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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.

Eight polonium-beryllium neutron sources, two alpha sources and eleven plutonium-beryllium neutron sources were shipped in June. Five polonium-beryllium neutron sources, one alpha source, and five plutonium-beryllium neutron sources were for other AEC sites.

Twenty-nine tanks of influent water were processed during this period. Nine of the tanks required no pretreatment. Eighteen of the tanks required one treatment of amorphous carbon and calcium chloride. One tank was treated twice before its final filter count was low enough to be passed through the system. Work continues on the treatment of sludge with caustic to prevent gas formation in the drums which are transported to Oak Ridge for burial.

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

TABLE I

Discharge Volume	747,600 gallons
Total Alpha Activity	4.8 millicuries
Total Beta Activity	22.9 millicuries
Activity Density	
Alpha	1.9 cts/min/ml
Beta	9.0 cts/min/ml

NUCLEAR RESEARCH AND DEVELOPMENT

GENERAL WEAPONS DEVELOPMENT

Process development work is being conducted in connection with the fabrication and chemical processes for plastics, high explosives, gases, metals, alloys, and other material for weapons components and the fabrication and recovery associated therewith.

Plastics Project

The asbestos-filled diallyl phthalate plastic used in molding operations at Mound Laboratory must meet specifications more rigid than are common in industry. Development work has been directed at the determination of the effects of process variables and toward achievement of better control of these variables in order that plastic which meets specifications may be consistently produced. In addition, efforts will be made to find plastics and fillers which would provide improved properties.

Plastics Formulation - Six full-size runs were made in the sigma-blade mixer. These formulations are summarized in Table I.

TABLE I

Diallyl Phthalate Formulations-Dapon Plus Filler 25 Pounds

Run No.	Parts Per 100 Parts Dapon 35						Weight of Change Minus Solvent lb	Residual Volatiles %	Peak Power KW
	Filler a/b	Solvent c/d	Monomer	Catalyst e	Pigment f/g	Stearate h/i			
B46	40 60	275 -	4	2	2.5 0.5	1.5	26.3	12.3	1.59
B47	40 60	200 35	5	3	2.5 0.5	1.5	26.6	11.0	1.50
B48	40 60	200 35	5	3	2.5 0.5	2.0	26.6	10.5	1.58
B49	40 ⁱ 60	200 35	5	3	2.5 0.5	2.0	26.6	14.6	1.15
B50	- 100	100 -	5	3	2.5	2.0	26.5	6.0	1.75
B51	40 60	200 35	5	3	2.5 5.0	2.0	26.6	12.6	1.60

a. Cellulo 200 (asbestos)
b. Hydrate-PD-121 (kaolinite)
c. Acetone

d. Benzene
e. Tert-butyl perbenzoate
f. Scarlet 25AD IR 6440

g. Rutile TiO₂
h. Zinc Stearate
i. Calcium Stearate
j. Cellulo 100

Runs B46, B47, and B48 were made in one week, but the maintenance requirements of the sigma-blade mixer make it doubtful if more than five runs can be made per two week period on a continuing basis.

In both runs B47 and B48, 35 parts of benzene was added at about the midpoint of solvent removal. Mixing was quite good throughout both runs.

Run B49 was identical to run B48 except that Cellulo-100 was used in place of Cellulo-200. Mixing was not as good with the longer-fiber Cellulo-100 even though the total mixing time was 33 hours. There are definite processing advantages to using the shorter-fiber Cellulo-200.

Run B50 was made without asbestos, using 100 parts of kaolinite as the mineral filler. Excellent mixing was experienced throughout this run. The power input increased rapidly near the end of the run, the major rise taking place in less than an hour. Although the residual volatile content of the product was only six per cent, the product was quite soft. It had a greater tendency to cold-flow than did formulations containing Cellulo having twice this residual volatile content.

In run B51, prewetting of the dry ingredients was tried. A dilute solution of prepolymer (10 per cent of the varnish diluted with 70 parts of acetone) was filtered onto the kaolinite and asbestos in the sigma-blade mixer. This was mixed for one-half hour before the remaining varnish was added through the filter. The stearate, pigment, and TiO_2 were then added as a slurry in acetone and the batch was processed in the usual manner. It is felt that there was some slight improvement in the mixing in this run, and a further study of the effect of pre-wetting will be made.

Formulations B45, B46, B47, B48, B49, and three lots of B50 have been milled on the differential rolls.

Five lots of B45 were milled to final net input powers slightly over the minimum net input powers which were obtained during each run. The net input powers at cut-off were 1.68, 1.30, 1.20, and 1.21 KW. Six lots of B46 were milled in the same manner, and the net power at cut-off varied between 1.34 and 1.49 KW.

It is believed that some polymerization occurred in the sigma-blade mixer during the solvent removal steps, probably during periods when the mixer was stopped. Under these conditions the temperature in the center of the mass could rise well above the surface temperature, probably sufficiently high to give a reasonably rapid rate of polymerization. The wide variation of net input powers obtained in milling B45 could be explained by the fact that different mill lots contained varying proportions of material from the center of the mass.

Care was taken to avoid stopping the sigma-blade mixer for more than a few minutes during runs B47 and B48. All twelve of these mill lots were milled to approximately 1.1 KW final net input power and milling times were considerably longer than for B45 and B46. Apparently very little polymerization took place during solvent removal in runs B47 and B48.

B49 was formulated with Cellulo-100 in place of Cellulo-200. It did not handle on the mill rolls as well as did B47 and B48 and mill times were shorter. Some evidence of partially wetted asbestos could be seen in the milled sheets. Formulations using Cellulo-200 have definite processing advantages over formulations using Cellulo-100, and also give a more uniform product.

B50 was formulated with Hydrite-PD-121 as the only mineral filler. It was very plastic and milled without the slightest difficulty. It transferred almost immediately to the hot roll. The ease with which

B50 processed through all stages indicates definitely that our processing difficulties are caused primarily by the asbestos and are increased by the long-fiber content of the Cellulo-200.

Work has continued on the effect of residual solvent and the problem of its removal.

The loss in weight of several DAP resins when dried under several conditions are given in Table II, while the effectiveness of several combinations of time and temperature when an air circulating oven is used as shown in Table III.

TABLE II

Volatile Content of DAP Resins

Sample No.	Weight Loss on Drying, Per Cent									
	24 hr at		Vacuum Oven				96 hr at		Circulating Oven	
	25°C	70°C	48 hr at	65 hr at	25°C	70°C	25°C	70°C	24 hr at	65 hr at
		25°C	70°C	25°C	70°C	25°C	70°C	25°C	25°C	25°C
B44-6	-	-	1.93	3.90	-	-	-	-	-	-
B45-1	1.64	3.81	-	-	2.09	4.10	-	-	1.59	2.60
B48-1/6C	0.31	1.90	0.33	2.03	-	-	0.41	-	-	-
Mesa B-1143	-	-	0.36	0.76	-	-	-	-	-	-
Mesa B-1433	-	-	0.05	0.58	-	-	-	-	-	-
Mesa B-1543	-	-	0.44	0.98	-	-	-	-	-	-

TABLE III

Effect of Drying DAP Resins in Air Circulating Oven

Sample No.	Condition of Drying Time hr	Drying Temp. °C	Flow Time at 5 Tons Sec	Weight of Test Cup g	Residual Volatiles, % Vacuum Oven for 48 hr at 25°C at 70°C	
B48-1/6C	72	25	6.68	39.04	0.28	1.54
B47-1/6CD1	72	38	9.99	39.58	0.25	1.26
B47-1/6CD2	32	45	10.73	39.80	0.017	0.81
B47-1/6CD4	21	55	11.15	39.70	0.015	0.85
B47-1/6CD3	23	60	13.78	39.66	0.06	0.37
B47-1/6CR ^a	-	-	9.15	39.58	-	-

a. Dried 72 hours on laboratory bench top; all other B47 samples were aliquots of B47-1/6CR

It is believed that some polymerization took place during 23 hours heating at 60°C, but that little if any polymerization took place during 21 hours at 55°C. Since a drying cycle of less than 24 hours is definitely desirable for production, it is probable that a temperature above 45°C but below 60°C can be used. This work will be continued with large quantities of DAP molding powder.

It appears after drying has proceeded for a few hours that the solvent concentration at the surface is reduced to a low value, so that the drying rate becomes limited by the rate of diffusion of the solvent from the interior to the surface of the particle. Thus small particle size is desirable.

The rate of polymerization is probably rather slow even at 60°C but may be sufficiently high to produce a thermal gradient between the center and the surface of the larger particles. B47-1/6CD3 was a 200 gram sample, and hence, any nonuniformity in the sampling of this material would probably be in a nonuniform distribution of particle size. Four flow time measurements were made of B47-1/6CD3; these gave 13.15, 14.39, 13.12, and 14.49 seconds. If it is assumed that the larger particles polymerized more than the smaller particles and that two 14.4 second samples had a greater percentage of larger particles than the 13.1 samples, the flow time data can be explained. The 13.1 second cups weighed 39.40 grams while the 14.4 second cups weighed 39.88 and 39.89 grams. This weight difference can be explained if it is assumed that the internal temperature of the larger particles was significantly higher than that of the smaller particles.

It would therefore appear to be important that our product contain no large particles, and it is probably desirable to minimize the number of particles larger than say 16 or perhaps 20 mesh. Since it is also desirable for our product to contain no very fine material, a special grinding technique should be developed. Some of the data obtained to date on the use of various grinder screen sizes or combination of screen sizes are given in Table IV.

It will be seen that stiff material gives a higher percentage of fines than does soft material. Fortunately it is probable that material milled to net input powers between 1.10 and 1.20 KW will have proper flow for production molding. Also, double grinding or the use of finer screens tend to give a smaller average particle size in the -10 + 16 mesh range, so the benefit is actually greater than that indicated in Table IV.

It has been shown that relatively small amounts of residual solvent have a definite effect on the cup flow test flow time, and that the weight of the test cut is an indication of the relative solvent content of the material. Unfortunately, it is not possible to accurately remove exactly the same amount of material from the test flow cups when the flash is removed; hence, cut weights cannot be used as a quantitative measure of the residual solvent content.

Table V shows the variation in flow test cup weight with particle size for three materials, and the variation of cup weight with time for two.

The data for B42-1/5S are particularly interesting. The <16 >20 mesh fraction gave cups of lower weight than did the >16 mesh fraction, while the weights of the <20 >40 fraction cups were essentially the same as those from the >16 fraction before the fractions were dried. After drying, the cup weights increased in the expected order. The anomaly was hence due to residual solvent in the material.

It now seems definite that part of the variation of flow time with particle size is due to variation in residual solvent content.

The solvent effect is greatest with materials milled to low input powers since they contain a larger amount of residual solvent.

TABLE IV
Effect of Grinding Technique on Particle Size Distribution

First Grinding Second Grinding ^g	Mesh Size, in.						
	1/8	1/8	3/32	3/32	3/32	1/16	1/16
Sample Number Mesh Range	B42-1/5S Wt. % ^a	B42-1/5S Wt. % ^a	B46-1/6S Wt. % ^b	B47-1/6 Wt. % ^c	B48-1/6 Wt. % ^c	B50-1 Wt. % ^d	B50-2 Wt. % ^e
<10 >100	95.32 ^f	92.80	92.12	95.60	95.41	92.25	86.47
<100	4.68	7.20	7.82	4.40	4.59	7.75	13.53
> 10	5.48	0.00	0.00	0.02	0.02	0.00	0.00
<10 >> 16	41.35	7.68	5.97	-	-	1.90	3.07
<16 >> 20	18.15	31.45	31.09	-	-	27.15	-
<20 >> 40	18.72	36.03	37.67	-	-	39.62	-
<40 >> 60	7.49	11.45	10.05	-	-	12.79	-
<60 >> 80	2.63	4.00	5.04	-	-	7.67	-
<80 >>100	1.50	2.19	2.30	-	-	3.12	-
<100>200	2.34	3.47	3.85	2.65	2.65	4.92	7.12
<200>325	1.17	1.80	1.61	0.72	0.77	2.12	3.85
<325	1.17	1.93	2.47	1.00	1.10	0.71	2.56

a B42-1/5 milled to 2.05 KW net input power

b B46-1/6 milled to 1.40 KW net input power

c B47-1/6 and B48-1/6 milled to 1.10 KW net input power

d B50-1 milled to 1.02 KW net input power

e B50-2 milled to 2.20 KW net input power

f Contains 0.72% of >8 and 4.76% of <8 >10 mesh

g Material greater than 20 mesh reground.

TABLE V
Variation in Weight of Flow Test Cups with Particle Size and with Age

Mesh Range	Weight of Flow Test Cups in Grams					
	B35-2/5S at 864 hr	B41-1/6S at 73 hr	B47-1/6S at 840 hr	B42-1/5S at 120 hr	B42-1/5S at 696 hr	B42-1/5S at 1152 hr ^a
>16	38.50	39.2	-	39.53	39.85	39.86
< 16 > 20	38.67	39.46	39.89	39.45	39.81	39.91
< 20 > 40	38.80	39.53	40.02	39.54	39.88	39.97
< 40 > 60	39.4	39.77	40.04	39.87	40.00	40.04
< 60 > 80	39.5	39.9	40.16	40.053	40.09	40.07
< 80 > 100	39.7	40.0	-	-	40.10	40.11
< 100 > 120	39.8	-	-	-	-	-
< 100 > 140	-	-	-	-	40.15	40.17
< 120 > 140	39.9	40.2	-	-	-	-
< 140 > 200	-	-	-	40.20	40.19	-
< 200 > 325	-	-	-	-	40.23	40.24
< 200	40.1	40.3	40.47	-	-	-
< 325	-	-	-	40.24	40.28	-

a Air dried on laboratory bench for 18 days at room temperature

Probably of more importance is the fact that higher residual solvent content results in flow test cups of lighter weight. The design of the mold is such that the volume of plastic in the mold is constant if the mold closes completely. Thus, differences in weights of flow test cups indicate difference in the density of the cured plastic. With the same material, a lower density is possible only if the molded piece is porous. Residual solvent must therefore give porous moldings, with the porosity increasing as the solvent content is increased.

Previous work showed that samples milled to the same net power input gave products having flow times which did not diverge greatly from the average. There were variations, however, which were difficult to explain. It now appears probable that many of these variations were due to variations in the residual solvent content. Table VI lists the flow times and cup weights for 12 mill lots (six from each of two sigma-blade mixer runs) all milled to essentially the same net input power. It can be seen that, in general, lower flows are associated with lower cup weights.

TABLE VI

Comparison of Flow Times with Flow Test Cup Weights for
Samples Milled to the same Net Input Power

B48 AND B47 AT 74 HR AFTER GRINDING

Sample No. ^a	Net Input Power KW	Flow Times at 5 tons Sec	Flow Cup Weight g	Sample No. ^b	Net Input Power KW	Flow Time at 5 tons Sec	Flow Cup Weight g
B47-1C	1.10	8.14	39.30	B48-5	1.09	6.49	38.98
B47-2C	1.10	8.30	39.37	B48-2	1.09	6.54	39.00
B47-4C	1.10	8.45	39.37	B48-6	1.09	6.68	38.99
B47-6C	1.09	8.63	39.37	B48-1	1.09	6.84	39.01
B47-3C	1.10	8.83	39.43	B48-4	1.10	6.85	39.00
B47-5C	1.13	9.00	39.42	B48-3	1.09	6.99	39.02
B47-1/6C	1.10	9.15	39.58	B48-1/6C	1.10	6.68	39.04
B47-1/6SI ^c	1.10	11.80 ^d	39.85	B48-1/6SI ^c	1.10	11.23	39.55
B47-1/6SII	1.10	12.98 ^d	40.00	B48-1/6SII	1.10	10.90	39.70
B47-1/6SIII	1.10+	13.08 ^d	40.28	B48-1/6SIII	1.10	11.85	40.03

a B47 dried in air circulating oven at room temperature

b B48 dried on laboratory bench at room temperature

c Mesh range of fractions: C, <10>100; -SI, <100>200; -SII, <200>325; -SIII, <325.

d Flow times and weights after 246 hours, samples left spread on trays on laboratory bench.

The flow data for the "S" fractions indicate that the stabilized flow times of B48 are not radically different than the stabilized flow times of B47. The large difference in flow time between the B47C and B48C mill lots are therefore due to the higher residual solvent content of the B48 materials.

The hardness of a DAP molding is a function of the degree of cure and of the porosity of the molding. For a given material the hardness increases rapidly with mold closure time, but it soon becomes

essentially constant as the mold closure time is increased. The use of hardness as a method of determining cure therefore does not appear to be promising. Additional data on the variation of hardness with mold closure time are given in Table VII, which also gives a rough comparison of the Rockwell M scale and the Rockwell H scale when used for our materials.

TABLE VII

**VARIATION OF HARDNESS WITH MOLD CLOSURE TIME:
COMPARISON OF ROCKWELL M AND H SCALES**

Material, B45-3/4

Mold Closure Time Min	Flow Time at 5 Tons Sec	Hardness Rockwell M Scale	Flow Time at 5 Tons Sec	Hardness Rockwell H Scale
0.5	7.58	92	9.18	82
1.0	8.30	94	8.66	89
1.5	8.63	101	8.62	90
2.0	8.41	103	7.73	94
2.5	8.38	105	8.18	94
3.0	7.57	106	8.15	96
3.5	8.12	106	7.43	95
4.0	8.51	107	7.85	95
5.0	8.59	108	7.85	96
6.0	8.63	106	7.50	97
8.0	8.21	104	-	-
10.0	8.71	107	-	-
12.0	8.04	106	-	-
15.0	8.11	106	-	-
20.0	8.56	106	-	-

Filters have been installed in the air inlet to the Formulation Room (E-119), and in the air inlet to the steam-heated circulating air oven. The Engineering Division has been requested to design a new system for driving the sigma-blade mixer. This will include a slip clutch in the motor drive, out-board antifriction bearings, and materially improved shaft seals. New shafts, precision ground to fit the sigma-blades, will be installed as soon as the front sigma blade on order arrives. These shafts will be made of high speed drill rod but will not be heat treated. It is believed that the high tensile strength and low elongation of this material will eliminate the twisting encountered with the soft stainless steel shafts currently used.

Evaluation - An evaluation of the physical properties of B44 has been begun but will not be reported in detail until next month. B44 contains 30 parts of Cellulo-200 and 70 parts of Hydrate-PD-121. Preliminary indications are that B44 will have reasonably good physical properties, but that they will probably be inferior to those of formulations containing 40 parts of Cellulo-200.

Service - Mesa Batch No. B-1433 gave a significantly lower percentage of rejected heads in Production Molding for reasons other than inclusions than did Mesa Batch No. B-1143. A comparative study of these two resins was requested by Production Molding. The weight losses on chloroform extraction, and on heating in a vacuum oven (both at room temperature and at 70°C) were determined. The chloroform extracts were evaporated to dryness and examined. The extract from B-1433 appeared to be less soluble in chloroform and to contain a much larger proportion of material which appeared to be white and opaque, and less of material having a glassy appearance. The weight loss data are summarized in Table VIII.

TABLE VIII

Comparison of Mesa DAP Batches B1143 and B1433

Mesa Batch Number	Weight loss on CCl ₄ Extraction Per Cent	Weight loss in Vacuum Oven at		Flow Time at 5 Tons Sec
		23°C	70°C	
B-1143	39.6	0.36	0.67	10.88
B-1433	32.9	0.05	0.58	15.16

No differences were found which could not be explained by a difference in the degree of cross-linking as indicated by the flow times. No good explanation for the difference in rejection rates between these two batches was found.

Complete flow curves were run on Mesa Batches B-1433 and B-1543. Both of these had flow times definitely above 14 seconds at 5 tons. However, they have given reasonably low rejection rates in production molding and are being used.

Materials on which dry blending tests had been run were received from the J. H. Day Company and from Patterson-Kelly Company, Inc. These samples have been evaluated.

Visual observations and flow time measurements indicate that adequate mixing was obtained with a ribbon blender in less than one hour. A mixture of Acme Natural 3S, Batch 1141 (flow, 7.57 sec at 5 tons) and Acme Blue 2S, Batch 1076 (flow, 16.13 sec at 5 tons), and Acme Blue 2S, Batch 1076 (flow, 16.13 sec at 5 tons) was used for this test. The blending operation was very dusty.

Test on the blending of Hydrite PD-121, zinc stearate, and pigment were made in a ribbon blender, and in a twin-shell blender with intensifier bar. Visual observation and spectrographic analysis for zinc were used to measure the homogeneity of the blends.

The ribbon blender gave a reasonably good dispersion of the zinc stearate and the pigment in the bulk of the Hydrite, but the Hydrite formed small balls which rode on top of the mixture and were not broken up after one-half hour. Mixing in the ribbon blender was therefore not satisfactory. This was also a dusty operation.

Mixing with the twin-shell Blender with intensifier bar appeared to be excellent.

If a method can be developed for defelting the Cellulo asbestos before charging to the sigma-blade mixer, all ingredients can be blended during this process, and dry blending will not be required. Materials

for making a sigma-blade mixer charge (except for monomer and pigment) have been shipped to the Patterson-Kelley Company for wet blending tests in a twin-shell blender with liquid-addition, intensifier bar. It is felt that the combination of slow speed mixing and high speed stirring may give the desired result. This test has been delayed by a vacation at the Patterson-Kelley plant.

Correspondence and conversation with Mr. W. C. Sturgeon of the Patterson Foundry and Machine Company resulted in a recommendation of their type GPM mixer for our liquid, defelting blending. Since the GPM mixer is a massive, slow-speed mixer which might offer little, if any, advantage over a sigma-blade mixer, no further action has been taken pending the tests to be made at Patterson-Kelley.

Several conferences have been held to outline the production process and equipment as firmly as possible at the present time. The biggest question still remaining is the equipment for the pre-blending step.

Helium-3 Recovery Project

Mound Laboratory has been given the responsibility of recovering helium-3 from Savannah River's gaseous wastes. A process involving both chemical and isotopic purification is being developed.

Gas Purification - Outgassing of the gas purification line was continued. For evacuation, the system divides into two parts. Connected to one of the diffusion pumps are two feed gas tanks, a feed manifold, and two copper furnaces. This unit can be pumped down to a pressure of about three microns of mercury. When the pump is cut off, the pressure initially rises at the rate of about one micron of mercury per minute till it reaches six microns, at which point it levels off to no perceptible rise for several more minutes. The remainder of the system, connected to a second diffusion pump, includes a compressor, filters, a liquid air cold trap, a product manifold, and two product collection tanks. This section can be pumped down to a pressure of about 11 microns of mercury. When the pump is cut off, the pressure rises at the rate of about five microns per minute till it reaches 35 microns, at which pressure it levels off to no perceptible rise for the next several minutes.

Loading and Unloading Facility - The trailer unloading system for Savannah River Plant by-product gas has been installed in R-105. The system was tested under helium pressure with the helium leak-detector and was found to be vacuum-tight.

Permanent connections to the manifold for a Pirani vacuum gauge and two gas sampling points are being made.

Loading of the by-product gas into trailers at Savannah River Plant is expected to begin about September 1, 1957. It is estimated that the helium-3 content of the gas will be about three per cent.

Tritium Recovery Project

A process is to be developed for the unloading of tritium reservoirs, chemical and isotopic purification of the tritium, and reloading of the reservoirs. The process is to include the capability of loading and unloading of special high pressure loadings. The initial process development will include the recovery of tritium from Los Alamos Scientific Laboratory's decayed salt, aqueous and gaseous materials.

Some design criteria have been provided the Engineering Division in connection with the hot gas facility. Arrangements are being made with Los Alamos Scientific Laboratory to procure from them various tritium containing materials from which the tritium must be recovered. Estimates have been made as to the relative merits of low temperature distillation and gaseous thermal diffusion in the preparation of pure tritium from these wastes. Several sources of supply of liquid hydrogen have been investigated as to cost and time for procurement. Savannah River Plant was visited to discuss the progress on the bottling of reservoirs. The Cryogenic Engineering Laboratory at National Bureau of Standards, Boulder, Colorado, was visited to discuss their potential contributions to low temperature distillation studies.

Isotope Separation

Mound Laboratory is responsible for the production of several isotopes needed in research projects at several Division of Military Applications sites. Particular emphasis is placed on the investigation and use of the thermal diffusion process for separating gaseous isotopes. Processes and facilities to handle both inert and radioactive gases are being developed.

Gaseous Thermal Diffusion - Work is being continued on the continuous-flow separation of neon isotopes in the four twelve-foot columns connected in series cascade. Using normal neon as a feed gas, 20 milliliter samples containing as high as 91 per cent neon-22 have been obtained from the bottom of the cascade. This concentration was attained after 90 hours of alternate flow and steady state operation. There was a total of eight liters STP gas throughout during the flow operation. During this time normal neon was introduced into a port six feet below the top of the cascade and depleted neon containing about 1.5 per cent neon-22 was pumped from the top of the cascade. The cascade was operated at a pressure of 345 millimeters of mercury, with center wire temperatures of approximately 900°C.

One of the major problems that has been encountered in this separation process is the accumulation of these gases is believed to be concentration of air contamination of the feed gas by the columns rather than air leaks into the system since all detectable leaks have been eliminated. Removal of the impurities from enriched neon has been effected by very slow passage of the gas through two activated charcoal beds held at liquid nitrogen temperatures and through a magnesium bed held at 625°C. Typical analyses of three samples of gas purified by this treatment are as follows:

<u>Sample</u>	<u>Treatment</u>	<u>% N₂, O₂ and A</u>
1	None	75.03
2	Charcoal Bed	0.9
3	Charcoal Bed and Magnesium Bed	0.11

At present two large samples of enriched neon 22 have been collected. Their analyses are as follows: No. 1, 400 milliliters containing 70 per cent neon-22 and less than 0.2 per cent nitrogen, oxygen, and argon; No. 2, 300 milliliters containing 76 per cent neon-22 and less than 0.15 per cent nitrogen, oxygen, and argon. These samples were removed from the bottom of the cascade with a glass Toepler pump and are stored in 500 milliliter pyrex vessels.

Design and development work is being continued on the gas handling pumps to develop techniques to remove gas from the columns and store it at elevated pressures with no appreciable gas loss or introduction of contaminants.

A total of 50 neon gas samples were analyzed on the mass spectrometer this month. This work was done in conjunction with the above experimentation. In addition, two standard neon samples were prepared one containing tank neon and the other containing approximately three per cent dried air. These samples were run and compared on the mass spectrometer. The results obtained were used to calibrate the instrument for analysis of air contamination of neon samples.

Liquid Thermal Diffusion - A 48-hour and a 96-hour run have been completed, employing a 50-50 volume per cent mixture of D_2O and H_2O as feed. In both runs, a nominal hot wall temperature of $35^\circ C$ and a nominal temperature difference of $17^\circ C$ prevailed. The fractions from both runs gave evidence of corrosion. Most of the fractions had a yellowish color, and a reddish-brown precipitate formed on standing. In addition, the feed fraction from the 96-hour run was contaminated with graphite from a port plug employed in a repair operation.

The densities of the supernates from the fractions from the 48-hour run were determined with a one-milliliter Lipkin pycnometer. No consistent evidence of separation was obtained. Discrepancies may be attributed to varying degrees of corrosion. It is planned to centrifuge the fractions from the 96-hour run before determining the densities of the supernates.

Test runs with reagent-grade tributyl phosphate have indicated satisfactory physical and chemical characteristics for column operation. There was no visible evidence of corrosion or gas accumulation, and the drainage rate seemed satisfactory. The maximum temperature difference attainable was similar to that attained with the benzene-carbon tetrachloride system, namely, 45 to $50^\circ C$.

Approximately 0.5-molar solutions of cerous nitrate and yttrium nitrate in tributyl phosphate have been prepared. The crystalline hexahydrates were agitated with the organic solvent, and the emulsified water of hydration was removed by centrifuging. After the exact compositions of the two solutions are determined, they will be mixed in the proper proportion to give a feed solution equimolar in the two salts.

A voltmeter has now been permanently installed on the instrument panel. A double-throw switch makes it possible to measure the voltage drop through either column heater.

~~SECRET~~**Materials of Weapons Interest**

Process development for the production of small amounts of unusual materials is occasionally required to meet the needs of the weapons program.

Ionium Project

The extraction of Ionium from partially-processed raffinates from the Mallinckrodt Uranium Refinery is being completed to fulfill previous commitments.

Experiments were conducted to increase the distribution coefficient of thorium in the dummy sulfated feed solution. The results of this work are shown in Table I.

TABLE I**Distribution Coefficient of Thorium in Sulfated Solution**

From Nitric Acid	Initial Aqueous Nitrate Ion Normality		Distribution Coefficient $C_{org.}/C_{aq.}$
	From Aluminum Nitrate	Total	
4	0	4	0.58
2	2	4	1.70
1	3	4	5.86

On the basis of the results listed above, 25 gallons of the "hot" sulfated feed from Mallinckrodt Chemical Works were added to 25 gallons of two-molar aluminum nitrate solution to give a solution which was approximately one-normal in nitric acid and three-normal in aluminum nitrate. Although much mechanical difficulty was encountered with the mixer-settler units, including clogged aqueous ports, clogged organic ports, gear wear which caused a change in torque on motors and a decrease in rpm on the stirrers, and their blockage and leaks, the extraction process gave good results. These results are shown in Table II.

TABLE II**Extraction Unit Results**

Sample No.	Organic Product		Aqueous Raffinate	
	Th g/liter	U g/liter	Th g/liter	U g/liter
A	2.75	4.41	0.08	0.15
B	4.30	7.61	0.09	0.09
C	3.35	4.67	0.08	0.11
D	3.55	5.10	0.10	0.14

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The partition unit has been requiring more strip solution than anticipated, and the partition flow-sheet is still being developed. However, most of the difficulty has been mechanical.

In an effort to decrease the time required for this final ionium production, solid aluminum nitrate rather than the two-molar solution will be added to the next feed batch. This will reduce the total volume of the feed solution by over 50 per cent.

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Protactinium Project

The production for Albuquerque Operations of a gram of protactinium-231 from unprocessed Sperry-Filter-Press residues from Mallinckrodt is being completed.

Production - The raffinates from the first extraction have all been recycled through the units. It is not yet known how much of the protactinium was recovered.

All of the protactinium should be in one homogeneous batch by July 22. This requires about four batch precipitations of the protactinium on a ferric phosphate carrier and dissolution of the resulting slurries and precipitation on a titanium phosphate carrier. The titanium phosphate carrier will be dissolved and the protactinium will be concentrated and purified by solvent extraction.

Purification - The protactinium concentrate recovered from operations during FY 1956 is being homogenized and consolidated before further purification. It was estimated in last month's report that purification work could be begun by July 15, and that the protactinium would be purified and in aqueous solution by September 1. However, because unexpected difficulties were encountered in current HH-Building process work, it was necessary to suspend the consolidation and purification work this month in favor of process development.

Process Development - In last month's report a procedure was described for concentrating the protactinium recovered in two-molar phosphoric acid on a relatively small ferric phosphate precipitate. Because of the diversity of the batches of raw material, the conditions developed for precipitating protactinium on ferric phosphate were found to be inapplicable to the next two batches of product, and it was necessary to develop new conditions for accomplishing this purpose.

The first sample of product received for development had an indefinite history. It consisted of phosphoric acid strip solution from first-cycle processing of a variety of raw materials including some raffinate residue, some recycled raffinates, and some pre-processed solids from FY 1956 operations (sodium chloride process). The phosphoric acid strip solution was treated with the amounts of water and 50 per cent sodium hydroxide called for in the previous ferric phosphate precipitation method, but poor recovery of protactinium resulted from the use of these conditions. Additional sodium hydroxide was added until a heavy precipitate formed which carried all of the protactinium, but which also appeared to have carried nearly all of the iron. The precipitate was too bulky for storage, and a concentration step was required.

Meanwhile, a second batch of phosphoric acid strip solution with an equally indefinite history was obtained. However, in this case, no sodium hydroxide was added prior to receipt of the sample for development. Rather than develop separate processes for the two batches of phosphate product, it was found convenient to mix the batches to provide a homogeneous slurry. Nearly all of the precipitate from the hydroxide-treated batch went into solution when it was mixed with the second batch. A sample of this composite was then taken for development of a ferric phosphate precipitation method.

It was found that the efficiency of the protactinium-iron separation was dependent not only on the amounts of sodium hydroxide and water added but on the heating and stirring times. The best procedure consistent with the available equipment was found to be the following:

To 25 gallons of phosphate product, add 2.31 gallons of 50 per cent sodium hydroxide and ten gallons of water. Stir the mixture for five minutes without heating. Continue stirring, heat to 200°F, and continue stirring at that temperature for 30 minutes. Cut off the heat, and allow the mixture to cool to below 85°F without stirring. Siphon off and discard the clear supernatant liquor.

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When this procedure was performed on the large batches in the HH-Building, samples of the supernatant liquor were checked for protactinium. They had no count at 300 kev. Hydroxide precipitation tests indicated that approximately 65 per cent of the precipitable solids were removed with the decanted supernatant liquor.

One more batch of phosphoric acid product remains to be homogenized and to be tested for optimum ferric phosphate precipitation conditions. It is anticipated that when this precipitation has been completed and the supernate discarded, all the ferric phosphate precipitates can be transferred as water slurries to a single Pfaudler kettle for homogenization. A titanium precipitation procedure will be developed to concentrate the protactinium further and to remove nearly all of the iron now associated with the protactinium.

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~~CONFIDENTIAL~~**REACTOR DEVELOPMENT PROGRAM****CIVILIAN POWER REACTORS****HOMOGENEOUS REACTOR**

The Civilian Power Reactor Program is concerned with the development of reactors suitable for the production of power or heat for civilian use. Homogeneous reactors are one of the types being investigated. In a homogeneous reactor, the fuel is by definition evenly dispersed in the moderator, so that solutions of uranium-233, uranium-235, or plutonium-239 are acceptable fuels if their chemical and physical properties permit low capital and operating costs.

Plutonium Solubility Studies

A project has been initiated to investigate aqueous systems containing plutonium as a fuel for a homogeneous reactor. Initially, the solubility of various plutonium salts in aqueous solution up to 300°C will be measured. The maximum concentration of plutonium will be ten grams per liter. A second solubility study will be made on an aqueous system containing up to 300 grams of uranium per liter and five grams of plutonium per liter.

Five series of runs were made on the solubility of the system plutonium (III) sulfate-sulfuric acid-water. Results are listed in Table I. A summary of all results for the plutonium (III) sulfate system is given in Figure 1. Experimental difficulty was encountered in the range of acid concentrations from 0.6-normal to 1.01-normal. Experiments were repeated in this concentration range but the situation was not clarified.

The proposed procedure (CF-57-6-31, "Mound Laboratory Technical Activities Report Through June 15, 1957") for the preparation of plutonium (III) carbonates must be revised because plutonium (III) hydroxide is too strong a reducing agent to be stable in water. Other procedures are being investigated for preparation of plutonium (III) carbonate.

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TABLE I

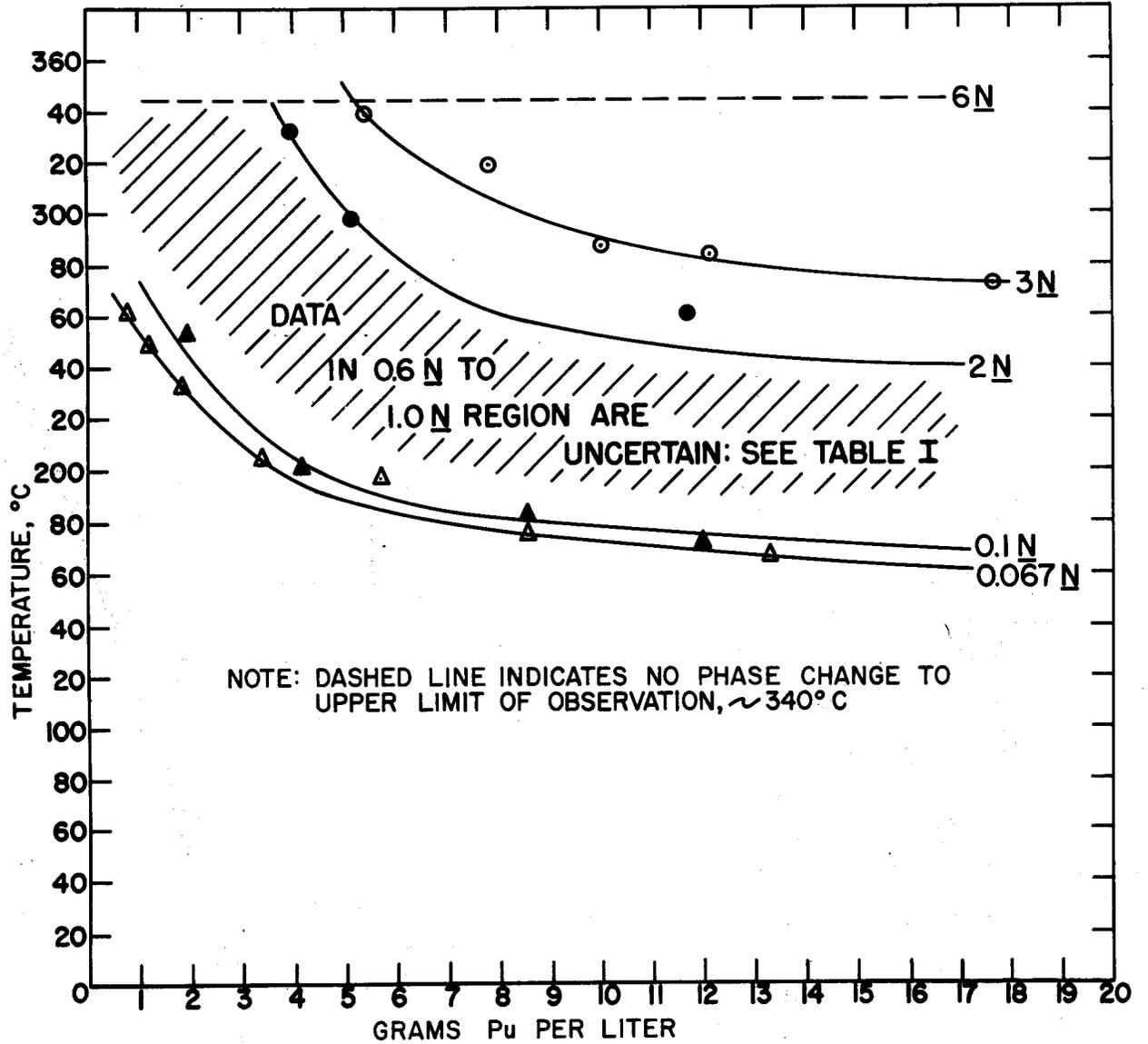
Plutonium (II) Sulfate Solubility in Sulfuric Acid-Water System

0.62 N H ₂ SO ₄		1.0 N H ₂ SO ₄		2.0 N H ₂ SO ₄		3.0 N H ₂ SO ₄		6.0 N H ₂ SO ₄	
Temp. of Phase Change °C	Pu Conc. g/liter	Temp. of Phase Change °C	Pu Conc. g/liter	Temp. of Phase Change °C	Pu Conc. g/liter	Temp. of Phase Change °C	Pu Conc. g/liter	Temp. of Phase Change °C	Pu Conc. g/liter
218.0	12.62	212.4	21.63	260.5	11.74	272.8	17.66	>342.5	14.98
226.2	8.02	211.5	19.41	254.3	6.41	284.0	12.15	>340.5	10.70
231.2	5.65	204.6	12.02	299.5	5.15	287.8	10.04	>341.5	6.43
256.0	3.22	212.4	9.02	333.0	3.90	319.5	7.81	>340.8	4.28
276.6	1.80	245.2	6.66	>350.5	1.98	339.7	5.41	>341.7	2.14
293.8	1.52	229.0	6.59	>349.7	1.52	>340.5	5.20	>343.2	1.28
299.9	1.17	267.6	6.00	>348.2	0.67	>342.7	4.60	>342.7	0.86
		234.4	5.16			>341.3	3.14		
		252.8	3.69			>346.0	2.84		
		256.4	3.20			>344.3	2.39		
		328.0	2.53			>338.8	2.16		
		349.3	1.51			>340.5	1.61		
		308.4	1.32			>341.3	1.54		
		309.3	1.11						

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SOLUBILITY OF PLUTONIUM(III) SULFATE IN THE SULFURIC ACID-WATER SYSTEM

FIGURE 1

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~~SECRET~~**AIRCRAFT PROPULSION REACTORS****LIQUID CYCLE REACTOR**

The Aircraft Propulsion Reactors activity is concerned with the development of reactors suitable for the propulsion of aircraft or missiles or suitable for the production of auxiliary power for such vehicles. A liquid-cycle reactor is one of the types under development. In such a reactor, a liquid containing the fuel circulates through the reactor and a heat exchanger. The fluid for operating a turbine or jet is heated at the exchanger. Suitability of liquids for such reactors is based on extensive knowledge of their physical and chemical properties.

Fused Salts Research Project

The Liquid Cycle Reactor is considering the use of a fused-salt fuel system. Mound Laboratory had been assigned the problem of determining the phase relationships and physical properties of the components of some of the proposed fuel systems. Mound Laboratory's participation in this program has been concluded.

LiF-RbF-BeF₃ Ternary System - The phase equilibrium and temperature contour diagram summarizing the data obtained by differential thermal analysis, the identification of primary phases and boundary intersections of thermal gradient quench samples and slowly-cooled compositions, high temperature filtration studies, and X-ray diffraction appears as Figure 1. This is the same diagram as in last month's report since recent data have indicated a need for minor revision only.

Differential thermal analyses of compositions selected in the Li₂RbBe₂F₇-BeF₃ region yielded data in agreement with this portion of the diagram as previously presented. Apparently the high viscosity of fused salt mixtures in the high beryllium fluoride region leads to poor definition of the transitions.

Examination of compositions in the RbBeF₃-LiRbBeF₄ region by differential thermal analyses and thermal gradient quench techniques indicated that the RbBeF₃-LiRbBeF₄-Rb₂BeF₄ peritectic should be placed at 48 mole per cent beryllium fluoride, 12 mole per cent lithium fluoride, and 40 mole per cent rubidium fluoride, and that the area assigned to RbBeF₃ is larger than that indicated in Figure 1. The boundary between RbBeF₃ and Rb₂BeF₄ will be redrawn to include this larger area.

Examination of thermal gradient quench samples adjacent to the RbBeF₃ primary phase area revealed an abundant amount of quench growth RbBeF₃ which generally persisted through the entire length of a gradient tube. The quench-growth RbBeF₃ made identification of the true primary phase and its boundary intersection very difficult by optical methods. X-ray diffraction techniques were of no assistance under these conditions. The two ternary compounds, LiRbBeF₄ and "Li₂RbBe₂F₇", and RbBeF₃ cannot be identified accurately in the presence of one another. Therefore, mainly liquidus temperature and boundary intersection data obtained by differential thermal analyses were used for the construction of this portion of the phase diagram.

The primary phase in Composition R-46, Table 1, was found to be "Li₂RbBe₂F₇". At the valley intersection, Li₂BeF₄ appeared. Examination of this composition confirmed the location of the boundary line separating the primary phase areas assigned to Li₂BeF₄ and "Li₂RbBe₂F₇" in Figure 1.

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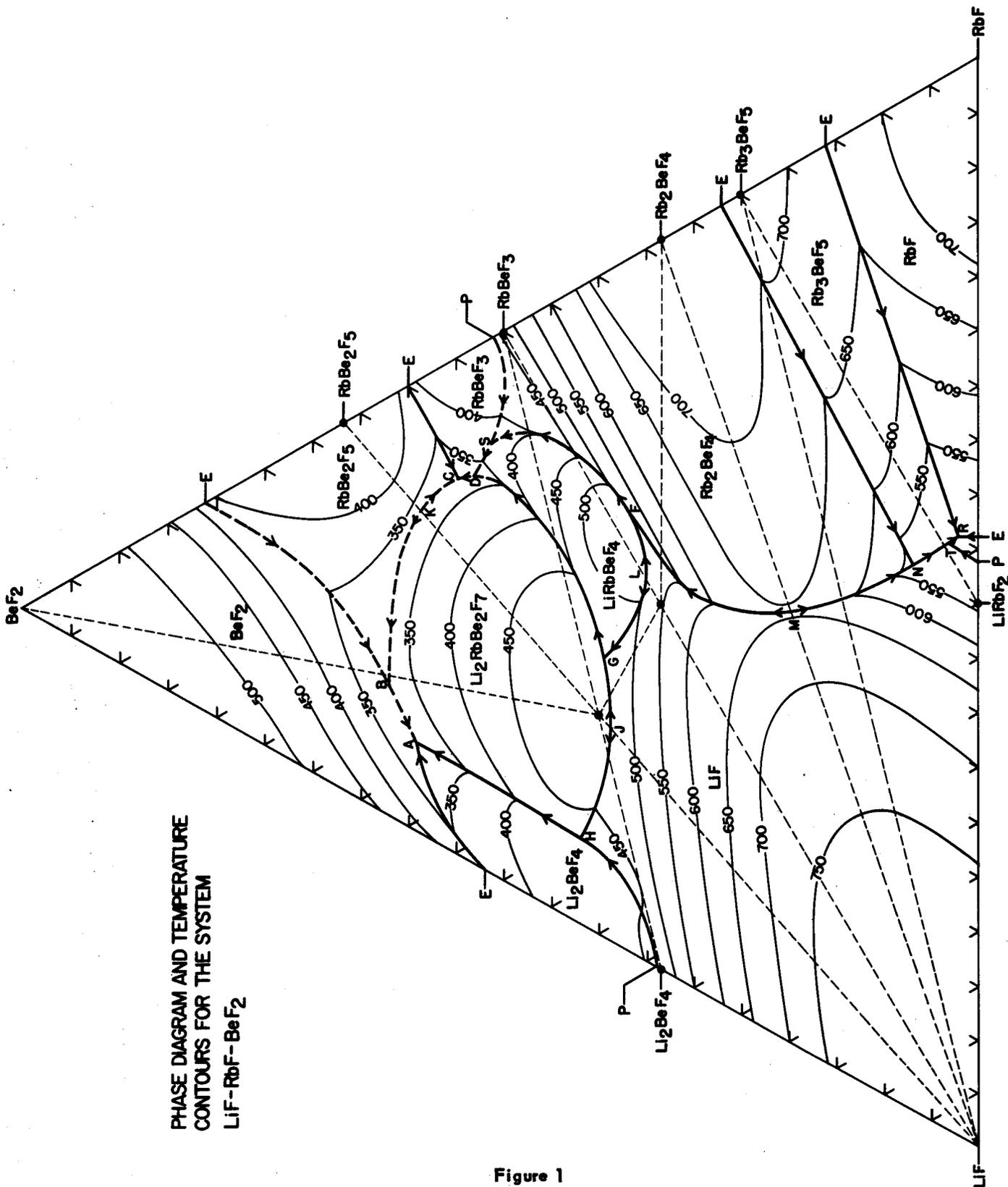


Figure 1

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TABLE I

Thermal-Gradient Quenching Data for the LiF-RbF-BeF₂ System

Comp. No.	Composition, Mole Percent			Phase Change Temperature °C	Phases Found Just Above Phase Change	Phases Found Just Below Phase Change	Interpretation
	LiF	RbF	BeF ₂				
40	15	35	50	358	Liquid	Liquid-Comp. A*	Liquidus and primary phase.
41	10	40	50	385	Liquid	Liquid-Comp. B*	Liquidus and primary phase.
42	30	5	65	405	Liquid	Liquid, BeF ₂	Liquidus and primary phase.
43	5	35	60	407	Liquid	Liquid, RbBe ₂ F ₅	Liquidus and primary phase.
45	13	29	58	336	Liquid	Liquid, RbBe ₂ F ₅	Liquidus and primary phase.
46	45	10	45	430	Liquid	Liquid, Comp. A	Liquidus and primary phase.
					Liquid, Comp. A	Liquid, Comp. A, Li ₂ BeF ₄	Boundary intersection.
47	15	40	45	430	Liquid	Liquid, Comp. B.	Liquidus and primary phase.
				400	Liquid, Comp. B	Liquid, Comp. B, Rb ₂ BeF ₄ *	Boundary intersection

* Not verified by X-ray diffraction

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In the last report the temperature of the $\text{LiF-LiRbF}_2\text{-Rb}_3\text{BeF}_7$ peritectic was not reported. The temperature of this peritectic has been determined to be 470°C .

Li_3UF_7 , Single Crystal Study - The space group and unit cell of a single crystal of Li_3UF_7 have been determined. The crystal was tetragonal. All hkl were present with hko present only when $h+k=2n$. The space group indicated was $D_4^2 - P4/n$ mm. It was found that $a = 6.111 \pm 0.005\text{\AA}$ and $c = 12.706 \pm 0.005\text{\AA}$. Experimentally the density was determined to be 4.84 g/cm^3 . At first inspection, c appeared to be 6.353\AA but the presence of some weak reflections indicated the value given above. The number of molecular units per unit cell, n , is equal to 4.

Preliminary calculations indicate that the crystal examined may be a defect-type of structure. The density of Li_3UF_7 was calculated to be 4.769 g/cm^3 on the basis of the following structure. One set each of the eight and sixteen-fold positions are occupied by fluorine atoms. One set of the four-fold positions are occupied by 3.5 uranium atoms with the remaining 0.5 four-fold positions occupied by the remaining fluorine atoms. If other single crystals of this compound can be produced, the study of its structure will be continued.

High Temperature Calorimeter - The first attempt to measure the thermal conductivity of Composition 30 was made with Cell 2 with the reference junction in a solid nickel cylinder in the dummy side of the calorimeter. Results reported in a previous report, CF-57-5-42 indicated that a large, unexpected correction was necessary because of the thermal resistance R , from sample to calorimeter and the unbalance of heat capacities between the sample and dummy side. In order to eliminate this difficulty, three more cells (No. 3, 4, and 5) were made with a reference junction inserted in a hole (0.063-inch by 1.13-inches deep) drilled in the wall of the outer shell. It was necessary to increase the wall thickness to 0.09 inch for this purpose. Composition 30 was used in Cells 3 and 4 and Composition 70 in Cell 5. All results obtained with these cells show a high degree of consistency. In order to save time and to double check the results, the calorimeter was by-passed and measurements continued in a furnace. This also enabled the remeasurement of Cell 2 with the reference thermocouple in a sleeve which had a light press-fit to the sample. The conductivity of Composition 30 in the liquid state as determined from all these measurements is $K = 0.00203 \pm 10$ per cent, with little temperature dependency. The limit indicated is a confidence factor, probably somewhat larger than a mathematically-derived one. The value of K in the solid state is essentially the same with a greater possibility of error because of voids caused by shrinkage during freezing.

Table II gives the characteristics of the cells and samples. Cell 2 is the same cell described in CF-57-5-42. Cell 3, with a gap of only 0.08 inch, was designed to impede convection and to cause the final results to depend more upon the heat capacity of the nickel bob than upon the salt. This is indicated in the table as the per cent of the total area in $^\circ\text{C-sec}$ originating from each (72 per cent vs 28 per cent). Cell 4, having a gap of 0.181 inch and a small bob, was much more subject to convection, and the value of K depended on the salt more than the bob (65 per cent vs 35 per cent). The dimensions of Cell 5 are considered optimum at this time.

Table III gives the complete results on Cells 2, 3, and 4 in the order run. Spotwelded thermocouple junctions of Chromel-Constantan have given little trouble except for the first set which did not track properly. This might account for the large values of K (solid) during the first heating cycle of Cell 3, although previous heat treatment may also be the cause, since the sample had been air cooled from 700°C prior to loading. Results show that no discontinuity exists in passing through the curie point of nickel (352°C), at which point the bob gives the greatest contribution to the area. Since the heat to the bob must pass through two salt-nickel films and the heat to the salt passes through only one, the constancy of K between samples and during salt or nickel phase changes can only mean a negligible film effect.

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TABLE II

Sample Characteristics

Cell	Bob O.D. in.	Container I.D. in	Weight of Nickel g	Weight of Sample g	Area (°C-sec) Due to Nickel %	Area (°C-sec) Due to Salt %	Salt Prepared
2	0.4825	0.7410	70.27	43.96	67	33	MOUND
3	0.3837	0.5436	146.31	19.12	72	28	ORNL
4	0.2450	0.6075	97.62	33.69	35	65	ORNL
5*	0.3830	0.6122	119.06	26.65	60	40	ORNL

* Composition 70, others Composition 30.

Since a value of $K = 0.00198$ was obtained with Cell 3 with no indication of a large error, and a tentative value of $K = 0.0035$ was reported previously with Cell 2, it was decided to remeasure K with Cell 2 in another furnace. First, however, Cell No. 3 was inserted (with a slip-fit) to the center of a 6-inch long, 2-inch O.D. by 0.800-inch I.D. nickel cylinder. A Chromel-Alumel thermocouple was inserted in the nickel cylinder and connected to a Foxboro controller. This assembly, with the normal differential couples in place, was inserted in a Type M2718 Hevi-Duty Electric Company furnace equipped with a brass liner of 2-inch I.D. by 2.25-inch O.D. The only furnace control at first was the Foxboro with an ON-OFF of 230 volts in series with a Ward-Leonard potentiometer. With this relatively crude set-up an average value of $K = 0.00183$ was obtained, in agreement with the calorimeter results within the possible error.

Two thermocouple wells were then drilled in the center and periphery of a solid nickel cylinder of 1.06-inches O.D. and 2 inches long. This cylinder was countersunk into the nickel block and the assembly inserted into the furnace as before. Three runs ranging from 30°C to 670°C gave $0.10 \text{ cal-sec}^{-1} \cdot \text{°C}^{-1} \cdot \text{cm}^{-1}$ as the conductivity of nickel whereas 0.13 is reported in most literature. This test indicates that there is negligible error originating from the thermocouples in measuring the thermal conductivity of Composition 30.

Next, the 1.06-inch cylinder was bored for a light press fit to Cell 2. Thus, with the exception of the discontinuity of the press fit, Cell 2 was similar to Cell 3 with the reference junction in the outer periphery of the sample. This assembly was lowered in the furnace as before. At this time differential ON-OFF control was obtained by connecting a 10 ohm - 50 watt Ohmite potentiometer across the Foxboro switch. The average of four runs was $K = 0.00205$, in excellent agreement with data obtained with Cell 2. Another run with rapid heating from 530 to 700°C gave $K = 0.00221$ with some indication of an increase in heat transfer when the differential temperature across the salt reached about 11°C.

Cell 4 was constructed to accentuate the effect of convection. It was first run in the furnace. During these runs the thermocouples were somewhat "noisy" but there was strong indication that values of K increased with temperature (0.00212 to 0.00268) as though some convection was unavoidable as the viscosity decreased. One run with a fast heating rate showed an abrupt temperature drop across the salt as the temperature differential was rising to about 9°C.

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TABLE III

Complete Thermal Conductivity Results on Composition 30 in Order Run

Cell	Environment	T ₁ °C	T ₂ °C	ΔT °C	K cal-sec ⁻¹ - cm ⁻¹ -°C ⁻¹	Remarks	
3	Calorimeter	141.9	227.4	85.5	0.00315		
3	"	227.4	326.7	99.3	0.00272	First set of thermocouples not tracking properly and "noisy".	
3	"	326.7	426.8	100.1	0.00242		
3	"	426.8	504.4	77.6	0.00243		
3	"	76.2	141.9	65.7	0.00218		
3	"	141.9	227.4	85.2	0.00227		
3	"	227.4	327.0	99.6	0.00226		
3	"	327.0	426.5	99.5	0.00207		
3	"	277.2	326.9	99.7	0.00241		
3	"	326.9	426.6	99.8	0.00217		
3	"	426.6	504.2	77.6	0.00210		
3	"	504.8	515.7	11.0	0.00240 ^a		
3	"	515.7	524.6	8.9	0.00219 ^b		
3	"	523.5	623.1	99.6	0.00180		
3	"	525.1	574.4	49.4	0.00211		
3	"	574.4	624.3	49.9	0.00196		
3	"	160.3	259.7	99.4	0.00216		
3	"	504.5	526.5	22.0	0.00205 ^c	New set of thermocouples, with excellent results.	
3	"	526.5	582.7	55.8	0.00198 [*]		
3	Furnace	126	265	139	0.00157	Large temperature oscillations because of single ON-OFF furnace control.	
3	"	264	399	135	0.00175		
3	"	530	630	100	0.00177 [*]		
3	"	630	530	100	0.00166 [*]		
3	"	265	400	135	0.00200 ^d		
3	"	530	630	100	0.00186 ^d		
3	"	630	530	100	0.00191 ^d		
3	"	530	630	110	0.00196 ^d		
Solid Nickel	"	30	260	230	0.10		Thermocouple wells in center and periphery of 1.06-inch solid nickel cylinder.
"	"	265	485	220	0.09		
"	"	485	670	185	0.11		
2	"	26	126	100	0.00168	Temperature oscillations reduced 80 per cent by differential ON-OFF Foxboro control.	
2	"	126	226	100	0.00176		
2	"	226	326	100	0.00186		
2	"	326	441	115	0.00196		
2	"	530	580	50	0.00201 [*]		
2	"	585	642	62	0.00196 [*]		
2	"	642	700	58	0.00214 [*]		
2	"	700	525	175	0.00211 [*]		
2	"	525	470	55	0.00217 ^e		
2	"	470	530	60	0.00198 ^e		
2	"	200	305	105	0.00213		
2	"	530	700	170	0.00221 ^f		
4	"	530	568	38	0.00212 [*]		
4	"	565	630	65	0.00230 [*]		
4	"	630	680	50	0.00268 [*]		
4	"	530	700	170	0.00326 ^g		
4	Calorimeter	35.6	61.2	25.6	0.00143 ^h	Precision of measurement about 1 per cent.	
4	"	475	Through	ΔH	0.00160 ⁱ		
4	"	525.6	576.9	51.3	0.00221 [*]		
4	"	577.2	625.3	48.1	0.00224 [*]		
4	"	529.9	79.9	50.0	0.00216 [*]		
4	"	579.4	627.4	48.0	0.00228 [*]		
4	"	515.5	524.4	9.3	0.00187 ^k		
4	"	524.4	544.6	20.2	0.00204 [*]		

* Plotted on Figure 2.

- a. 85% of ΔH (melting); ρ = 3.80 g/cm³
- b. 15% of ΔH (melting); ρ = 3.68 g/cm³
- c. ΔH = 48.2 cal/g, ρ = 3.75 g/cm³
- d. Sample can inverted

- e. ΔH = 56.8 cal/g, ρ = 3.75 g/cm³
- f. Fast heating, 11°C differential
- g. Fast heating, 9°C max. diff.; sharp break attributed to convection.

- h. Fast cooling, causing voids and low K values in solid.
- i. ΔH = 1.5 cal/g of 475°C break (which appears in all samples after fast cooling.
- k. 15% of ΔH 50 cal/g included.

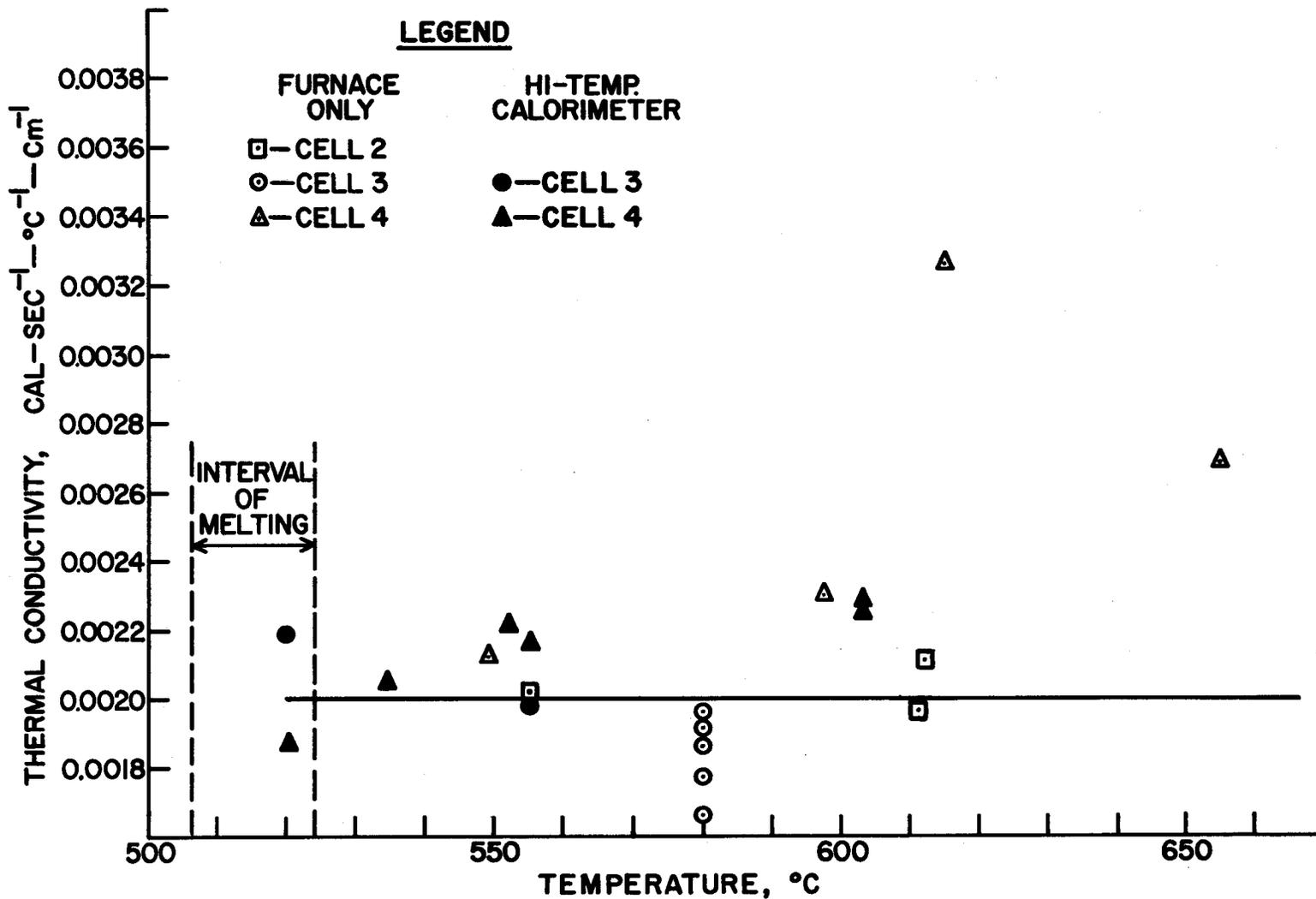


FIGURE 2

UNCLASSIFIED

THERMAL CONDUCTIVITY OF COMPOSITION 30 IN LIQUID STATE

Cell 4 was then put in the calorimeter. In the temperature range from 524.4°C to 544.6°C (just above melting) K was 0.00204, with values at higher temperatures ranging from 0.00216 to 0.00228. In these runs the differential temperature was about 1°C as in the furnace runs.

These conductivity cells become a crude calorimeter for the determination of ΔH of phase changes after the conductivity of the salt has been determined. In Table III ΔH was determined with the high temperature calorimeter and used to calculate K. The reverse of this can be done, i.e., K can be determined before a phase change and the area under a phase change can then be converted to calories. This method was applied to the 475°C break, which occurs only after rapid quenching. A value of 1.5 cal-gm⁻¹ resulted, in agreement with the calorimeter results.

The thermal conductivity measurement of Composition 70 in Cell 5 has not been completed. The initial value obtained, under adverse conditions, is $K = 0.0022 \text{ cal-sec}^{-1}\text{-cm}^{-1}\text{-}^\circ\text{C}^{-1}$. Two differences were noted between this salt and Composition 30. There was no 475°C break after fast cooling and there was much more heat of melting above 522°C. Melting began at essentially the same temperature, 506°C.

DEPARTMENT OF DEFENSE

U. S. ARMY SIGNAL ENGINEERING LABORATORIES

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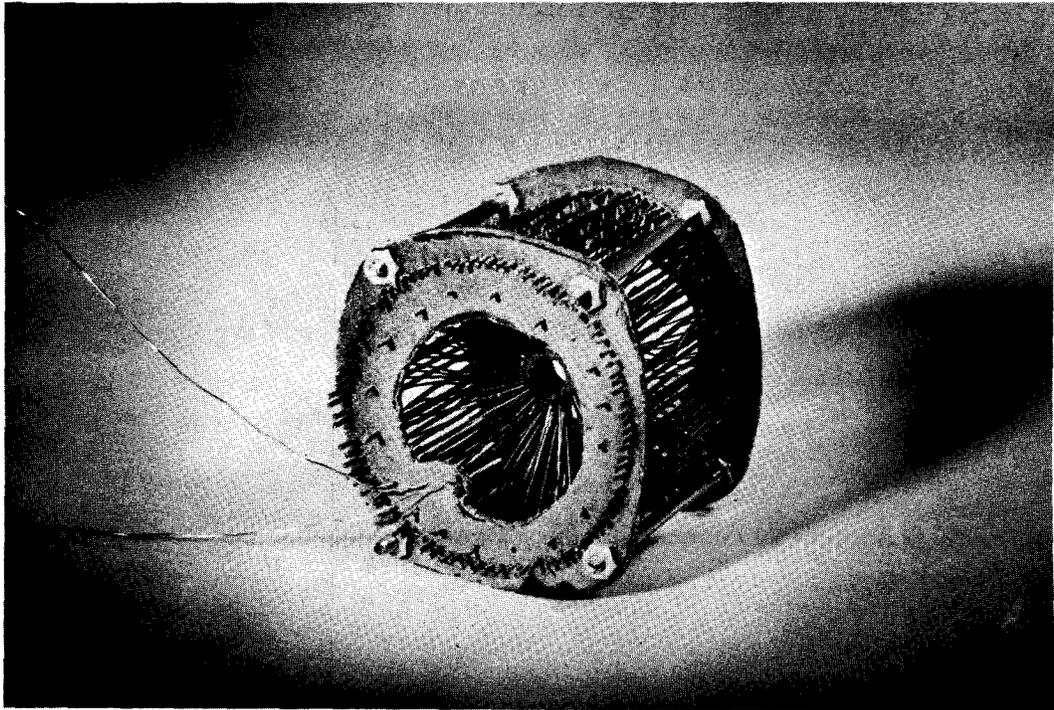
COMPONENTS BRANCH

(Contract No. R-50-799935-SC-01-91)

Nuclear Battery - Thermoelectric Type

The development of a system for the direct conversion of the energy of radioactive decay as heat into electrical energy is under investigation for the U. S. Army Signal Engineering Laboratories by Mound Laboratory. Radioactive materials are being evaluated as heat sources with consideration given to availability, cost, half-life, shielding, health hazard, and efficiency. Thermocouples, and factors for optimum shape and configuration for maximum efficiency output will be considered, and a prototype thermal battery will be constructed.

A thermoelectric generator having 120 junction pairs of the type described in MLM-CF-57-6-31 ("Monthly Technical Activities Report Through June 15, 1957") was constructed and being tested for output voltage and efficiency. Figure 1 is a photograph of the prototype generator.



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Figure 1 - Prototype Thermoelectric Generator