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MOUND LABORATORY
Operated By
MONSANTO CHEMICAL COMPANY
MIAMISBURG, OHIO

MONTHLY TECHNICAL ACTIVITIES REPORT

AUGUST 15, 1955

BY

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POLONIUM-210 OPERATIONS

This program is concerned with the production of polonium sources and the disposal of radioactive waste. Work is being done to improve the present methods and processes.

Ten polonium-beryllium neutron sources and two alpha sources were made and shipped in July. Seven of the neutron sources were for other AEC sites.

Data for the Waste Disposal Operations are shown in Table I.

TABLE I

DISCHARGE VOLUME	472.200	GALLONS
TOTAL ALPHA ACTIVITY	7.9	MILLICURIES
TOTAL BETA ACTIVITY	35.7	MILLICURIES
ACTIVITY DENSITY		
ALPHA	5.0	C/MIN/ML
BETA	22.0	C/MIN/ML

LIVERMORE PROJECT

The Livermore program is concerned with production of lithium deuteride blocks, pressed and canned to meet specifications of a Livermore Laboratory research project.

INTRODUCTION

All of the required number of cans containing enriched material were completed and were sent to Livermore, California, in shipments numbered 4 and 5. No more of this type material will be processed. The "C" and "D" type material will be processed at Livermore. About 48 kilograms of normal salt was weighed and pressed into blocks. Thirty-four kilograms remain to be pressed and then all the pressing will be completed.

OPERATIONS

DRY-BOX ATMOSPHERE. During the canning operations the dew point remained well below the required -77°F . The oxygen content remained well below the tolerance levels.

PRESSING, CURING, SEALING, AND FINAL OPERATIONS. Twenty-six batches of enriched material were cured and sealed into aluminum cans. All this material was processed and sent to Livermore as shipment number 5 and consisted of the following:

- 260 size 2-inch standard cans
- 65 size 2-inch dimple cans
- 181 size 6-inch standard cans
- 66 size 6-inch dimple cans
- 60 size 2-inch standard cans (as samples for analysis)

The remainder of the normal material is now being processed. About 82 kilograms will be required to be pressed into about 3,400 blocks. At present about 48 kilograms has been pressed into 2-inch blocks.

Since Livermore was not able to deliver the "C" type material before the dry-boxes were converted from enriched to normal material processing, Livermore will now process the "C" and "D" type materials into sampler disks. This change occurred because the experiments on the enriched material will be conducted very soon. With the completion of the processing of the normal material, the project will be concluded.

All necessary equipment and the small aluminum containers used to make the sampler disks will be shipped to Livermore on August 16, 1955.

ANALYSIS. The hydroxide analysis of the enriched material in shipment number 5 varied from 200 to 1,900 parts per million. These values were all upper limits since impurities were found to contribute to the results as hydroxides. It was discovered, by mass spectrometer analyses, that a considerable amount of nitrogen was given off during the hydroxide analyses. Investigation confirmed the possibility that nitrides of lithium were formed during the preparation of the enriched metal. Nitride impurities from 800 to 7,000 parts per million were reported by Oak Ridge in the enriched material.

ALUMINUM CAN PREPARATION. The cans and lids required for sealing all the remainder of the normal salt have been completely processed and the work in that department has been concluded.

THORIUM PROJECT

The thorium project is 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.

INTRODUCTION

Since the thorium project was cancelled in May, work has continued on the development of a process flow sheet for the extraction of thorium from Brazilian hydroxide sludge. The pilot plant solvent extraction work has continued but all other work on this project has been stopped.

SOLVENT EXTRACTION

Seven pilot plant runs were made during this period. The column conditions are presented at the end of this report in flow sheet form. It is believed that the conditions are more clearly stated in this manner. An explanation of this new system follows:

A horizontal line through the column depicts an interface e.g., in column 1, run number 7, the interface is at the top showing that this column is aqueous continuous. If the interface is at the bottom, the column is organic continuous. The numbers in the circles are flow ratios taking feed into the first column as unity. All other column ratios are based on this. The arrows enter and leave the columns as nearly as possible to the position of the actual piping. The flow rates and stream components are also shown. Frequency and amplitude of pulsing are shown below the column as well as either the notation vent. "in" or vent. "out". This notation refers to the block holding the Venturi tubes on the pneumatic pulser. When the block is "in" it allows for maximum coalescence time after the downward pulse, and when "out" it allows for maximum coalescence time after the upward pulse. Each full column has 24 feet containing pulse plates.

The analytical results (Table I) are those of samples taken about 11 hours after the columns were "on stream", so the conditions should be close to actual column equilibrium.

Analytical results are available on six sets of samples from run number 7 (Table I) and plots of concentration *versus* time indicate that 2½ hours "on stream" gives approximately equilibrium conditions. The rare earth data for this run are not as good as those for run number 6 (Table II); the major change was in the acid concentration of the feed. The acid concentration of the feed in run number 6 was 3.4 *N* and of run number 7 was 4.2 *N*.

Runs numbered 8, 9, 10, and 11 were made using the fifth (solvent clean-up) column only. Samples were taken but were not analyzed since the column did not run correctly for a sufficient length of time to reach equilibrium. The flow sheets for these four runs appear at the end of this report.

Run number 8 was made using the conditions of the Catalytic Construction Company flow sheet. A flow rate check showed the sodium carbonate solution to be entering at 0.25 gal/hr. instead of the desired 0.08 gal/hr., but the column still appeared to be caustic deficient. It was decided to try again; however, the lowest pump speed delivered 0.12 gal/hr. This resulted in the conditions for run number 9. The column flooded on two trials

TABLE I

ANALYTICAL RESULTS RUN NUMBER 7

STREAM	THORIUM (gm/l)	URANIUM (gm/l)	NITRIC ACID (N)	RATIO (Th/U)	RARE EARTHS		
					Sm (ppm)	Gd (ppm)	Dy (ppm)
ORGANIC FROM 1ST COLUMN (EXTRACTION)	21.88	0.57	0.269	38.3	800	550	6
ORGANIC FROM 2ND COLUMN (AQUEOUS SCRUB)	22.22	0.50	0.266	44.5	180	45	18
ORGANIC FROM 3RD COLUMN (ORGANIC SCRUB)	27.01	0.17	0.166	159.	-	-	-
ORGANIC FROM 4TH COLUMN (STRIP)	0.12	0.63	0.022	0.190	-	-	-
RAFFINATE FROM 1ST COLUMN	4.89	0.23	0.109	21.2	-	-	-
TNT* PRODUCT FROM 3RD COLUMN	30.43	0.0013**	0.442	23400.	55	20	6
REFLUX 2ND TO 1ST	12.79	0.23	0.712	55.6	15,000	7,800	1,400
REFLUX 4TH TO 3RD	46.59	0.081**	0.448	57.5	-	-	-

*THORIUM NITRATE TETRAHYDRATE

**BY FLUORIMETER

TABLE II

RARE EARTH ANALYSIS RUN NUMBER 6

STREAM	Sm (ppm)	Gd (ppm)	Dy (ppm)
ORGANIC FROM 1ST COLUMN	~200	~100	~100
ORGANIC FROM 2ND COLUMN	N.D.*	1.2	5.5
TNT PRODUCT FROM 3RD COLUMN	2.1	0.8	0.8
REFLUX 2ND TO 1ST	4000	1300	400

*N.D. - NOT DETECTABLE

under these conditions. Increasing the sodium carbonate solution to 0.24 gal/hr. was still unsatisfactory. This scheme was then tried in batch countercurrent experiments and emulsions were obtained under caustic deficient conditions.

Run number 10 was based on the results of the batch countercurrent experiments but the column still flooded after being "on stream" a little over an hour with the organic phase continuous. The column was then operated with the aqueous phase continuous, but the result was poorer.

Run number 11 was set up to introduce the sodium carbonate solution into the top of the column instead of the middle, but the column flooded even under those conditions. The pulsing rate was then reduced from 60 to 35 cycles per minute and the column seemed to work better. By this time the supply of organic overflow from column 4 was exhausted and the experiment was terminated.

Run number 12 was set up using only 12 feet of stripping section since analytical results from previous runs indicated low thorium in the overflow from column 3. This was not the case in this run as can be seen in Table III.

TABLE III
ANALYTICAL RESULTS RUN NUMBER 12

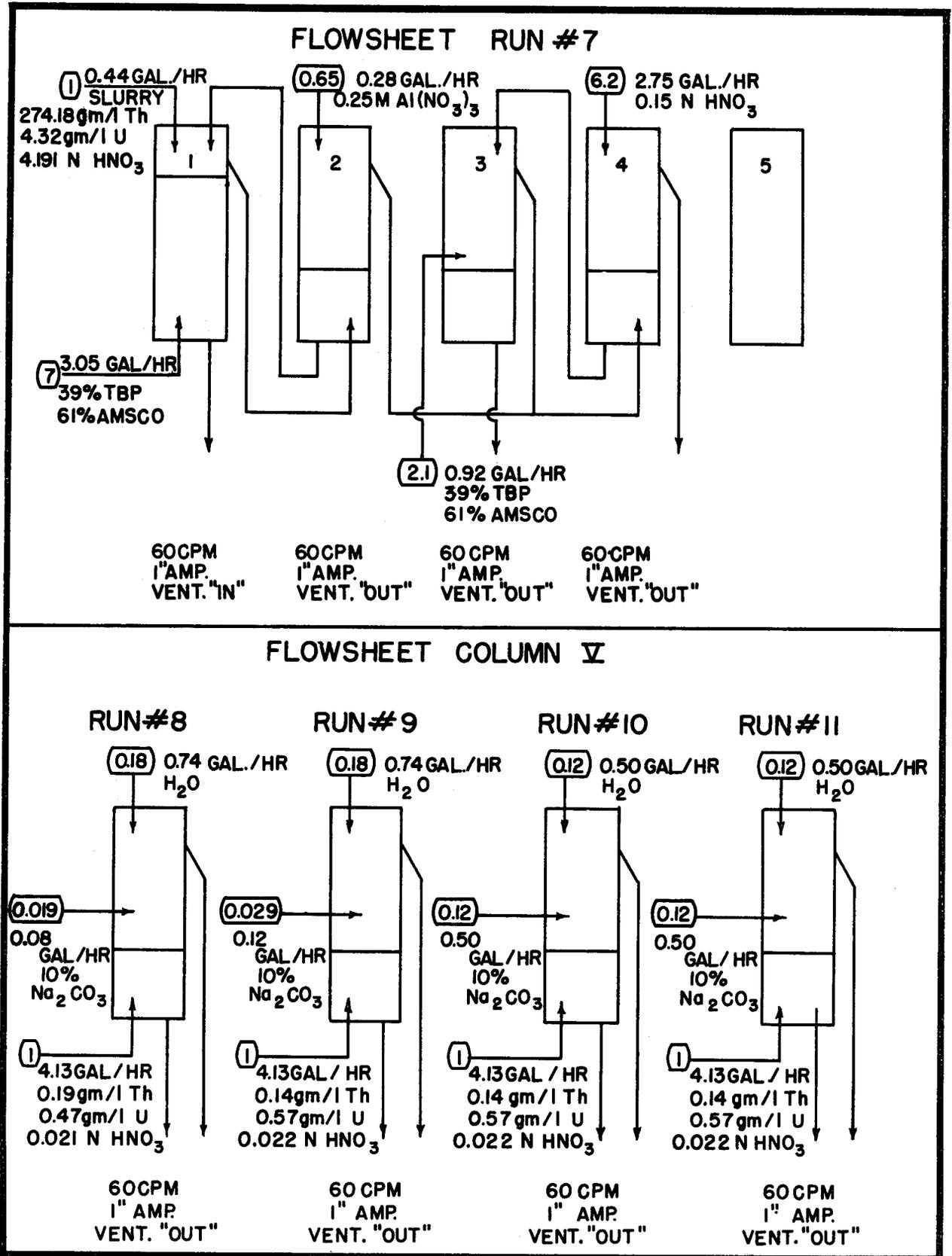
	Th (gm/l)	U (gm/l)	Th/U
ORGANIC FROM 3RD COLUMN	4.54	1.28	3.6
TNT PRODUCT FROM 3RD COLUMN	52.46	0.0006*	87,430.
ORGANIC FROM 5TH COLUMN	0.16	0.03	5.3

*FLUORIMETER

The high thorium concentration being fed to the fifth (solvent clean-up) column caused large quantities of thorium hydroxide to precipitate. Therefore this was not a satisfactory check on the operation of column 5 at 35 cycles per minute, although it seemed to work more satisfactorily even with the excess of thorium.

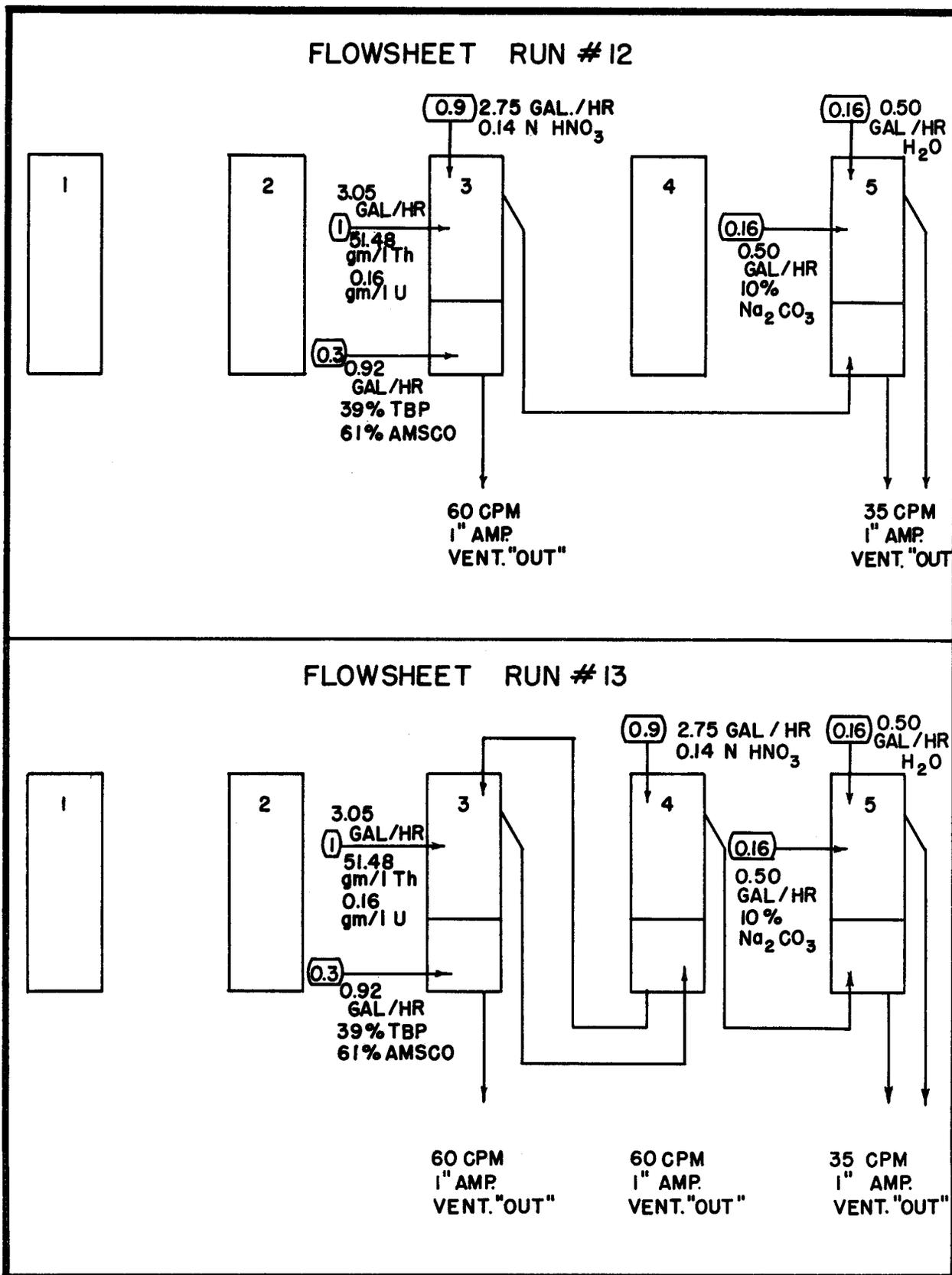
No analyses are presented for run number 13 because mechanical experimentation during the run interfered with the attainment of equilibrium. It was determined and $\frac{3}{4}$ inch amplitude, instead of 1 inch, caused some of the tendencies for emulsion formation to disappear.

All of the solvent on hand has been cleaned up. It is now planned to run the remainder of the slurry feed through the columns. Then the clarified feed available from filtration experiments will be used.



5890

Figure 1



5891

Figure 2

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

The differential thermal analysis and petrographic studies have been directed toward the determination of quasi-binaries and compatibility triangles throughout the ternary diagram, NaF-BeF₂-UF₄. Some differential thermal analysis data for the preliminary establishment of the liquidus surface in the high sodium fluoride end of the ternary are presented in Table I. Heating and cooling rates of 0.5°C per min., 0.8°C per min., and 1.3°C per min. were used, the values given in Table I being based primarily on the heating curves.

The study of thermal gradient quenches of various compositions has been suspended in favor of the study of equilibrium conditions in slowly cooled melts. A quasi-binary has been established on the join between Na₃UF₇ and Na₂BeF₄. Further attempts to produce β-Na₃UF₇ have proven unsuccessful. A quench was made at 497°C, but both petrographic and X-ray examination of the material showed it to be α-Na₃UF₇. A quench at 460°C to produce the β form yielded diffraction lines of Na₂UF₈ and some strong, unassigned lines. A study of the detectability of beryllium compounds in the presence of uranium compounds has been initiated. A sample containing about 33 weight percent NaBeF₃ in the presence of Na₇U₈F₃₁ gave no NaBeF₃ lines. A second sample of 37 weight percent Na₂BeF₄ in Na₂UF₈ gave extremely faint lines of the beryllium compound.

TABLE I
RESULTS OF DIFFERENTIAL THERMAL ANALYSIS STUDIES

COMPOSITION MOLE PERCENT			TRANSITIONS °C					LIQUIDUS °C
UF ₄	BeF ₂	NaF	S-S	S-S	E	UNASSIGNED HEATING	UNASSIGNED COOLING	
.16	4	80		450	483	500, 532	553, 520	586
16	8	76		450	483	500, 540	553*, 519	567
16	12	72	320	450	483	510	510	542
16	16	68	320		483	521	519	566
16	20	64	320	342		504	498*	575
16	24	60	320	342			545	555
12	6	82		450		501, 540	520*	565
12	28	60	320	342				528
19	32	49		342		360*, 369		588

*POORLY DEFINED TRANSITION OR DOUBTFUL BECAUSE DATA IS INSUFFICIENT

PHYSICAL PROPERTIES

The densities of seven ternary mixtures were measured this month. The compositions varied from 46 mole per cent NaF, 42 mole per cent BeF₂, 12 mole per cent UF₄ to 46 mole per cent NaF, 46 mole per cent BeF₂, 8 mole per cent UF₄. Densities of each mixture were measured between their liquidus point and approximately 900°C.

The viscosities of seven ternary mixtures of sodium, beryllium, and uranium fluorides were measured over the temperature range 500 to 900°C. The viscosity of a special salt mixture from ORNL also was measured from 350 to 900°C.

TABLE III
DENSITY AND VISCOSITY OF FUSED SALTS

SAMPLE MOLE % SALT	600°C			800°C		
	(CENTIPOISE)	$\frac{(gm)}{cm^3}$	$\frac{cm^4 \times 100}{sec}$	(CENTIPOISE)	$\frac{(gm)}{cm^3}$	$\frac{cm^4 \times 10^2}{sec}$
58 NaF 30 BeF ₂ 12 UF ₄	12.8	3.165	4.0	3.4	3.002	1.1
52 NaF 36 BeF ₂ 12 UF ₄	12.8	3.105	4.1	2.8	2.985	0.94
46 NaF 42 BeF ₂ 12 UF ₄	17.7	3.41	5.2	3.4	3.28	1.04
76 NaF 14 BeF ₂ 10 UF ₄	SEMI SOLID			4.9	2.902	1.7
70 NaF 20 BeF ₂ 10 UF ₄	11.9	3.190	3.7	2.8	3.039	0.92
64 NaF 26 BeF ₂ 10 UF ₄	10.0	3.130	3.2	3.2	2.962	1.08
58 NaF 32 BeF ₂ 10 UF ₄	12.7	2.956	4.3	2.7	2.801	0.96
52 NaF 38 BeF ₂ 10 UF ₄	10.6	2.922	3.6	2.7	2.796	0.97
46 NaF 44 BeF ₂ 10 UF ₄	28	2.790	10.0	6.3	2.928	2.2
ORNL #EE378SC	8.8	2.317	3.8	3.0	2.205	1.4

The results for the following ternary mixtures show a break-point in the graphical representation of density vs temperature which would indicate a phase separation. The middle reading of temperature and density as recorded in Table III is taken at the break-point while the first and third readings are additional points from which the slopes above and below the break-point may be determined.

TABLE III
BREAK POINTS FOR TERNARY MIXTURES

SAMPLE	TEMP. (°C)	DENSITY (gm/cc)
58 MOLE PER CENT NaF, 34 MOLE PER CENT BeF ₂ , 8 MOLE PER CENT UF ₄	550	2.817
	712	2.700
	900	2.599
52 MOLE PER CENT NaF, 40 MOLE PER CENT BeF ₂ , 8 MOLE PER CENT UF ₄	550	2.805
	698	2.697
	900	2.593
46 MOLE PER CENT NaF, 46 MOLE PER CENT BeF ₂ , 8 MOLE PER CENT UF ₄	500	2.811
	693	2.695
	900	2.580

Room temperature measurements of the specific heats of the ternary salts used in viscosity determinations were made using present calorimeters. These results agree closely with the sum of the specific heats of the individual pure salts which comprize the ternary. By taking the complete sample from the viscosimeter, inhomogeneties within the sample; introduced by slow cooling of the melt, can be ignored. Results are given in Table IV.

TABLE IV
SPECIFIC HEATS AT ROOM TEMPERATURE

SAMPLE MOLE PER CENT	(25.5° - 28.5°C) CALORIES PER GRAM PER °C		PER CENT DIFFERENCE MEAS. W. R. T. CALC.
	MEASURED	CALCULATED FOR MIXTURE	
58 NaF 30 BeF ₂ 12 UF ₄	0.1759	0.1777	-1.0
76 NaF 14 BeF ₂ 10 UF ₄	0.1907	0.1865	+2.2
70 NaF 20 BeF ₂ 10 UF ₄	0.1881	0.1866	+0.8
64 NaF 26 BeF ₂ 10 UF ₄	0.1983	0.1867	+6.2
66.67 NaF 33.33 UF ₄	0.1252	0.1257	+0.4

ANALYTICAL METHODS

Work was continued on the regular analysis of ternary mixtures by the uranium electro-deposition method. The backlog of mixtures awaiting analysis has been almost eliminated leaving only some previously analyzed binary mixtures to be rechecked. The search for a satisfactory method of fluoride analysis of air samples from fluoride-salt working areas has been abandoned temporarily because of other analytical demands. It is hoped that additional manpower and time will permit resumption of this work as well as other experimental work intended to assist in improving our analytical techniques.

More intensive work has been done on the determination of uranium by fluorescent X-ray spectrometry. Various methods for dissolving uranium fluoride mixtures and for shortening the time of preparation of solutions for fluorescent analysis have been investigated. A satisfactory and fairly rapid procedure has been adopted. Renovation of the XRD-3 fluorescence unit will be required to obtain the expected accuracy of 0.2 percent. This machine is being serviced and several modifications are being made. The modifications include cooling coils for the solution sample box and installation of a krypton proportional counter. The krypton counter will give almost a threefold increase in counting rate.

FILTRATION STUDIES

The use of the filtration technique has been continued to chart the course of the "valley" from the ternary eutectic at 11.9 mole percent UF_4 - 15.8 mole percent BeF_2 - 72.3 mole percent NaF toward the NaF- BeF_2 side of the ternary diagram. Two mixtures with different compositions of UF_4 , BeF_2 , and NaF were prepared. They were given a hydrogen fluoride purification treatment and loaded into separate filtration cans. Samples of each mixture were filtered at several different temperatures and the filtrates were chemically analyzed for uranium and beryllium. The results of these experiments are given in Table V. It should be noted that both starting compositions are on the high NaF side of the "valley". It will be necessary, therefore, to make filtrations using compositions which are on the BeF_2 side of the "valley" to determine whether the same "valley" will be followed.

TABLE V
LIQUIDUS COMPOSITIONS BY FILTRATION

STARTING COMPOSITION MOLE PERCENT			TEMPERATURE OF FILTRATION °C	*FILTRATE COMPOSITION MOLE PERCENT		
UF_4	BeF_2	NaF		UF_4	BeF_2	NaF
5	20	75	520	7.8	20.3	71.9
5	20	75	527	7.4	22.6	70.0
5	20	75	539	6.4	24.7	68.9
2	27	71	540	4.8	26.2	69.0
2	27	71	552	3.2	26.5	70.3
2	27	71	558	2.7	27.0	70.3

*AVERAGE OF TWO DETERMINATIONS

HYDRIDE STABILITY

The dissociation pressure of a technical grade of lithium deuteride was found to be over 2-½ atmospheres at 337°C.

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 installation of equipment in the HH building to process the raffinate residue received from Mallinckrodt Chemical Company has been completed. The deficiencies noted in the previous report have been corrected with the exception of the problem of adequate venting of the Pfaudler kettle in which the hydrochloric acid - sodium chloride solution is heated to boiling. A spray chamber is being constructed for installation in the vent line. It is believed that its use will reduce the hydrochloric acid vapor carried to the scrub tower and thus permit the scrub tower to function normally.

The first drum of the raffinate residue is now being processed by research personnel. Drum No. 20, which contained 288 pounds of material, is being used for the "shakedown" run. At this time the protactinium processing procedure has been carried through the hydrochloric acid digestion step and the "salting out" step. Since solubility tests had shown that the solubility of sodium chloride in the hydrochloric acid solution of the raffinate residue is approximately 30 per cent greater than in a water solution of the same hydrochloric acid concentration, this excess of sodium chloride was added in the "salting out" step. The tests had also indicated that the carrier efficiency was improved by the additional sodium chloride.

EXPERIMENTAL DEVELOPMENT

A balanced three-factor experiment was devoted to the effect of hydrochloric acid concentration in the solution step, the hydrochloric acid concentration of the carrier wash liquid, and the addition of titanium trichloride to increase the carrier efficiency. Titanium trichloride, when added to the solution resulting from the first step of the process, i.e., treatment of the raffinate residue with dilute hydrochloric acid, is oxidized to titanium (IV) by the nitric acid. The titanium (IV) precipitates as titanium phosphate during the "salting out" step as the chloride concentration approaches saturation. A beneficial effect on the carrier efficiency was observed with the addition of 0.015 ml of 20 per cent titanium trichloride solution per gram of filter cake originally dissolved. Increasing the amount of titanium trichloride solution added to 0.1 ml per gram of cake accentuated the beneficial effect but yielded a carrier that was too bulky to handle readily during processing. In this experiment the titanium trichloride solution was added before the addition of the sodium chloride. A second experiment was carried out in which the order was reversed; the acid solution of the filter cake was saturated with sodium chloride before the titanium trichloride solution was added. The same effect, an increase in the carrier efficiency, was noted in both cases. Increasing the acidity of the solution used to dissolve the filter cake from 1 normal to 2 normal increased the 27 kev gamma activity carried but reduced the amount of 300 kev gamma activity carried. Measurements of the 68 kev gamma radiation eliminated a variation in the thorium-230 content as a possible explanation for this difference. It may be that increasing the acid concentration reduced the bulk of the carrier, rather than increased the quantity of protactinium carried. As was expected, increasing the concentration of the hydrochloric acid in the carrier wash reduced the carrier efficiency.

The eighty drums of raffinate residue received from the Mallinckrodt Chemical Company have been properly tagged to preserve the identification of each drum. Each drum has been sampled. Each sample will be analyzed for protactinium content before the corresponding drum will be processed. Thus, in each batch, it will be possible to follow the effectiveness of the separation and concentration of the protactinium in each step of the process.

ANALYSIS OF OTHER SOURCES

Eight samples prepared from so called "airport sludge" were received from Mallinckrodt Chemical Company and analyzed for protactinium. Four different samples of "airport sludge" had been taken by Mallinckrodt personnel. Each of these samples was digested with hot 60 per cent nitric acid, a sufficient quantity of acid being added to obtain a final nitric acid concentration of 0.5 normal. The slurry obtained was filtered. The insoluble residue was washed with 0.5 normal nitric acid and dried at 140°C. The filtrate and wash liquid were combined. Four samples of insoluble residue and a sample of the nitric acid liquor obtained from each of the four original samples were received by Mound.

Samples of the four dried cakes were analyzed spectrographically and found to be similar, qualitatively, to "Sperry A", the first raffinate residue received from Mallinckrodt Chemical Works at the inception of the protactinium program. The major constituents in all cases included magnesium, silicon, and probably iron. Calcium was a major constituent in one sample and a minor constituent in the other three. Titanium was reported in all four samples of cake; as a minor in three cases, and as a trace in the fourth. Other minor and trace constituents were copper, sodium, zirconium, manganese, nickel, cobalt, molybdenum, lead, lithium, barium, and chromium.

Preliminary examination of the four samples of solution showed that there was little, if any, protactinium in any of them. All were fairly rich in thorium-230, as was to be expected. All four of the dried cakes contained appreciable amounts of protactinium, together with traces of thorium-230, thorium-227, and radium-223. A 50 kev gamma peak was found in both solutions and cakes, and has been attributed, tentatively, to lead-210.

Analysis for protactinium has been completed on all eight samples. The counting rates of the protactinium recovered from the nitric acid solutions were so low, however, that a clear cut 27 kev gamma peak was obtained in only one case. The following table summarizes the results obtained:

SOLUTIONS

SAMPLE NUMBER	PROTACTINIUM FOUND (micrograms/liter)	IDENTIFICATION OF PROTACTINIUM	THORIUM-230 FOUND (milligrams/liter)
1B	1.1	PROBABLE	7.5
2B	1.0	PROBABLE	4.2
1T	0.8	QUESTIONABLE	5.5
2T	4.0	CERTAIN	3.1

INSOLUBLE RESIDUES

SAMPLE NUMBER	PROTACTINIUM FOUND (parts per million)	THORIUM-230 PRESENT
1B	0.05	+
2B	0.05	+
1T	0.05	+
2T	0.06	++

RAW MATERIAL BALANCE

SAMPLE NUMBER	PROTACTINIUM IN SOLUTION (%)	PROTACTINIUM IN RESIDUE (%)
1B	5.1	94.9
2B	6.3	93.7
1T	3.8	96.2
2T	31.1	68.9

The methods of analysis used for obtaining the results given above were necessarily somewhat different from those which have been developed in connection with the protactinium recovery program. In dealing with the nitric acid solutions, it was found that the titanium precipitation method could be used if phosphoric acid were added. However, considerable quantities of other inert materials also precipitated and interfered with the solution of the titanium precipitate. Investigation showed that the protactinium could be quantitatively recovered directly from the nitric acid solutions in di-isobutyl carbinol if the solutions were first made either eight normal in nitric acid or six normal in hydrochloric acid. The efficiency of separation was tested by spiking one of the solutions with protactinium and extracting first with tributyl phosphate and then with di-isobutyl carbinol. The di-isobutyl carbinol contained 95 per cent of the protactinium which had been added.

The insoluble residues did not dissolve in sulfuric, hydrochloric, or perchloric acids. After treatment with sulfuric acid, most of the protactinium remained in the residue. It was not possible to use the solvent extraction technique which has been successfully used in treating insoluble residues in the protactinium recovery process. It was found, however, that the protactinium spectrum could be clarified by treating the cakes with a hot saturated solution of tetrasodium ethylenediaminetetraacetate (Versene). Radium and thorium isotopes were solubilized in the Versene, while protactinium and most of the inert constituents remained insoluble.

The quantitative estimates given in the foregoing table are based on the gamma counting rates at 27 kev energy (for protactinium) and 68 kev energy (for thorium-230). Corrections were made for gamma counting efficiency and self-absorption, on the assumption that the materials counted were similar to those found in the protactinium recovery process. Because

of the extremely low protactinium content of the nitric acid solutions, the uncertainty of the values given may be as high as 100 per cent, except in the case of Solution 2T where the uncertainty is not higher than 25 per cent. In the insoluble residues, the probable error is estimated to be no greater than 25 per cent.

The thorium-230 values given are based on tributyl phosphate extraction of the solutions. In one sample, the thorium-230 was stripped from the organic phase into a very dilute phosphoric acid solution. The solution was made ammoniacal, and the resulting precipitate (probably thorium-230 phosphate) was mounted and alpha-counted. The alpha-gamma ratio obtained from this sample was used as a conversion factor in determining the amount of thorium-230 present in the four solutions. Since no thorium-230 estimate was desired, no further attempt was made to obtain a more precise conversion factor.

It may be concluded from the results reported that: (1), little or no protactinium is present in the solutions resulting from contacting the "airport sludge" with nitric acid; (2), protactinium, when present, is not extracted from these solutions into tributyl phosphate; and (3), little or no consideration need be given to establishing a protactinium material balance for this material.

