process must produce ionium suitable for use by Los Alamos as a tracer.

To date the work on this project has been limited to the design of the process development facility and to those laboratory studies which can be performed with the equipment and materials which are available in the laboratory Equipment for the laboratory bench scale studies and for the "hot" development work is being investigated as to availability, cost, etc. so that when money is made available, procurement can start immediately.

Specifications on the product ionium have been received from Los Alamos.

Preparation of a large supply of simulated feed material suitable for bench scale studies is in progress. This simulated feed material will contain thorium-232, rare earth elements, trace amounts of uranium, but no ionium (thorium-230). It is planned to make use of the simulated feed material in both batch countercurrent and miniature mixer-settler studies. The batch countercurrent studies will include a tributyl phosphate extraction, a scrub, a partitioning, and a stripping operation. The batch countercurrent studies will give information which will be applied to the miniature mixer-settler operation.

The partially processed raffinates from which we will extract the ionium will be delivered to us as a fluoride slurry. Some preliminary work was done in which thorium fluoride was precipitated in a stainless steel beaker and then allowed to stand for approximately three weeks. When an attempt was made to remove the fluoride precipitate from the stainless steel beaker, it appeared to adhere very tenaciously, particularly to the bottom of the beaker. Dissolution of all the precipitate was possible by treatment with aluminum nitrate at 125°C in about two hours Formation of the aluminum fluoride complex was carried out in the same stainless steel beaker in which the fluoride precipitate was originally made. It was not practical to transfer the adhered fluoride precipitate from the beaker. From these observations, we should consider the use of stainless steel shipping containers which could be used for dissolution of the fluoride slurry in preparation for the extraction of the ionium.

THORIUM PROJECT

The thorium project was directed toward development and operation of a process suitable for extraction of thorium from Brazilian monazite sludge and AEC waste materials. The process must produce a thorium salt suitable for the preparation of metallic thorium of high purity.

Since the thorium project was cancelled, work has continued on the development of a flow sheet for the extraction of thorium from Brazilian hydroxide sludge. This study has consisted of development work on feed preparation, batch countercurrent solvent extraction, pilot plant operation, and analytical methods. From this work a flow sheet has been developed for the operation of the first purification cycle for a thorium refinery using Brazilian hydroxide sludge for the raw material. From this flow sheet we believe that we can extrapolate the operating conditions for a second cycle of purification.

This complete study is being compiled into a final report from which design and operating information will be available should anyone desire to build and operate a thorium refinery.

REACTOR PROGRAM

FUSED SALTS RESEARCH PROJECT

The Aircraft Nuclear Propulsion Project is considering the use of a fused-salt fuel system. Mound Laboratory has been assigned the problem of determining the phase relationships and physical properties of the components of some of the proposed fuel systems.

PHASE STUDIES

Additional compositions in the ternary system, $NaF-BeF_2-UF_4$, were examined petrographically confirming the existence of quasi-binary systems on the join between $NaBeF_3$ and $Na_7U_6F_{31}$ and on the join between Na_2BeF_4 and Na_3UF_7. Liquidus, solidus, and solid-transition data obtained from differential-thermal analysis also validated the existence of these quasi-binary systems. It was also shown rather conclusively by these examinations that no additional quasi-binary systems existed in the area bounded by the above joins.

Numerous examinations of compositions on the join between $NaBeF_3$ and UF_4 were made. Many of these showed the presence of only $NaBeF_3$ and UF_4 indicating that a quasi-binary system existed along the join. However, in many cases, UF_4 was absent and in its place was a green crystalline material which was similar to UF_4 but possessed lower refractive indices. X-ray diffraction patterns of this material ruled out the possibility that the unidentified crystalline phase was UF_4 altered by solid solubility. Attempts by differential thermal analysis and petrography to investigate the possibility of a ternary compound have proved inconclusive. Certain thermal effects shown by D.T.A. do not correspond to transitions which can be attributed to the phases present. However, all compositions on this join have given similar thermal breaks. It was noted that UF_4 was generally the phase present in rapidly cooled melts, whereas it was frequently replaced by the unidentified green crystalline phase in slowly cooled melts.

In several areas of the ternary diagram it has been difficult to obtain reproducible liquidus values. The thermal breaks for the liquidus temperatures often are poorly defined or may ascend or descend 5° to 15°C in each subsequent run. However, all transitions occurring below the liquidus temperature remain unchanged. Direct observation of the fluoride melts heated in an inert atmosphere has been helpful in solving this problem and in explaining some of the unassigned transitions obtained by D.T.A.

Rupture of D.T.A. sample tubes at the sealing weld often occurred at temperatures above the liquidus. This difficulty has been corrected by acetylene welding of the crimped end followed by the application of a thick nickel bead to completely cover the first weld. No breakdowns have occurred in more than twenty-five sample tube preparations welded in this two-step manner.

FILTRATION STUDIES

Work for the month was concerned with locating the valley which separates the phase area between Na_2BeF_4 and the $[NaF-UF_4]$ compounds. The results of these experiments are given in Table I.

TABLE I

LIQUIDUS COMPOSITIONS BY FILTRATION

NO.	STARTING COMPOSITION MOLE PER CENT			TEMPERATURE OF FILTRATION (°C)	COMPOSITION OF FILTRATE MOLE PER CENT		
	UF_4	BeF_2	NaF		UF_4	BeF_2	NaF
A	5	28	67	534	8.1	25.7	66.2
B	7	27.5	65.5	528	8.7	24.6	66.7
C	12	20	68	530	9.4	25.1	65.5

Apparently compositions A and B are on one side of this valley, while composition C is on the opposite hill. However, the filtrates taken at nearly the same temperature (528-534°C) from all three samples have nearly the same composition. This fact tends to indicate that a true valley has been found. More samples have been collected in this area, but the analyses are not completed, and the results will be reported next month.

Since the petrographic work is uncertain as to what is occurring along the line which connects UF_4 and $NaBeF_3$, some filtrations are being made along this line. The only analysis available now shows that a mixture of 20 mole per cent UF_4, 40 mole per cent BeF_2 and 40 mole per cent NaF when filtered at 574°C has a liquidus composition of 16.3 mole per cent UF_4, 44.0 mole per cent BeF_2 and 39.7 mole per cent NaF which is very close to this composition line and is not inconsistent with this line being quasi-binary.

PHYSICAL PROPERTIES

The densities and viscosities of two binary mixtures of LiF and BeF_2, four binary mixtures of NaF and BeF_2, and eleven ternary mixtures, including samples from the NaF, BeF_2, UF_4 system and four special samples from the NaF, LiF, BeF_2 system, were measured over the temperature range 500° to 900°C. The values of the viscosities and densities of these samples at 600° and 800°C are given in Table II. Plots of the logarithm of the viscosity as a function of the reciprocal of the absolute temperature gave linear curves similar to those previously obtained from other mixtures of these salts.

The densities of five ternary mixtures, in addition to those given in Table II, were measured this month. Densities of each mixture were measured at 50°C intervals between their liquidus point and approximately 900°C. The density values obtained at 600°C and 800°C are given in Table III.

A method of measuring heat conductivities of solids, liquids, or gases has been developed. The method is a differential transient one and therefore, if adapted to high temperature measurements, differential-thermal analyses of the sample can also be obtained. A simple set-up was assembled and used to measure the heat conductivity of water and of lucite at room temperatures. Two holes were drilled close to the axis of each of two solid aluminum cylinders, 3.0 inches long by 0.629 inches in diameter, into which were inserted four thermistors The thermistors formed a resistance bridge whose unbalance was fed to a Speedo-max recorder with a built-in integrator. One of the aluminum cylinders was lowered directly into a water bath controlled to within ± .0003°C. The other cylinder was successively insulated from the same bath by the five samples whose conductivities were to be measured, i.e. two water-filled jackets of 1.530 and 1.066 inches I.D. and a lucite cylinder 2.000 inches in diameter turned down to first 1.501 inches and then to 1.101 inches.

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TABLE II

VISCOSITY AND DENSITY OF FUSED SALTS

COMPOSITION MOLE PER CENT	600°C			800°C		
	η (Centipoise)	ρ (gm/cm^3)	η/ρ	η (Centipoise)	ρ (gm/cm^3)	η/ρ
ORNL NO. 338	9.90	2.038	4.86	3.70	1.941	1.91
56 NaF 16 LiF 28 BeF_2	6.04	1.976	3.06	2.84	1.895	1.50
64 NaF 5 LiF 31 BeF_2	6.89	2.090	3.30	3.61	1.991	1.81
63.5 NaF 7.5 LiF 29.0 BeF_2	6.99	2.074	3.37	3.28	1.972	1.66
69 LiF 31 BeF_2	7.11	1.917	3.71	3.18	1.837	1.73
50 LiF 50 BeF_2	22.2	1.981	11.2	5.94	1.901	3.62
57.3 BeF_2 42.7 NaF	33.6	2.110	15.9	6.26	2.000	3.13
18.3 BeF_2 81.7 NaF	SOLID	-	-	3.32	2.086	1.59
52.2 BeF_2 47.8 NaF	10.7	2.097	5.10	3.32	1.993	1.67
25 BeF_2 75 NaF	6.61	2.125	3.11	2.90	2.183	1.33
76 NaF 18 BeF_2 6 UF_4	11.0	2.663	4.13	3.31	2.543	1.30
70 NaF 24 BeF_2 6 UF_4	5.72	2.633	2.17	2.03	2.502	0.81
64 NaF 30 BeF_2 6 UF_4	8.42	2.600	3.24	3.30	2.475	1.33
58 NaF 36 BeF_2 6 UF_4	8.13	2.560	3.18	2.68	2.442	1.10
52 NaF 42 BeF_2 6 UF_4	12.5	2.574	4.86	3.50	2.447	1.43
76 NaF 20 BeF_2 4 UF_4	8.94	2.433	3.67	3.73	2.326	1.61
70 NaF 26 BeF_2 4 UF_4	9.71	2.476	3.92	3.84	2.345	1.68

TABLE III

DENSITIES OF FUSED SALTS

COMPOSITION	DENSITY AT 600°C (gm/cm³)	DENSITY AT 800°C (gm/cm³)
76 NaF 22 BeF_2 2 UF_4	SOLID	2.149
70 NaF 28 BeF_2 2 UF_4	2.310	2.209
64 NaF 34 BeF_2 2 UF_4	2.254	2.146
58 NaF 40 BeF_2 2 UF_4	2.226	2.116

The experimental procedure is as follows: With the bath maintained a sufficient time at temperature T_1 to obtain a constant bridge potential, the Speedo-max is set to sweep out zero area. The bath is then changed to a different temperature T_2 and controlled until the bridge output is again constant. If the bridge is well balanced this will be the same constant bridge potential as at temperature T_1. The total counts of the integrator during the transient interval are recorded. These counts are converted to area (A) in °C - seconds by the proportionality factor which is determined by the area in counts per second swept out when the sample side and dummy side are in separate baths controlled at two different temperatures. The heat conductivity of the sample can then be determined by the equation:

$$k = \frac{(T_2 - T_1)}{A}\left[C_{(s)} \quad \frac{r_1^2 - r_2^2}{4} \quad \frac{r_2^2}{2} \ln \frac{r_1}{r_2} + C_{(al)} \frac{r_2^2}{2} \ln \frac{r_1}{r_2} \right]$$

where k = thermal conductivity in cal - $°C^{-1}$ - sec^{-1} - cm^{-1}

$T_2 - T_1$ = change of bath temperature in °C

A = transient area in °C - sec

$C_{(s)}$ = heat capacity per unit volume of sample in cal - $°C^{-1}$ - cm^{-3}

$C_{(al)}$ = heat capacity per unit volume of aluminum cylinder in cal - $°C^{-1}$ - cm^{-3}

r_1 = O.D. of sample in cm

r_2 = I.D. of sample in cm (or O.D. of aluminum cylinder).

It was noticed that the apparent conductivity of water increased as the transient temperature difference across the water was increased. Thus in the case of the larger jacket, when the maximum temperature difference was 0.3 °C the apparent conductivity was about twice the true conductivity. The apparent conductivity plotted against the maximum temperature difference of the transient appeared to be essentially a straight line with the true value as an intercept. Accordingly a least-squares fit of the data was made giving the intercept and probable error as $(1.495 \pm .032) \times 10^{-3}$ cal $-$ sec^{-1} $-$ $°\mathrm{C}^{-1}$ $-$ cm^{-1} and a slope of 0.0045 cal $-$ sec^{-1} $-$ cm^{-1}. When the smaller jacket was used the result was $(1.416 \pm .014) \times 10^{-3}$ with a slope of .0002444. This is a reduction in slope by nearly a factor of twenty and must mean that convection currents were nearly eliminated in going from a thickness of water sample of 0.45 inches to 0.22 inches. The weighted mean of the two values for the conductivity is $(1.442 \pm .010) \times 10^{-3}$. The handbook quotes a value of 1.44×10^{-3} at 15°C. This experiment points out two useful possibilities; first that greater thicknesses of liquid may be used than are generally believed practical, and second that even if convection occurs an **extrapolation** to the true value may be possible.

One disadvantage of this method is that the heat capacity of the sample should be known. However it can be determined by determining $\Delta T/A$ for two or more different sample radii. Three values of $\Delta T/A$ were determined for lucite.

$k = .000458$ cal $-$ sec^{-1} $-$ $°\mathrm{C}^{-1}$ $-$ cm^{-1} and $C = .384$ cal $-$ $°\mathrm{C}^{-1}$ $-$ cm^{-3} from the O.D.'s of 2.000 and 1.501 inches, $k = .000446$ and $C = .368$ from the O.D.'s of 2.000 and 1.101 inches, and $k = .000441$ and $C = .357$ from the O.D.'s of 1.501 and 1.101 inches. The average of these values are $k = .000448$ and $C = .370$. From other sources a more accurate value of C is 0.42. If this value is assumed then the conductivity becomes .000486, .000481, and .000467 respectively for the O.D.'s of 2.000, 1.501, and 1.101 inches.

In conclusion, the real advantages of this method of measuring conductivities seem to be that the heater is conventional and is located in the bath (or temperature equalizing block) leaving only a temperature detecting device interior to the sample. The temperature rise of the bath can be obtained also by conventional equipment. If a heater were used interior to the sample, the presence of the heater would make the measurement of temperature much more difficult.

ANALYTICAL METHODS

Work has continued on improving the precision and accuracy of the X-ray fluorescence method. The scintillation counter equipment has been set-up and tests are being made. Much time has been lost repairing faulty circuitry. A borrowed pulse height analyzer has been added which reduces background counts from 300 - 400 ct/min to 1 - 2 ct/min. The counting equipment has been tested with a Po-210 source and counting probability errors are now reduced to about 0.1%.

Mylar plastic has been tested as a window material in the X-ray liquid cells. It has been found unsatisfactory because of ionic diffusion through the material.

PROTACTINIUM SEPARATION PROJECT

A program has been undertaken to isolate and purify a gram of protactinium-231. This material is important since it will provide a relatively stable isotope to study the physical and chemical properties of the 27-day protactinium-233 which will be created in the Th-232 → Pa-233 → U-233 sequence in thorium-breeder blankets.

PROCESSING

The concentration of the protactinium in the HH Building is being continued according to the process involving the digestion of the raffinate residue as received from the Mallinckrodt Chemical Company with 2 normal commercial-grade hydrochloric acid. This processing procedure has been described in earlier technical activities reports. Laboratory work has indicated that additional washing of the carrier after treatment with 12 per cent sodium hydroxide is desirable. Therefore this material is now being washed twice with hot water before it is transferred to the R-building.

Breakdown and malfunction of the processing equipment has caused considerable delays. The plugs which had been originally installed in the glass lining of one of the Pfaudler kettles failed during one of the runs. The hydrochloric acid solution leaked into the steam chest and destroyed some of the condensate lines and the steam trap. Teflon plugs fitted with tantalum compression bands were installed. These plugs have been satisfactory to date. The pumps used to transfer the various slurries obtained during the processing have not operated satisfactorily. Nearly constant attention by the maintenance force has been required.

The filtration of the neutralized waste liquors from the process became a major problem. Three to four days were required to dispose of the waste from one drum. After extensive testing the waste disposal department and health physics department agreed to handle the neutralized waste liquors in the facilities at the Waste Disposal building. Thus no filtering of the neutralized waste liquors will be necessary in the HH-building. Elimination of this slow filtering operation will increase the processing rate considerably.

A process control laboratory has been set up on the second floor of the HH-building. Thus the operating personnel can follow the processing steps directly without transportation of various samples to the R-building for routine analysis.

Each drum of raffinate residue is being examined to determine the behavior of the material during the first two steps of the concentration process, i.e., the solution of the raffinate residue and the precipitation of the internal carrier. The failure of the first step, the solution step, to recover more than 40 per cent of the protactinium from the residue in one batch (a mixture of drums 23 and 40) had indicated the necessity to predict the performance of each batch and avoid any such low recoveries in the future.

In the analysis of a sample from each drum, an attempt is made to simulate in the Laboratory the first two steps of the concentration process as carried out in the plant with one exception, the phases are separated by centrifuging rather than allowing for settling of the solids and decantation. This change may give a somewhat optimistic yield of protactinium but will not mask any gross differences among the drums.

The samples from the drums are being analyzed with respect to the location and accessibility of the drums in the storage lot.

The following procedure is being used: Two five-gram samples of each drum are stirred with appropriate amounts of tap water and commercial-grade hydrochloric acid for two hours and then allowed to stand overnight. After separation of the phases by centrifuging, water-softener-grade sodium chloride is added to each supernate. The mixture is heated to 90°C with stirring for one hour and allowed to stand overnight. After centrifuging, the carrier precipitate is washed with 0.5 N hydrochloric acid. The wash liquor is added to the supernate which is equivalent to the waste liquor.

The recovery of the protactinium from the waste liquor is effected by adding titanium carrier to the combined supernate and wash liquid from the carrier precipitation. This solution is heated at 95°C for one hour. After cooling to room temperature the precipitate is separated by centrifuging. The precipitate is washed with water and recentrifuged. The resultant liquids are discarded.

The residue remaining from the first HCl solution step, the carrier precipitate from the salting step, and the carrier precipitate from the supernate plus wash are each digested in concentrated sulfuric acid. Both of the carrier precipitates are completely soluble in the sulfuric acid. The residue from the first solution step is only partly soluble in sulfuric acid, but the soluble portion contains virtually all the protactinium. The sulfuric acid solutions are each mixed with distilled water and hydrochloric acid to make the resultant solutions 9 normal in sulfuric acid and 6 normal in hydrochloric acid. These solutions are extracted with a mixture of di-isobutylcarbinol and Amsco. The organic mixture being previously saturated with six normal hydrochloric acid. It appears that the protactinium is extracted into the organic phase with a partition coefficient of about 40 to 1.

The organic phases, containing the protactinium, are all counted in the gamma pulse height analyzer at 27, 95, and 300 kev. Since the counting geometry is the same in all cases, the results can be expressed as percentages of protactinium in the undissolved residue and the carrier precipitate with the protactinium in the titanium precipitate representing the loss of protactinium to the waste solution. Table I contains analyses of six drums by this method, with the last column expressing a relative protactinium content

TABLE I

PROTACTINIUM ANALYSES

	PER CENT PROTACTINIUM IN		RELATIVE PROTACTINIUM
DRUM NO.	RESIDUE	CARRIER	CONTENT OF DRUM
31	8.4	75.4	1.00
32	3.9	71.4	0.99
33	3.5	73.7	1.10
34	2.4	81.5	1.35
30	24.5	63.3	1.12
20	3.2	84.2	0.89

EXPERIMENTAL DEVELOPMENT

Small-scale ion-exchange column experiments are in progress to determine suitable operating conditions for further purification of the concentrate produced in the HH building facilities. This investigation is a continuation of the experiments described in the September Technical Activities Report, Central File No. 55-9-32. The feed solution used, as well as the column operating conditions, are the same as previously described. In order to determine the influence of further increasing the resin bed depth, runs with bed depths of 6 centimeters ($D = D_3$) and 8 centimeters ($D = D_4$) were carried out. Two runs were made at each bed depth, one at the lower feed concentration ($F = F_1$) and one at the higher feed concentration ($F = F_2$). The distribution of the 27 kev activity is shown in Table II. As in the previous investigations, essentially all of the protactinium appears in the elution fraction.

The behavior of the color bands formed on the columns during the runs had suggested that increasing the column length would increase the chemical purity of the protactinium fraction. However, as indicated in Table III, the concentration factors obtained do not show this to be true. There even may be a tendency for the concentration factor to decrease with increasing column length.

TABLE II

DISTRIBUTION OF 27 KEV GAMMA ACTIVITY IN ION-EXCHANGE EFFLUENTS

(Per Cent of Total Feed)

COLUMN 5 (D_3F_1)	COLUMN 6 (D_3F_2)	COLUMN 7 (D_4F_1)	COLUMN 8 (D_4F_2)
FEED EFFLUENT			
0.81	0.49	0.50	0.30
WASH EFFLUENT			
4.7	1.7	0.20	0.30
ELUTION EFFLUENT			
ACCUMULATIVE PERCENTAGES FOR SUCCESSIVE 4 ML ALIQUOTS		ACCUMULATIVE PERCENTAGES FOR SUCCESSIVE 10 ML ALIQUOTS	
0.01	0.08	0.02	0.09
0.03	0.10	1.3	3.7
0.05	0.17	64.2	59.0
1.6	1.6	85.6	84.0
14.4	15.2	91.1	92.7
49.0	54.7	94.4	96.0
73.6	77.6	96.4	97.7
83.3	88.1	97.8	98.7
88.5	93.3	98.5	99.4
91.7	96.2	99.1	100.0
93.1	97.8		
94.6	98.2		
95.7	99.0		
95.7	99.6		
95.7	100.0		
96.2	100.4		
96.9	100.7		
97.3	101.0		
STRIP EFFLUENT*			
7.3	4.9	6.0	4.0

*THESE VALUES PROBABLY FROM ACTIVITY NOT ASSOCIATED WITH PROTACTINIUM

TABLE III

WEIGHTS OF DRIED RESIDUES FROM ELUTION EFFLUENTS AND CONCENTRATION FACTORS FOR EIGHT STUDIES OF COLUMN OPERATION

COLUMN NUMBER	SYMBOL	TOTAL RESIDUE FROM ELUTION EFFLUENT (grams)	TOTAL RESIDUE FROM FEED (grams)	CONCENTRATION FACTORS FOR COLUMN OPERATION	WEIGHT OF RESIDUE FROM ELUTION EFFLUENT EVAPORATED TO DRYNESS PER GRAM OF RAFFIDUE ($gm \times 10^4$)
1	D_1F_1	0.0175	0.4795	24.4	2.01
2	D_2F_1	0.0229	0.4795	20.9	2.63
3	D_1F_2	0.0244	0.4795	19.6	2.80
4	D_2F_2	—	—	—	—
5	D_3F_1	0.0244	0.4795	19.6	2.80
6	D_3F_2	0.0310	0.4795	15.5	3.56
7	D_4F_1	0.0364	0.4795	13.2	4.18
8	D_4F_2	0.0344	0.4795	13.9	3.95

A series of experiments were carried out on the slurry obtained in the concentration of drum 19 in order to improve the procedure for dissolving this slurry to prepare the ion-exchange column feed solution. The concentrate from drum 19 contained 87 per cent of the protactinium in the original raffinate residue and had a volume of 9.5 liters. The slurry liquor obtained by centrifuging a sample of the slurry had a basicity equivalent to being 1.25 molar in sodium hydroxide.

Table IV shows the effect of final acid normality, milliequivalents of hydrochloric acid per milliliter of slurry, and washing with water (prewash) on solution efficiency and solution stability. The "final acid normality" is based on the known basicity of the slurry supernate with no consideration being given to the basicity of the solids in the slurry. A one-hour solution time was employed. Increasing the solution time to three hours gave little or no improvement. The water wash (prewash) consisted of a five-fold dilution with water followed by a ten-minute stirring period. "Unstable" solutions are those which showed cloudiness either immediately or on standing.

Prewashing generally increased the solution efficiency. This increase was most marked with 8- and 9-normal acids when a greater quantity of acid was used. However, under the same conditions, prewashing decreased the stability of the "9-normal" acid solution. When the amount of acid used was decreased by about one-half, and the slurry was prewashed, the 8- and 9-normal acids yielded both high solution efficiency and solution stability.

[redacted]

TABLE IV

SOLUTION OF SLURRY FROM CONCENTRATION PROCESS

EFFECT OF HYDROCHLORIC ACID CONCENTRATION AND QUANTITY AND OF PREWASH OF SLURRY ON SOLUTION EFFICIENCY AND STABILITY

FINAL ACID NORMALITY	HCl IN SLURRY (meq/ml)	PER CENT 27 KEV GAMMA ACTIVITY DISSOLVED		PER CENT 300 KEV GAMMA ACTIVITY DISSOLVED		STABILITY OF SOLUTION	
		NO PRE-WASH	PRE-WASH	NO PRE-WASH	PRE-WASH	NO PRE-WASH	PRE-WASH
6	14.5	89.9	95.8	94.1	97.4	U*	S**
7	19.9	85.5	96.7	98.0	97.9	U	U
8	27.9	66.0	96.2	95.2	97.0	U	U
9	41.2	66.5	96.1	71.0	96.3	S	U
8	15.	84.0	96.9	89.0	98.0	U	S
9	15.	92.0	96.0	94.8	96.0	U	S

*U INDICATES UNSTABLE

**S INDICATES STABLE

POLONIUM-210 AND ACTINIUM-227 RESEARCH

Research on these isotopes is confined to completion of long term projects which were initiated prior to fiscal 1956.

On March 14, 1955 the results of the calorimetric half-life measurements of Ac-227 were reported. The grand mean average of the six individual values reported then was $T_{½}$ = 21.871 years with an internal consistency of ± 0.018 years and an external consistency of ± 0.029 years. Since then another series of power measurements have been made. The results now becomes $T_{½}$ = 21.862 years with ± 0.012 years external consistency. The probable error is therefore about 5 days, or 0.06 per cent.

[redacted]