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MLM-CF-57-9-41

MONTHLY TECHNICAL ACTIVITIES REPORT THROUGH SEPTEMBER 15, 1957

By J. F. Eichelberger and
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Date: September 15, 1957
Issued: OCT 11 1957

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Miamisburg, Ohio Operated By
MONSANTO CHEMICAL COMPANY
Research and Engineering Division

United States Atomic Energy Commission
Contract Number AT-33-1GEN-53

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1ST REVIEW-DATE: 4/1/98	DETERMINATION (CIRCLE NUMBER(S)
AUTHORITY: ☐AOC ☒ADC ☐ADD	1. CLASSIFICATION RETAINED
NAME: T.M. FLANAGAN	2. CLASSIFICATION CHANGED TO:
2ND REVIEW-DATE: 4/13/98	3. CONTAINS NO DOE CLASSIFIED INFO
AUTHORITY: ADD	4. COORDINATE WITH:
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TABLE OF CONTENTS

	Page
PRODUCTION PROGRAM	4
NUCLEAR RESEARCH AND DEVELOPMENT	5
General Weapons Development	5
Plastics Project	5
Formulation	5
Evaluation	8
Service	10
Bridge-Wire Metallurgy	11
Physical Testing	11
Metallographic and X-ray Studies	13
Helium-3 Recovery Project	14
Gas Purification	14
Loading and Unloading Facility	14
Tritium Recovery Project	14
Isotope Separation Project	16
Gaseous Thermal Diffusion	16
Liquid Thermal Diffusion	16
Materials of Weapons Interest	18
Ionium Project	18
Protactinium Project	19
REACTOR DEVELOPMENT PROGRAM	20
Civilian Power Reactors	20
Homogeneous Reactor	20
Plutonium Solubility Studies	20
DEPARTMENT OF DEFENSE - U. S. ARMY SIGNAL	21
ENGINEERING LABORATORIES	21
Components Branch	21
Nuclear Battery - Thermocouple Type	21

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.

Twelve polonium-beryllium neutron sources and four alpha sources were shipped in August 1957. Four of the polonium-beryllium sources and one of the alpha sources were for other AEC sites. One plutonium-beryllium neutron source was shipped during the month of August 1957.

Twenty-three tanks of influent water were processed during this period. Five of the tanks required no pretreatment. Twelve of the tanks required one treatment of activated carbon and calcium chloride. Six tanks were given further treatment with barium chloride and sodium sulfide to reduce their beta activity level.

Data for the Waste Disposal Operations are given in Table 1.

Table 1

WASTE DISPOSAL DATA

Discharge Volume	624,800	gallons
Total Alpha Activity	5.5	millicuries
Total Beta Activity	16.5	millicuries
Activity Density		
Alpha	2.6	cts/min/ml
Beta	7.8	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 materials 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.*

Formulation The production equipment has been operating satisfactorily with the exception of the 5-micron filter. After several runs in which reasonable filtration rates were obtained using a vacuum, difficulty was encountered with clogging of the 5-micron filter. This was partially due to evaporation of solvent at the discharge side of the filter, and the application of pressure rather than vacuum was found to be advantageous. A new lot of Dapon-35 gave varnishes which were much more difficult to filter, and, since we were not equipped to use pressures exceeding 15 to 20 psig, the 5-micron filter was removed from the system after about half of the varnish for run B65C had been filtered. A Major Work Order has been written to procure a 5-gallon, paint spray tank with a stainless steel liner. This will permit filtrations at pressures up to 100 psig. Only the 25-micron filter will be used until this equipment becomes available.

Eleven sigma-blade mixer runs were made during this report period. Of these, four were for production batch PB1 (B59 through B62), two were experimental runs (B63 and B64) and five (B65A through B65E) were for the second production batch, PB2-65.

The formulation data and sigma-blade mixer end-points are summarized in Table 1.

As noted in last month's report, the separate prewetting technique presently used results in excellent kneading action throughout the entire sigma-blade mixer run. This results in a marked decrease in the fluctuations of power input, and gives a steep rise in power input as the end point is reached. Table 1 shows that use of the recording wattmeter permits determination of the endpoint within satisfactorily narrow limits, but that radical changes in formulation require a change in calculation.

Since the present Cuno filters cannot be flushed out or back-washed, a rather large and variable hold-up results, which causes significant variation in the amount of varnish introduced into the sigma-blade mixer from run to run. A fairly high percentage of this hold-up can be drained by inverting the filters, so this has been adopted as a standard procedure.

Ninety-five lots have been milled during this report period. Prevention of polymerization and control of the end point of solvent removal in the sigma-blade mixer has eliminated mill lots which could not be milled to as low a power input as was desired. The material prepared by the new separate wetting technique has superior milling characteristics, and no difficulty has been encountered in milling all lots to the desired net power input.

Table 1

DIALLYL PHTHALATE FORMULATIONS*

USING SEPARATE PREWETTING TECHNIQUE

Run Number	Parts per 100 Parts Dapon 35					Peak Power Input KW	Residual Volatiles %
	Cellulo 200	Hydrite PD-121	Acetone	Zinc Stearate	Calcium Stearate		
B59	40	60	235	3	-	1.3	11.1
B60	40	60	235	3	-	1.3	10.3
B61	40	60	235	3	-	1.35	11.2
B62	40	60	237	3	-	1.3	11.2
B63	20	80	215	3	-	1.36	9.3
B64**	40	60	200	3	-	1.3	12.4
B65A	40	60	246	-	3	1.4	12.5
B65B	40	60	229	--	3	1.3	12.3
B65C	40	60	200	-	3	1.45	12.4
B65D	40	60	225	-	3	1.3	11.3
B65E	40	60	244	-	3	1.3	12.0

* Weight of charge minus solvent, 26.7516; 5 parts monomer; 3 parts tert-butyl perbenzoate; 2.5 parts Scarlet 25AD; 0.5 parts TiO_2.

** Non-fibrous ingredients added to sigma-blade mixer as dry powder.

The thirty mill lots which were blended to make production batch PB1 illustrate the close control of milling which is now routine. These 30 lots represent five sigma-blade mixer runs. In some cases both mills were used simultaneously with different persons determining the net input power at cut-off.

The milling data for these mill lots are given in Table 2, while the flow times of the blends of the six mill lots of each sigma-blade mixer run are given in Table 3.

A given formulation, processed by a given technique, and milled to a given net power input will result in a product having a definite flow time within narrow limits. It has been shown that two different formulations milled to the same net power input will generally have different flow times. There is now evidence that two identical formulations processed by different techniques but milled to the same net power input may have different flow times.

Thus, the flow times obtained for B58 through B62 were lower than expected on the basis of the fact that essentially identical formulations were processed without separate prewetting. Again, run B57, which was identical to runs B58 through B62 except that the solvent was 100 percent benzene, gave a flow time of 8.28 seconds at five tons when milled to a net power input of 1.20 KW, although the mill times varied from 46 to 49 minutes.

Table 2

MILLING DATA FOR PRODUCTION BATCH PB1
(B58 THROUGH B62)

Mill Lot Number	Mill Time min.	Max. Surface Temp., °C	Milled Weight, Kg	Net Input Power, KW
B58-1	19.5	59	2.02	1.20
B58-2	17.25	59	2.10	1.20
B58-3	19	59	2.02	1.20
B58-4	19.5	59	2.05	1.20
B58-5	26.5	58	1.95	1.20
B58-6	23	58	2.04	1.20
B59-1	19.5	59	2.02	1.20
B59-2	19.5	59	2.06	1.20
B59-3	16	59	1.98	1.20
B59-4	17	56	2.02	1.20
B59-5	15.5	57	2.05	1.20
B59-6	19.5	56	1.82	1.10*
B60-1	20	58	2.04	1.21
B60-2	22.5	58	2.03	1.22
B60-3	19.5	59	2.03	1.20
B60-4	18	59	2.05	1.21
B60-5	18	58	2.03	1.21
B60-6	22	59	2.05	1.23
B61-1	16.5	58	2.00	1.20
B61-2	18.5	58	2.05	1.18
B61-3	22	59	1.98	1.20
B61-4	23	60	2.01	1.21
B61-5	24	58	2.10	1.20
B61-6	22	59	1.98	1.20
B62-1	23	59	2.02	1.20
B62-2	17.5	58	2.00	1.20
B62-3	17	58	1.97	1.21
B62-4	21	58	1.90	1.20
B62-5	15	57	2.10	1.20
B62-6	18	57	2.03	1.22

* Milled to 1.10 KW net input, as this lot was known to be well under standard weight.

Table 3

MOBILITY DATA FOR PRODUCTION BATCH PB1

(B58-1/6D THROUGH B62-1/6D)

Sample Number	Flow Time at 5 Tons sec	Deviation from Average sec
B58-1/6D	10.02	-0.29
B59-1/6D	10.27	-0.04
B60-1/6D	10.33	+0.02
B61-1/6D	10.19	-0.12
B62-1/6D	10.74	+0.43
Average	10.31	

It is believed that the longer mill times and the lower flow times of B57 are indications that the product from B57 was more homogeneous than the other products, but definite proof of this has not been obtained to date.

All material is dried in an air circulating oven with partial by-pass for 20 hours at 55°C. This gives a product whose residual volatile content is about the same, or lower, than the Mesa DAP being used currently. Uniform drying results have not been obtained to date. This is believed to be caused by a variable tendency toward a pseudo-sintering of the ground material which results in a variable porosity during most of the drying period. Preliminary results indicate that this effect can be minimized by stirring the plastic several times during the first few hours of drying.

Since drying is more complete in a given period of time the smaller the particle size, work is being continued on the development of a technique to reduce the maximum particle size to a minimum without producing an excessive amount of undesirable fines.

The Gyrocentric screen has been on hand for some time but has not yet been installed. Large quantities of plastic are accumulating pending its availability for the removal of fines. This is delaying the delivery of two 100-pound batches for Production Molding, and hence, their evaluation of our material.

Evaluation It is evident that it is not possible to mold consistently sound test bars with the present equipment and molds. This makes relative evaluation of different materials very difficult and precludes any absolute evaluation at the present time.

The small transfer-molding press has been ordered. Knock-out and bolt-hole locations have been specified, and a request for the design of molds for this press is being initiated.

The physical property values determined since the last reporting of tabular data are summarized in Table 4 and 5. All Mound Laboratory samples were formulated with 40 parts of Cellulo-200 and 60 parts of Hydrite-PD-121 except B44, B50, and B56 which had ratios of Cellulo to Hydrite of 30 to 70, 0 to 100, and 10 to 90, resepctively.

Eleven pounds of PB1 were screened with the Rotap to remove the material finer than 100 mesh since the Gyrocentric screen is not yet working. This was given to Production Molding. One-day operation of one press produced 380 heads which were carried through the first X-ray reading. Accepted heads totaled 94 per cent. The rejects were 4.0 per cent for inclusions, 1.3 per cent for cracks, and 0.8 per cent for voids. The average cure was 2.7 minutes with no sign of attack by the chloroform.

Some of the inclusions may have been foreign material introduced during processing since our present facilities are inadequate to prevent some contamination. However, it is believed that a majority of the inclusions are probably due to magnetite. The magnetite content of the Cellulo-200 is very low, but essentially all of the magnetite which it contains is in particles sufficiently large to show up on the X-ray film. It is planned to pass the ground plastic over a magnetic separator in the production facility; this should reduce the rejects for inclusions significantly.

Table 4

PHYSICAL PROPERTIES OF DAP FORMULATIONS

TENSILE STRENGTH AND IMPACT STRENGTH

Sample Number	Tensile Breaks Good	Tensile Breaks Bad	Range of Tensile Strength Values psi	Avg. Tensile Strength psi	Izod Impact Strength ft-lb per in. of notch
Mesa B1569	6	0	6206 - 7212	6823	0.266
Mesa B1660	5	1	6159 - 7619	7353	-
B44-2/4 + 6	4	2	4898 - 7376	6678	0.262
B47-1/6C	4	1	6250 - 8145	6497	0.232
B49-1/2CD	3	1	8127 - 8732	8345	-
B49-3/5D	3	0	6825 - 8438	7555	0.271
B49-3/5D	4	2	4651 - 6720	6116	0.256
B50-2	3	1	6000 - 6581	6216	-
B50-3	5	1	5520 - 7125	6417	0.263
B51-OD	2	1	4577 - 4642	4612	-
B52-1/6D	5	1	5664 - 7797	7154	-
B53-1/6D	5	1	5083 - 7451	6091	-
B55-1/6D	6	0	5424 - 6333	6004	0.284
B56	6	0	5354 - 7360	6097	0.271
B57-1/6	6	0	3280 - 7258	6592	0.271
B59-1/6D	5	1	6508 - 7565	6965	0.261
B61-1/6D	5	1	5966 - 8127	7068	0.269
PB-1	8	4	1782 - 7008	5764	0.278
PB-1*	3	1	5748 - 7008	6228	-

* Based on the four highest values of the 12 samples.

Table 5

HEAT DISTORTION TEMPERATURE AND ARC RESISTANCE OF DAP FORMULATIONS

Sample Number	Heat Distortion Temperature, °C	Arc Resistance sec
Mesa B1569	164	-
B44-2/4 + 6	153	-
B47-1/6C2	155	-
B49-1/2 CD	154	134.5
B49-3/5 D	170	135.8
B50-3D	142	-
B55-1/6D	156	137.8
B56D	154	-
B57-1/6D	150	133.9
B59-1/6D	151	132.5
B61-1/6D	143	134.9
PB-1	157	136.8

Service One member of the group spent two days in Chicago at the National Engineering Company where tests were run using the Simpson Mix-Muller A mixing time of 120 minutes appeared to give a consistency equivalent to 16 hours mixing in the sigma-blade mixer. The Mix-Muller therefore appears satisfactory for the initial liquefaction of the asbestos-varnish mixture. Certain modifications of the standard design will be required. These include: (1) stainless steel construction; (2) scraper blades for the wall and the mullers; (3) a vapor-tight cover; and , (4) modification of the lubrication system for the muller loading-springs and central drive shaft to prevent extraction of lubricant by the solvent.

A second member of the group spent one day in East Liverpool, Ohio at the Patterson Machine and Foundry Company. High-speed stirring experiments were run on material which had been treated in the Mix-Muller at Chicago. It was not possible to stir the mixture with a double-cone stirrer. However, a T-turbine stirrer was found to be satisfactory. When the T-turbine stirrer was first started, it merely turned in the thixotropic mass, stirring a very limited volume around the stirrer. The volume being stirred slowly increased, and, after an hour the entire mass was being stirred. The mixture was then stirred for two hours to give a relatively thin, creamy-appearing mass.

This material was shipped to Mound Laboratory where catalyst and monomer will be added, and the material will be processed for further evaluation.

A considerable amount of time has been spent in consultation with Engineering and Production Division personnel to firm-up equipment specifications and layout for the Plastics Production Facility. The basis for design is a charge of 150 pounds of prepolymer.

A complete flow curve was run for Mesa DAP B-1733. The flow was 11.15 seconds at five tons. This is the third 1000-pound lot which has been softer than the purchase specifications.

Bridge-Wire Metallurgy

An investigation of interalloy effects between gold and solder has begun in order to obtain information which may be of benefit in minimizing wire failures in 1E26 detonator heads.

Physical Testing A long range physical testing program has been established to evaluate the effect of aging on the gold-solder bond. Conductivity measurements on completed detonator heads, and tensile strength measurements of gold wires at solder junctions will be made on units which will be subjected to chemical, heat, and vibration treatments over an extended period of time. Embrittlement or weakening of the gold-solder joint and changes in resistivity of the solder joint are expected to be accelerated at elevated temperatures.

Both the conductivity and tensile strength measurements will be made at various time intervals up to 48 weeks. Each group of specimens will be subjected to one of the following treatments: (a) heat versus time, the specimens being held at 140°C and 128°C; (b) heat cycling versus time, the specimens being heated to 140° and 128°C respectively, with three cycles per day; (c) vibration and heat versus time, the specimens being vibrated in the frequency range of 1 to 100 cycles per second at 128°C; and (d) heat and the effect of foreign or corrosive substances versus time, with the specimens being at 140° and 128°C in sealed capsules containing water vapor, HCl, HNO_3, or H_2SO_4 atmospheres. These test conditions were devised to simulate possible storage conditions and to accelerate any chemical reactions or compound formation that might cause a failure of the solder junction. The temperatures were chosen on the bases of visual observations of the reaction between gold and solder and differential thermal analyses of the present Pb-Sn-In solder. Thermal analyses indicated a strong, sharp solidus at 137° ± 3°C, a boundary at 144°C and a liquidus at 176° ± 3°C. It was found that the gold wire was easily attacked by the solder at 150°C within a short period of time, and that this attack always resulted in a rupture of the gold wire. The temperature, 140°C, which is just above the solidus temperature, was selected for study of the accelerated reaction between solder and gold without rupture of the gold wire at the solder joint. Possible solid state reactions are being investigated by thermal treatment of specimens at 128°C, just below the solidus temperature of the solder. Studies at 110°C are also being made to permit the extrapolation of data to ambient temperatures.

Three types of wire-solder junctions were selected for the tensile strength experiments. (1) A single gold wire was soldered to one terminal of a detonator head (as in actual soldering of the detonator bridge wire) with three-inch leads extending from each side of the soldered junction. (2) A two-to-three milligram solder square was drilled with a No. 67 drill, was threaded on a two-inch length of gold wire, and was beaded by applying rosin and heating in an oven at 200°C for five minutes. (3) The same procedure as in (2) was used except that the bead was made by a soldering iron at a temperature the same as is used to solder the bridge wires.

During the preparation of samples for tensile strength measurements, it was found that the removal of the rosin (flux) was difficult using methyl alcohol since scrubbing of these specimens was not possible. Various solvents were investigated which might permit a better and easier removal of the rosin and residual material.

The solvents investigated, arranged in decreasing order of solvent efficiency (i.e., rate of solution and quantity of rosin held in solution), were: chloroform, xylene, tetrachloroethylene, benzene, trichloroethylene, acetone, isopropyl ether, carbon tetrachloride, butyl alcohol, ethyl alcohol, ethyl acetate, amyl acetate, butyl acetate and methyl alcohol. Chloroform appeared to be the most effective solvent; one liter dissolved 1000 grams of rosin. One liter of xylene dissolved 800 grams; butyl alcohol, 350 grams; ethyl alcohol, 300 grams; and methyl alcohol, 200 grams. At near saturation, methyl alcohol showed considerable turbidity and undissolved material.

Carbon tetrachloride showed some residue upon standing. The solvent effect of the alcohols on rosin appeared to be in the following order: butanol, ethanol, and methanol.

Methanol was one of the poorest solvents available both in speed of dissolution and quantity of rosin held in solution. During soldering with the rosin flux, the heat converts a part of the rosin to a material which is not readily dissolved in methanol. Also, an amorphous hydrolysis product is formed in the rosin-alcohol mixture when exposed to atmospheric moisture to give a ternary mixture of water, alcohol, and rosin. The same substance or precipitate can be obtained by placing a drop of water in an alcohol-rosin solution. Alcohols in general, and methyl alcohol particularly, show the least solvent effect on these two substances. The alcohols were the only solvents of those tested which yielded a white precipitate upon the addition of water.

In an examination of solder beads placed upon gold wires heavily encased in rosin, chloroform, xylene, tetrachloroethylene, benzene, and trichloroethylene almost immediately and completely dissolved the rosin products without agitatation or scrubbing of the gold-solder junction. A further investigation was made of the effect of these four solvents and of ethanol upon the plastic of the detonator heads. Weight and dimension changes as a function of time were recorded for heads soaked in each solvent. Data were obtained after air-drying the formed plastic for 15 minutes after removal from solvent. No changes were observed except in the cases of chloroform and ethanol. Heads soaked in ethanol showed a decrease in weight of about 1.2 milligrams in 24 hours, and a total weight loss of 2.5 milligrams in 142 hours. No dimensional changes were observed. Heads soaked in chloroform exhibited an increase both in thickness and in weight. These data are summarized in Table 1 and Table 2. No change in thickness was observed after 24 hours immersion. Tests with chloroform and xylene on complete heads indicated that a maximum of one minute contact time without agitation removes all traces of rosin. On the basis of these tests, chloroform and xylene merit consideration as cleansing agents for detonator heads.

Tests are being conducted now to determine how rapidly detonator heads soaked in chloroform return to their original weights and dimensions. Tests are also in progress to determine long-term effects on the plastic detonator heads when soaked in solvents up to ten minutes, which is ten times the maximum cleaning time necessary.

Table 1

EFFECT OF CHLOROFORM UPON THE PLASTIC OF THE DETONATOR HEADS

Change in Thickness Dimensions with Immersion in Chloroform

Sample No.	Original Thickness in.	Time of Immersion hr						
		0.5	1	18	24	48	72	142
1	0.14272	-0.00004	0.00000	0.00000	-0.00002	0.00008	0.00008	0.00018
2	0.14302	-0.00010	-0.00008	-0.00004	-0.00004	0.00012	0.00022	0.00050
3	0.14298	-0.00002	-0.00006	0.00002	0.00002	0.00008	0.00012	0.00032
4	0.14278	-0.00018	-0.00018	-0.00004	-0.00004	0.00032	0.00056	0.00108
	Average	-0.00009 (-0.1%)	-0.00008 (-0.1%)	-0.00002 (0.07%)	-0.00002 (0.07%)	0.00015 (0.1%)	0.00025 (0.2%)	0.00052 (0.4%)

Table 2

CHANGE IN WEIGHT WITH IMMERSION IN CHLOROFORM

Sample No.	Original Weight g	Time of Immersion hr			
		24	48	72	142
1	2.1255	0.0040	0.0071	0.0098	0.0192
2	2.1380	0.0090	0.0212	0.0301	0.0612
3	2.1166	0.0064	0.0138	0.0192	0.0377
4	2.0131	0.0113	0.0508	0.0736	0.1452
	Average	0.0078 (0.4%)	0.0232 (1.1%)	0.0332 (1.6%)	0.0658 (3.1%)

Metallographic and X-ray Studies A new type of clear plastic casting resin, Vibrin X-1089, manufactured by Naugatuck Chemical Division of U.S. Rubber Company, has proved to be a satisfactory mounting material for mounting metallographic specimens. A small amount of tertiary butyl hydroperoxide added to the resin effects polymerization at low temperatures. No pressure is required for casting. Fifteen-milliliter samples of the resin were polymerized under varying conditions of temperature, time, and percentage of catalyst to establish a set of conditions for curing the plastic to the best form. Acceptable results in a short time interval were achieved with the addition of two-thirds of one per cent tertiary butyl hydroperoxide and by heating slowly with an infrared lamp from room temperature to 70°C over a period of thirty minutes. Polymerizing this resin to the desired hardness at a constant temperature of 50°C required approximately four hours. This material has the following advantages over other common plastic mounting materials; it polymerizes rapidly at low temperatures and without pressure; it is clear and practically colorless; it can be cured to any degree of hardness; it is not smeared or balled up by heat of friction during cutting or coarse grinding; and, it is resistant to the etching reagents employed. It has proved to be an ideal resin for mounting the solder-coated gold wire.

A representative sample has been cast in each of six binary systems between the four metals, lead, tin, indium and gold. These samples have been successfully polished and etched and will be used to assist in identifying the binary phases in more complex mixtures.

In preliminary tests, 18-gauge gold wires were coated with the lead-tin-indium solder at various temperatures and were maintained in an excess of this solder at temperatures up to 250°C. The results varied from an observed reaction at the interface between the gold wire and the solder to a complete solution of the gold wire in the solder. In the intermediate cases, in which the gold wire was partially dissolved, the gold appeared to dissolve uniformly so a small core with a circular cross section remained of the original gold wire. It had been anticipated that a distinct layer containing intermetallic material would appear between the gold wire and the solder. No defined layer has been found. The dissolved gold or solid phase containing gold appeared to be either completely soluble in or completely dispersed through the solder matrix. No color change could be detected in the solder due to the dissolved gold. The characteristic gold color was apparently eliminated by combination with the other components.

A total of 19 binary compounds of gold, indium, lead, and tin have been reported in the literature. Each of these compounds is being prepared for use as a basis for metallographic identifications and x-ray diffraction studies of any intermetallic compounds found in the gold-solder junctions.

Helium-3 Recovery Project

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

Gas Purification The two uranium pumps installed in the gas purification hood in R-106 were activated with hydrogen. A temperature of about 425°C was necessary to start the reaction. Once started, the reaction was maintained at a rapid rate at 200° to 300°C. Hydrogen gas was metered into the pumps through a tank of calibrated volume with a pressure gauge attached at one end. Pressure-volume-temperature date were used for the measurements. The results are given below.

Table 1

ACTIVATION OF URANIUM PUMPS

	Uranium Charge, grams	Calculated Take-up liters	Actual Take-up liters	Per Cent Take-up
Pump 1	1986.8	308	299	97.2
Pump 2	1957.7	303	305	100.

Assembly of the third uranium pump is nearly complete. A charge of 1971.8 grams of depleted uranium has been sealed in this pump.

Further work with the palladium diffuser has eliminated all of the significant leaks from the casing, and evaluation of the device is now in progress. It was discovered that the thermocouples used to determine the temperature of the palladium tubing provide a considerable localized heat loss from the tubing, so that the thermocouples indicate temperatures from 200° to 400°C lower than the actual tubing temperature. Unless this situation can be rectified, use of the thermocouples for activating a controller will not be reliable. It is planned to install thermocouples made of very fine wire in an attempt to bring the indicated temperatures more into agreement with the actual palladium tube temperature.

With the exception of the palladium diffuser, the processing line for Savannah River Plant by-product gas is ready for operation.

Loading and Unloading Facility Modifications of the by-product line for trailer loading has been completed at the Savannah River Plant. A recent check of the vacuum tightness of the trailer which is to be loaded first showed that the rate of rise of the pressure from an initially low level was unsatisfactorily high. Inspection indicated that outgassing of foreign materials present in the vacuum manifold might be responsible. The gas lines are being cleaned, and loading is expected to begin next month.

Tritium Recovery Project

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

Preliminary design of the hot gas facility by the contracting architect engineer firm has been completed. The final design is expected to be completed by October 31, 1957. This facility will contain a 24-foot, one-inch diameter thermal diffusion column and associated equipment for the separation of tritium from mixtures of the hydrogen isotopes. This column will be similar to columns in use at Los Alamos Scientific Laboratory and Savannah River Plant and will have a wide range of flexibility so that, during the tritium recovery work, further evaluations of thermal diffusion and associated equipment as applied to tritium separation can be made. Three additional columns, 24 feet long by ¾-inch diameter are being designed for cascade operation on the enrichment of various isotopes of the noble gages. These columns and associated equipment will be housed in a separate hood in the same area.

As an addition to the isotope separation facility at Mound Laboratory, it is planned to install a low temperature distillation unit for the fractional distillation of hydrogen isotopes. The still was developed for Mound originally by the National Bureau of Standards Cryogenic Laboratory at Boulder, Colorado, and all work on the still to date has been done at that site. Mound Laboratory has had a representative at Boulder for the past month assisting in the modification of the still and in coordinating the efforts between the two sites. The still is to be transferred to Mound Laboratory about October 1 of this year. A representative of National Bureau of Standards will assist in getting the still into operation. The still is a packed column unit, with Helipac type 3013 packing.

Experiments at Boulder with this still have been made during the past month to determine the effectiveness of the separation process. Four distillation runs with a gas mixture of approximately 85 per cent hydrogen and 15 per cent deuterium (unequilibrated) under total reflux conditions yielded over 99 per cent hydrogen at the top and over 99 per cent deuterium at the bottom. The HETP (height equivalent of one theoretical plate) of the packed still was found to be in the range of 0.5 to 0.6-inch. The apparatus is now being dismantled and crated for shipment to Mound Laboratory.

An area suitable for the storage of materials containing tritium is being prepared on the fourth landing of the East tower of T-Building. Arrangements are being made with Los Alamos Scientific Laboratory for the transfer of several hundred grams of tritium as contained in various materials to Mound Laboratory for recovery of the tritium. A large part of the tritium is in the form of LiT mixed with LiD and will be stored until the hot facility is ready for operation. The remainder of the tritium is in water and mixed gaseous states.

Isotope Separation Project

Mound Laboratory is responsible for the production of several isotopes needed in research projects at several Division of Military Application 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 Collection of the first cycle of enriched neon-22 from the four 12-foot thermal diffusion columns in cascade has been completed. A total of approximately six liters, STP, having an average neon-22 composition of about 90 per cent was collected. The neon purity of this gas is better than 99.5 per cent.

While collecting this quantity of neon-22, approximately 100 liters of neon-20 of about 98 per cent isotopic purity were accumulated as a by-product. It is planned to recycle this material through the thermal diffusion column cascade to scrub out most of the remaining neon-21 and neon-22. The gas was collected in a propane-type storage tank, and is being pumped into the column feed system as needed by means of the mercury piston pump developed previously.

A total of 51 neon samples were analyzed on the mass spectrometer this month. This work was done in conjunction with the above experimentation. In addition, eight hydrogen and helium samples were analyzed to evaluate the efficiency of a blower-type, magnetically-driven, circulating pump.

Liquid Thermal Diffusion Additional runs have been made on H_2O - D_2O, again employing a 50-50 volume per cent mixture as feed. Extension of the column running time to 144 hours, using a nominal temperature difference of 15°C and an average hot wall temperature of 35°C, again failed to effect any separation.

The addition of two more layers of asbestos tape to the outside of the column did not greatly affect the temperature characteristics of the column. However, a reduction in water flow rate to 2.1 liters per minute increased the average hot wall temperature to 40°C and the temperature difference to 19°C, but only at the expense of producing greater vertical gradients. A 72-hour run under these conditions again failed to effect separation.

A refrigerated water line was next added to supply coolant to the column. Using a water flow rate of 1.6 liters per minute and full power, an average cold wall temperature, hot wall temperature, and temperature difference of 14°C, 48°C, and 34°C, respectively, were obtained, but again at the expense of marked vertical gradients. A 72-hour run under these conditions produced fractions whose densities at 25°C varied more or less continually from 1.0475 at the top of the column to 1.0509 at the bottom of the column. Although these results might indicate some separation occurring, two complicating circumstances were noted. First, the densities of the fractions were less than that of the feed (1.0513). Second, the fractions were slightly yellow in color, with a gradual increase in color intensity in going from the bottom to the top of the column. A check run is being made.

Analytical work on duplicate 24-hour runs (Runs 33 and 34), in which a TBP solution 0.1-molar in $Ce(NO_3)_3$ and 0.1-molar in $Y(NO_3)_3$ was used as feed, has been partially completed. The average hot wall temperature and temperature difference were 60°C and 40°C, respectively. The results are shown in Table 1. Both metals tended to concentrate in the bottom fractions, with some separation occurring. Oxidation of cerous cerium appeared to be negligible. A 48-hour run has been completed with this system, but no analytical results are available.

Table 1

ANALYSIS OF FRACTIONS FROM
24-HOUR RUNS WITH THE Ce - Y - TBP SYSTEM

Fraction Number	Yttrium Moles Per liter		Cerium Moles per liter	
	Run 33	Run 44	Run 33	Run 34
1(Bottom)	0.173	0.178	0.223	0.218
3	0.120	--	0.150	--
5	0.096	--	0.100	--
7	0.029	--	0.050	--
9(Top)	0.013	0.013	0.015	0.017

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.*

The contents of the mixer-settler units have been emptied and have been added to other materials for batch processing. Approximately 75 grams of ionium were in a batch which required hand processing into final product. During removal of uranium, about 20 grams of ionium was extracted into the organic with the uranium. An attempt to boil down the remaining 55 drums of material for concentration resulted in the precipitation of thorium sulfate. This sulfate was re-dissolved in about 20 gallons of water and precipitated with ammonium hydroxide in order to decrease the sulfate content. Later TBP extractions for uranium removal have lowered the total ionium content to about 42 grams. This material is ready for shipment, although complete analytical data are not available.

Clean-up operations are aimed at distribution of the remaining ionium as follows: one tank to contain nearly all the ionium with some uranium; one tank to contain most of the uranium with very little ionium; organic solutions to contain neither ionium nor uranium; and, precipitated aqueous raffinates to contain the rare earths and common elements. The precipitated raffinates are being prepared for burial. The organic is clean and will be held pending treatment of the ionium tank. The contents of the uranium tank will be sent back to Mallinckrodt Chemical Works in the stainless steel drums in which the feed was received.

A rough separation of thorium and uranium is being made by stripping both elements from the organic raffinates with 10 per cent sodium carbonate solution, and bringing this solution to a pH of about 5 with nitric acid. This treatment leaves enough carbonate ions to continue to complex the uranium but leaves the thorium free to precipitate as thorium hydroxide. After this precipitate settles, the supernate is decanted and acidified with nitric acid until the carbonate is removed. The uranium is then precipitated with ammonium hydroxide.

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 titanium precipitate described in last month's report had a wet volume of about ten gallons. This precipitate was washed three times with dilute hydrochloric acid to remove soluble iron. No protactinium was lost in this washing process.

The titanium precipitate was placed in the fifty-gallon kettle in SW 1-B and 13 gallons of 66° Baume sulfuric acid was added. The resulting slurry was heated for one hour at approximately 150°C and is now being processed in SW 1-B.

The rupture of a kettle fitting in HH allowed about 100 milligrams of protactinium to run into the "hot" sump. The contents of the sump were pumped out into drums, but the polonium content was too high to allow safe processing. The drums, containing about 200 gallons of solution, will be held in the high-risk side of HH-Building until the polonium decays.

Purification Work has been resumed on preparing a homogeneous solution from the first gram of protactinium. After the homogeneous solution has been obtained, final purification will begin.

Raffinate Disposal Approximately 10,000 gallons of radioactive acid waste solution is on hand from the processing of the second gram of protactinium. This solution has been stored in lever-lock steel drums having polyethylene liners. At least two liners, each 0.004-inch thick, have been used in each drum. The liners have been developing leaks, and the solution has attacked the steel drums, thereby permitting the solution to be discharged on the ground.

Several methods of disposal of this material have been studied. Thirty-gallon, polyethylene-lined, fiber drums are on hand, and about 60 have been filled with raffinate. Of this 60, about 20 have developed leaks in a week. Examination indicates pin hole leaks in the liners.

It is believed that the pin holes were present when the drum was filled and did not develop upon standing. Extreme precautions will be taken with the next batch of drums to make sure that no pin hole leaks are formed during the filling operation.

The presence of a pin hole leak cannot be immediately detected. The solution seeps through the pin hole and slowly softens the wax-lined fiber covering. After the fiber becomes wet, its strength is decreased to such an extent that the head of liquid bursts the drum.

REACTOR DEVELOPMENT PROGRAM

CIVILIAN POWER REACTORS

Homogeneous Reactor

The Civilian Power Reactor Program in 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.*

Studies were continued on the chemistry and solubilities of plutonium carbonates.

A mixture of H_2 and CO_2 gases was bubbled through a slurry of plutonium (III) hydroxide in water. The blue, aqueous portion was then sealed in a capillary and was found to give yellowish-white crystals on heating to 139°C. The plutonium concentration was 1.10 grams per liter. In a second experiment plutonium (III) hydroxide was precipitated in a capillary tube. After washing the precipitate with water which had been saturated with CO_2, 50 lambda of water and three to five milligrams of CO_2 were added to the capillary. The tube was sealed, and, on heating to 138.5°C, fine, white needle-like crystals, which appeared yellowish on settling, came out of solution. The concentration of plutonium in the tube was 1.15 grams per liter.

Plutonium (IV) carbonate in 0.5-molar lithium bicarbonate solution has a saturation value of 0.057 grams of plutonium per liter at 24°C.

Plutonium (VI) has been prepared by oxidizing plutonium (IV) sulfate with ammonium persulfate. The resulting plutonium (VI) is precipitated with ammonium hydroxide, and the precipitated ammonium plutonate is redissolved in a 0.5-molar lithium bicarbonate solution. It had a saturation value of 5.77 grams of plutonium per liter at 24°C.

Several runs were made with lithium bicarbonate under CO_2 overpressure to check our results against those of Marshall, Laprest and Secoy. Lithium bicarbonate (0.5 molar) came out of solution at 98°C with no CO_2 overpressure, and at 208°C with a CO_2 overpressure of 1015 psi. This agrees with their results.

DEPARTMENT OF DEFENSE

U. S. ARMY SIGNAL ENGINEERING LABORATORIES (Components Branch)

(Contract No. R-50-799965-SC-01-91)

NUCLEAR BATTERY - THERMOCOUPLE 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 optimum shape and configuration for maximum efficiency output are being considered, and prototype thermoelectric generators are being constructed.

A thermocouple electrogenerator was constructed as described in last month's report (CF 57-8-34). It consisted of three "wheel" sections assembled around a 0.007-inch nickel sleeve about 5/16-inch long and 1/4-inch in diameter. The assembled generator was a thermopile having 122 junctions. To simulate the radioactive heat source, a resistance heater was wound about a small ceramic core mounted on ceramic spaghetti and was fitted with a Chromel Constantan thermocouple (see Figure 1). The heater and generator were closely fitted into a brass container insulated at the bottom by a Styrafoam disk, and the unoccupied interior space was filled with Santocel. The brass container was placed in a constant temperature bath at 28.4°C and was tested for overall efficiency at various power input levels. The results are listed in Table 1.

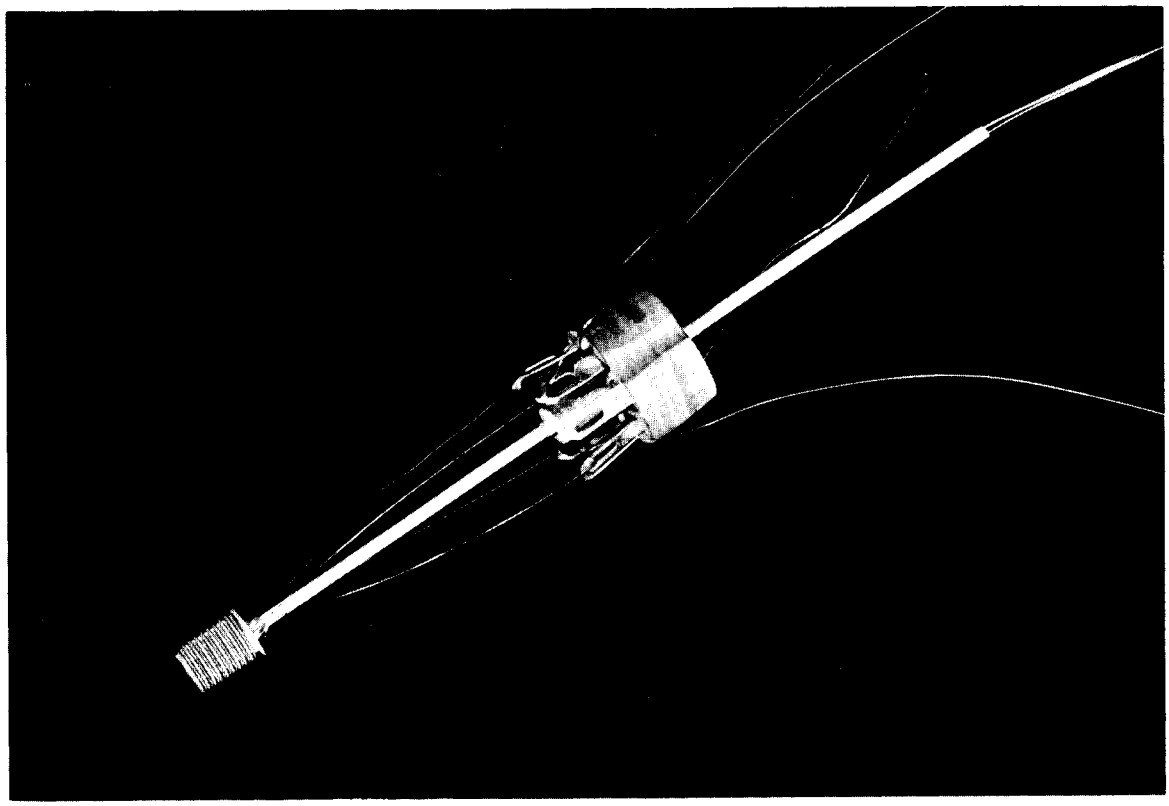

Figure 1

Table 1

EFFICIENCY OF THERMOELECTRIC GENERATOR

Power Input watts	ΔT_1 heater to water bath, °C	ΔT_2 hot to cold junctions, °C	$\Delta T_1/\Delta T_2$	Open Circuit Output Voltage volts	Load Resistance ohms	Power Output watts $\times 10^{-4}$	Efficiency %
1.155	99.8	48.5	2.058	0.362	40	8.17	0.071
2.397	189.4	97.7	1.939	0.751	40	34.38	0.143
3.626	269.9	142.2	1.898	1.122	42.5	76.01	0.210
5.042	357.0	188.8	1.891	1.592	42.5	140.6	0.279
7.620	481.1	275.4	1.869	2.154	42.5	275.7	0.362
9.912	577.5	312.5	1.845	2.667	42.5	422.3	0.426
13.167	674.3	367.3	1.836	3.190	40	603.4	0.458
15.265	735.7	408.3	1.802	3.580	42	754.5	0.494
19.37	832.6	477.7	1.743	4.251	45	1058	0.546
25.70	722.6*	558.6	1.294*	5.028	40	1501	0.584

* Styraform insulation melted.

Plotted values showed a few unexpected deviations which were explained when inspection showed the Styrafoam layer had melted at the higher temperatures, giving a somewhat lower efficiency. About half the theoretical maximum efficiency as calculated by Dr. Maria Telkes has been achieved for this particular thermocouple pair.