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MONTHLY TECHNICAL ACTIVITIES REPORT THROUGH OCTOBER 15, 1957

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MONSANTO CHEMICAL COMPANY
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MOUND DECLASSIFICATION REVIEW	
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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.

Eleven polonium-beryllium neutron sources and six alpha sources were shipped in September, 1957. Five of the polonium-beryllium sources and two of the alpha sources were for other AEC sites. There were no plutonium-beryllium neutron sources shipped during the month of September, 1957.

Thirty-one tanks of influent water were processed during this period. Four of the tanks required no pre-treatment. Nineteen of the tanks required one treatment of activated carbon and calcium chloride. Seven tanks were given a second treatment with the same reagents, and one tank required additional treatment with barium chloride to reduce the beta activity level.

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

Table 1

WASTE DISPOSAL DATA

Discharge Volume	808,000	gallons
Total Alpha Activity	7.0	millicuries
Total Beta Activity	30.4	millicuries
Activity Density		
Alpha	2.6	cts/min/ml
Beta	11.0	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 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 Twelve sigma-blade mixer runs were made during this report period. Eight of these were made to produce material for production molding while the other four were experimental batches. All runs used 40 parts of Cellulo-200 asbestos and 60 parts of Hydrite-PD-121 Kaolinite per 100 parts of Dapon-35 diallyl phthalate prepolymer. With the exception of batch B66, which was not pigmented, all runs contained 2.5 parts Scarlet 25AD, and 0.5 part TiO_2 per 100 parts of Dapon-35. Other pertinent data for these runs are summarized in Table 1.

Table 1

DIALLYL PHTHALATE FORMULATIONS USING SEPARATE PREWETTING TECHNIQUE

Run Number	Parts Per 100 Parts Dapon-35: Acetone	Tert-Butyl Perbenzoate	Monomer	Stearate: Zinc	Calcium	Weight of Charge Minus Solvent lb.	Peak Power Input KW	Residual Volatiles %
PB2-65F	217	3.0	5.0	-	3.0	26.75	1.30	12.9
B-66*	-	4.8	7.9	2.0	-	~17.5*	0.90	12.9
B-67	236	3.5	5.0	-	3.0	26.81	1.28	12.9
B-68	248	5.0	8.0	3.0	-	27.37	1.28	11.3
PB3-69A	235	3.0	5.0	3.0	-	26.75	1.25	12.3
PB3-69B	226	3.0	5.0	3.0	-	26.75	1.30	11.3
PB3-69C	225	3.0	5.0	3.0	-	26.75	1.35	12.1
PB3-69D	225	3.0	5.0	3.0	-	26.75	1.38	10.3
PB3-69E	225	3.0	5.0	3.0	-	26.75	1.28	11.5
PB3-69F	225	3.0	5.0	3.0	-	26.75	1.30	12.1
PB3-69G	225	3.0	5.0	3.0	-	26.75	1.28	11.7
B-70	225**	3.0	5.0	3.0	-	26.75	1.28	11.1

* Material was liquified in Mix-Muller and stirred with T-turbine stirrer; charge weight was about 2/3 of normal charge.

** Methyl ethyl ketone use in place of acetone.

PB2-65F was the sixth and final run of the second production batch. PB2 was essentially identical to PB1 except for the use of calcium stearate in place of zinc stearate. PB3-69A through PB3-69G comprised the third production batch. This was essentially identical to PB1. Two extra runs were made so that 50 pounds of material would be available for equipment specification experiments planned by Engineering Division.

B66 was run from material used in mixing tests done at National Engineering Company with the Simpson Mix-Muller and later at the Patterson Machine and Foundry Company with a T-turbine stirrer. The high monomer and catalyst concentrations were due to the adding of normal weights of these ingredients without realizing that approximately one-third of the original material had been lost in the experiments made with this material. B66 appeared to be the most uniform and homogeneous of any sigma-blade mixer run made to date.

B68 was made with essentially the same catalyst and monomer content as B66 in order to produce a full-size run for comparison with B66. It did not seem to be as homogeneous as B66.

B70 was a standard run except that methyl ethyl ketone was used as the solvent in place of acetone. The processing was similar to that of B57, the run in which benzene was used as the only solvent. Methyl ethyl ketone appears to act more like benzene than like either acetone or acetone-benzene mixtures.

A total of 94 mill lots were milled on the differential rolls during this report period. With the exception of B66-2, B66-3 and B66-4, all were milled to a nominal value of 1.30 KW net input power. Omitting PB3-69D-6, the range for 90 mill lots was 1.29 to 1.33 KW with an average of 1.303 KW. PB3-69D-6 stiffened somewhat more rapidly than was anticipated and exceeded 1.3 KW net input power while the operator was otherwise engaged. The milling data are summarized in Table 2.

Both PB2 and PB3 have been coarse-ground, aged, fine-ground, dried, screened (removing material smaller than 100 mesh), and blended. PB3-69C-1/6 and PB3-69D-1/6 were not screened. They were blended separately from PB3 to provide 50 pounds of material for drying and magnetic separation tests.

Experimental batches B66, B67, and B68 processed normally on the mill rolls, but B66 and B68 (high monomer and high catalyst) retained more flexibility in the milled sheet form. B68 had a much greater tendency to cake during grinding and drying. It also tended to bridge in the feed hopper of the gyrocentric screen.

Evaluation Only limited evaluation work has been done this month because of dismantling work being done in the E-Building and because most of our time has been used in producing material for Production Molding. Some test bars have been molded. A portion of these will be X-ray inspected before evaluation.

Service The remainder of PB1 was screened with the gyrocentric screen, and 84 pounds were turned over to Operations Division for Production Molding. PB2 was completed and the following samples have been shipped: 100 pounds to Production Molding, 10 pounds to Picatinny Arsenal, and 5 pounds to Los Alamos Scientific Laboratory. PB3 was completed; 100 pounds have been sent to Production Molding and 50 pounds have been set aside for Engineering Division.

Table 2

MILLING DATA FOR DAP FORMULATIONS

Mill Lot Numbers	Mill Time Range min	Mill Time Avg min	Max. Plastic Surface Temp. Range °C	Milled Wt. Range KW	Net Input Power Range KW
PB2-65A-1/6	17.0 - 19.5	18.5	60	1.92 - 1.99	1.30 - 1.31
PB2-65B-1/6	20.5 - 29.0	26.0	59 - 61	1.99 - 2.05	1.30 - 1.31
PB2-65C-1/6	18.0 - 23.5	20.5	59 - 61	2.01 - 2.05	1.30 - 1.31
PB2-65D-1/6	28.0 - 30.5	29.4	60 - 61	2.10 - 2.12	1.30 - 1.31
PB2-65E-1/6	17.25 - 24.0	21.25	59 - 61	2.00 - 2.10	1.30 - 1.32
PB2-65F-1/6	20.0 - 24.0	22.0	59 - 60	1.95 - 2.06	1.30 - 1.32
B66-1	28.5		60	2.00	1.30
B66-2	31.0		62	1.90	1.37
B66-3	33.0		63	1.93	1.35
B66-4	35.0		63	1.95	1.40
B67-1/6	19.0 - 26.0	21.8	59 - 62	1.95 - 2.02	1.30 - 1.32
B68-1/6	28.0 - 31.25	29.7	58 - 61	1.98 - 2.07	1.30 - 1.31
PB3-69A-1/6	25.0 - 27.0	26.2	59 - 61	2.06 - 2.10	1.30 - 1.30
PB3-69B-1/6	25.0 - 28.0	26.3	60 - 61	2.09 - 2.11	1.30 - 1.33
PB3-69C-1/6	25.0 - 27.0	25.9	60 - 61	2.00 - 2.05	1.30 - 1.31
PB4-69D-1/5	20.0 - 28.0	24.1	60 - 61	2.08 - 2.12	1.30 - 1.31
PB4-69D-6	20.0		61	2.12	1.37
PB4-69E-1/6	22.5 - 25.5	24.8	56 - 60	2.10 - 2.14	1.30 - 1.30
PB4-69F-1/6	19.0 - 25.0	22.8	60 - 60	1.96 - 2.15	1.29 - 1.31
PB4-69G-1/6	21.5 - 26.0	23.5	57 - 59*	2.00 - 2.10	1.29 - 1.31

* Different thermocouple system used on this mill run.

A complete flow curve was run on Mesa DAP B-1777. The flow time at five tons was 10.67 seconds, well below the specification of 14 ± 1 seconds.

Ingredients equivalent to one standard sigma-blade mixer run (except for pigment), and also sufficient material to make two gallons of non-fibrous ingredient slurry have been sent to Chemineer, Inc. These will be used for tests to establish design specifications for the 25 and 75-gallon mixers for the Plastic Production Facility.

Bridge-Wire Metallurgy

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

Physical Testing A series of tensile strength tests for the detection of embrittlement or weakening of the gold-solder junction has been made with specimens held at 140°C and 130°C for a period of one to three weeks. The majority of samples broke down at the solder junctions during the heat treatment; the samples held at 140°C broke within the first week, and the samples held at 130°C broke during the second or third weeks. The gold wire often appeared to be "wetted" by the solder for a considerable distance from the solder bead. Those specimens which had not ruptured during heating exhibited a greater tensile strength at the gold-solder bond than that of the gold wire alone. This phenomenon was attributed to the reinforcing of the wire by the solder layer even though the solder may have dissolved part of the gold wire.

Specimens sealed in a moisture-saturated atmosphere and held at a 130°C for a period of four weeks showed no decrease in tensile strength of either the solder-gold junction or the gold wire. Little "wetting" of the wire occurred, and the number of breakdowns occurring during the heat treatment were less. The solder beads were extremely corroded; there were large cracks and fissures in the surface. A black, oxide layer formed on the solder surface, and a general discoloration was found within the solder bead. Moisture appears to be the most deleterious single environmental factor examined to date. Samples contained in a moisture-saturated acid atmosphere gave results similar to those obtained with samples sealed in an atmosphere saturated with water vapor.

Conductivity measurements were made on production detonator heads with gold bridge wire soldered in place. These heads were held at various temperatures under various conditions. Heads heated continuously at 140°C showed a slight decrease in resistance after the first week of heat treatment because the solder "wetted" the entire length of gold wire with a thick, but uneven, coat of solder, and thus reduced the total resistance. The increase in resistance after the second week of treatment would suggest that the gold wire had undergone a partial dissolution. These solder mounds were badly puddled and very uneven. Some heads held at temperatures of 130°, 120° and 110°C showed considerable increase in resistance. In each case of increased resistance there was visual evidence of an elongation of the gold wire as if the wire had pulled out at one or both solder mounds. The pulled-out sections of the gold wire appeared to be coated with solder and to have elongated many times the amount necessary to compensate for any expansion and contraction of the plastic head. The deformation of the gold wire was most serious at the higher temperatures, particularly for those samples which had been cycled. No complete ruptures were observed nor was an infinite resistance obtained for these wires. The solder mounds remained smooth and showed no puddling as in the 140°C specimens. However, slight puckering at the edges of the mounds was in evidence for all samples indicating that some flow occurred as low as 110°C.

Those specimens which showed practically no change during the first week also showed little change during the second week of treatment. Those which had shown considerable change initially were also increasingly affected during the second week of treatment. Since some samples were unaffected, it is suggested that variations occurred in the fabrication of the gold-solder bond. The tension placed upon gold wire, the effectiveness with which the gold wire was "tinned", the amount of solder on the wire outside the mound, etc., might have varied considerably.

Temperatures previously chosen for accelerating breakdown of the gold-solder bond have been in a partial liquidus region or a region of plastic flow of the solder. Consequently, tests are being made at the lower temperatures of 120°, 110°, 100° and 90°C. Since moisture appears to be extremely corrosive toward the solder mounds, evaluation tests at these temperatures are in progress at relative humidities controlled from zero per cent to 100 per cent.

Preparation of gold-solder bond specimens for physical testing requires a solvent for the rosin which is more effective than methanol. Scrubbing techniques such as are employed on production detonator heads cannot be used on the test specimens. As discussed in the previous technical activities report, MLM-CF-57-9-41, chloroform, tetrachloroethylene, and xylene are excellent solvents for the removal of excess rosin. Also, moisture in these solvents does not react with the rosin to form an amorphous, insoluble residue as is the case with the alcohols.

The examination of these solvents has been extended to observe any weight and dimension changes of detonator heads as a function of the cure-time of the plastic when molded.

The increase in weights found for heads cured one, two, three, and four minutes when immersed in the three solvents and in methanol for various periods of time up to 48 hours are listed in Table 1. After removal from the solvents, the heads were air-dried at room temperature for approximately 20 minutes before weighing. These data are the average increase in weight of three heads for each condition and are considered to be accurate to ± 0.1 milligram. The results are representative since it is difficult to produce heads under identical cure conditions. These data indicate that the effect of chloroform is greatest on the plastic cured one minute and that extended immersion in either methanol or chloroform is undesirable. The heads cured one, two, and three minutes are six, four, and two times, respectively, more affected by chloroform than the heads cured four minutes. The heads cured one minute showed a continuing increase in weight during immersion in chloroform, reaching a six per cent increase after 48 hours. Similarly-cured heads immersed in methanol showed a 0.2 per cent increase in weight in 48 hours. Plastic heads cured for two, three, and four minutes showed no appreciable change in weight after one-half hour immersion in any of the four solvents. Xylene and tetrachloroethylene had no observed deleterious effect upon the plastic regardless of curing time or to an exposure of up to 48 hours in the solvents.

Dimensional changes were evaluated under the same conditions. No increase in thickness greater than 0.0001 inch was observed except with the heads soaked in chloroform. Six hours exposure to chloroform for all cure times caused less than 0.0001 inch increase in thickness. After 48 hours immersion in chloroform, the thickness of heads cured one minute had increased 0.001 inch, heads cured two minutes had increased 0.0008 inch, heads cured three minutes had increased 0.0004 inch, and heads cured four minutes had increased 0.0001 inch.

Data were obtained reflecting the rate at which the heads which had been soaked in chloroform for 48 hours returned to original weight and dimensions while standing at room temperature. The change in weight versus time for each of the four cure times is plotted on Figure 1. Changes in weight were found to be the more sensitive method of measuring the effect of a solvent on a plastic head.

Table 1

WEIGHT CHANGE OF HEADS SOAKED IN VARIOUS SOLVENTS (Grams)

SOLVENT	2 min.	6 min.	12 min.	20 min.	30 min.	1 hr.	2 hr.	6 hr.	24 hr.	48 hr.
1 Minute Cure; Avg. Initial Weight, 2.2576 g.										
Chloroform	0.0004	0.0010	0.0016	0.0019	0.0025	0.0033	0.0055	0.0159	0.0650	0.1358
Xylene	0.0004	0.0004	0.0005	0.0004	0.0005	0.0002	0.0007	0.0008	0.0006	0.0008
Tetrachloroethylene	0.0003	0.0003	0.0004	0.0004	0.0004	0.0003	0.0007	0.0007	0.0006	0.0006
Methyl Alcohol	0.0002	0.0005	0.0005	0.0004	0.0005	0.0004	0.0009	0.0012	0.0019	0.0030
2 Minute Cure; Avg. Initial Weight, 2.2551 g										
Chloroform	0.0002	0.0003	0.0005	0.0006	0.0007	0.0014	0.0029	0.0079	0.0369	0.0847
Xylene	0.0002	0.0003	0.0003	0.0003	0.0003	0.0002	0.0006	0.0005	0.0005	0.0005
Tetrachloroethylene	0.0002	0.0005	0.0005	0.0004	0.0003	0.0002	0.0007	0.0008	0.0006	0.0005
Methyl Alcohol	0.0033	0.0004	0.0004	0.0003	0.0002	0.0003	0.0008	0.0010	0.0017	0.0027
3 Minute Cure; Avg. Initial Weight, 2.2441 g.										
Chloroform	0.0005	0.0006	0.0005	0.0006	0.0007	0.0009	0.0020	0.0046	0.0184	0.0437
Xylene	0.0003	0.0003	0.0002	0.0003	0.0002	0.0002	0.0007	0.0007	0.0005	0.0005
Tetrachloroethylene	0.0004	0.0004	0.0002	0.0003	0.0004	0.0003	0.0007	0.0009	0.0006	0.0007
Methyl Alcohol	0.0004	0.0003	0.0002	0.0002	0.0003	0.0001	0.0008	0.0010	0.0014	0.0024
4 Minute Cure; Avg. Initial Weight, 2.2498 g.										
Chloroform	0.0003	0.0003	0.0003	0.0004	0.0005	0.0006	0.0015	0.0027	0.0098	0.0225
Xylene	0.0004	0.0004	0.0003	0.0003	0.0003	0.0004	0.0008	0.0008	0.0006	0.0008
Tetrachlorethylene	0.0003	0.0003	0.0002	0.0003	0.0003	0.0002	0.0007	0.0007	0.0004	0.0006
Methyl Alcohol	0.0004	0.0004	0.0003	0.0003	0.0005	0.0003	0.0008	0.0009	0.0013	0.0023

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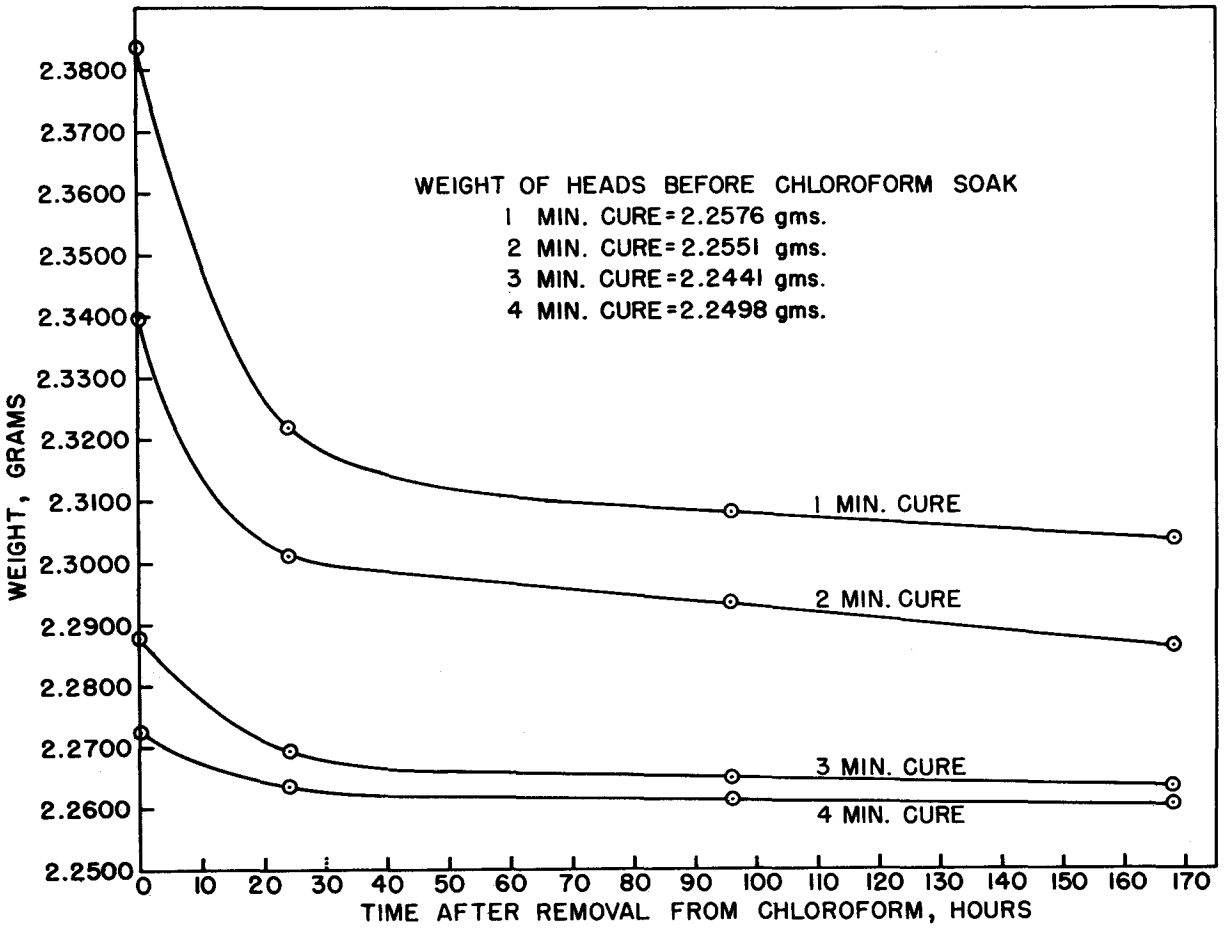

Figure 1 Weight Change of Heads Dried at Room Temperature After a 48-Hour Chloroform Soak

In the previous report, MLM-CF-57-9-41, it was reported that chloroform, tetrachloroethylene, or xylene could remove all rosin residues within one minute without agitation or mechanical scrubbing of a head. It appears that with a short immersion time, not exceeding one minute, both chloroform and tetrachloroethylene are acceptable solvents on the basis of these tests. Either of these solvents is recommended as a replacement for methanol for the following reasons:

1. No amorphous rosin residue is formed by the presence of moisture in the solvent.
2. Rosin is approximately five times as soluble in chloroform or tetrachloroethylene as in methanol.
3. Cleaning time in the solvent will not exceed one minute.
4. No mechanical scrubbing is necessary.
5. Fire hazard from methanol vapors is eliminated.

Metallographic and X-Ray Studies In order to correlate reactions between gold wire and lead-tin-indium solder as observed microscopically with the results of the conductivity and tensile tests, a series of tests are in progress in which samples for metallographic examinations are held at each of the temperature levels used in the other tests. A Pyrex tube filled with solder was held at 181°C in a constant temperature bath, and a length of 20-gauge gold wire was inserted in the molten column. The column was agitated for approximately one minute by shaking the tube while it was still in the constant temperature bath. This was necessary to bring the gold wire to the same temperature as the solder and to cause it to be completely wetted by the solder. The column was rapidly cooled, the glass was broken from the solidified metal, and the rod was sectioned into short lengths. The sections were mounted in bakelite and ground off normal to the axis of the coated wire. After polishing and careful measurement of the diameter of the gold wire, two specimens of each were placed in ovens held at 140°C. The specimens were then reground and polished, and the diameter of the gold wires was remeasured.

The initial treatment of coating the 20-gauge gold wire with an excess of solder was found to decrease the average diameter of the wire from 0.0327 inch to 0.0325 inch. Microscopic examination of the specimens after the first holding period of 24 hours at elevated temperatures revealed evidence of some melting in the samples. The 140°C specimens showed evidence of melting occurring throughout the solder. The 130°C specimens showed only very minor evidence of flow of the solder which indicated that the solidus of the solder may be slightly lower than 130°C. No evidence of solder flow was found in the 120°C and 110°C specimens, although melting at the interface between the gold wire and the solder was observed in both specimens. A lowering of the solidus temperature to below 110°C as a result of the addition of gold was indicated. After approximately 120 hours of thermal treatment, the average diameter of the gold wire was reduced from 0.0325 inch to 0.0316 inch, 0.0317 inch, 0.0319 inch, and 0.0320 inch for the 140°, 130°, 120°, and 110°C specimens, respectively. After approximately 200 hours, diameters of the 120 and 110°C specimens were further reduced to 0.0318 inch and 0.0319 inch. During the first 50 hours of thermal treatment, the gold which was dissolved from the surface of the wire could not be accounted for; no change in the microstructure of the surrounding solder was observed, and no layer of intermetallic material was formed between the wire and the solder. A layer of another material was observed to form after 50 to 75 hours of thermal treatment. When first observed, the layer measured from 0.0001 inch to 0.0002 inch in width. Polished specimens were mirror-like in appearance and were unaffected by the reagent employed for etching the solder. The layer was circular, well-defined, and had distinct edges except when formed at 140°C. The liquid present at temperatures above the solidus of the solder appeared to cause partial dissolution of the unidentified material, leaving the layer irregular in shape and dispersed outward into the solder. In previous tests described in MLM-CF-57-9-41 at temperatures greater than 140°C, the gold dissolved in the solder with no intermetallic layer formation. Extended thermal treatment of the 130°, 120°, and 110°C specimens caused a widening of the layer, and a second layer with a very fine grain structure formed between the gold wire and the original intermetallic layer. In all cases the second layer was narrow in comparison to the width of the original layer. The greatest formation of the intermetallic layers occurred in the 130°C specimen in which the inner layer grew to a width of 0.0008 inch and the outer layer grew to an average width of 0.0020 inch. This specimen has been photographed and will be scraped free of the solder surrounding the outer layer to facilitate examination of the layers by X-ray diffraction.

The rapid reaction rate between gold and solder at the original test temperatures indicated the need for preparation of additional specimens for testing at 100°C and at 90°C. Samples utilizing the 0.0015-inch diameter gold wire will be prepared for testing in the same manner.

Thirteen samples of binary compounds of the gold-indium-lead-tin system have been prepared. X-ray diffraction patterns have been prepared for the following compounds: Au_2Pb, AuSn, $AuSn_2$, $AuSn_4$, $AuIn_2$, AuIn, Au_3In, β-InSn, γ-InSn, and δ-InSn. The last three systems have been described by V. S. Valintiner, Z. Metallkunde 46: 6, 445(1955).

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 After all the detectable leaks on the diffuser were eliminated, the thermocouples and power leads were resoldered to their respective locations on the palladium tubing. A special silver solder with a melting point of approximately 800°C was used for this purpose.

Some preliminary experimentation was done with the diffuser in order to determine some of its operating characteristics and to determine the amount of gas leakage, if any, between the outside and inside of the palladium tube.

The diffuser was operated at 15 volts input, the lowest voltage obtainable from the 75 KW D.C. generator; the current drawn was 200 to 300 amperes, depending upon the cooling efficiency of the gas surrounding the palladium tubing and upon the amount of hydrogen in solution in the palladium. At this power input the tubing glowed bright red (about 800°C), except at the power input junction and at the thermocouple connections; these points were visibly much cooler. The seriousness of this heat leak is indicated by the fact that with the tube glowing red, the thermocouples indicated temperatures in the range of 350°C to 400°C.

The diffuser was permitted to pass tank hydrogen with an absolute pressure of approximately 1000 millimeters of mercury being maintained outside the palladium tubing. The tubing was connected to an evacuated 25-liter tank and the rate of pressure rise in the tank was observed. The pressure difference between the outside and inside of the tube diminished. After 30 minutes the 25-liter tank pressure was 624 millimeters which represented a total passage of approximately 20 liters of gas. During the first five minutes, however, the passage rate was 1.45 liters per minute, which is ample capacity for the gas purification system.

As a check on the effectiveness of the diffuser as a molecular filter, hydrogen containing 37 per cent helium was exposed to the palladium, and mass spectrometric analysis was made of the diffused gas. There was no detectable peak at mass 4, which indicated that the effluent from the diffuser was essentially 100 per cent pure hydrogen. Thus the mechanism of gas passage is diffusion only. A more reliable thermocouple will be installed before further experimentation is attempted.

Loading and Unloading Facility Equipment is being readied for some of the initial recovery of tritium from various gas residues which are to be transferred to Mound Laboratory from the Los Alamos Scientific Laboratory. The high nitrogen content of these residues makes the use of the process line line for the Savannah River Plant by-product gas undesirable for this work. Filling of the first trailer at the Savannah River Plant is expected to begin by the end of October.

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 at special high pressures. The initial process development will include the recovery of tritium from Los Alamos Scientific Laboratory's decayed salt, and from aqueous and gaseous materials.

The following low-temperature still equipment was received from National Bureau of Standards on October 3: distillation column; radiation shield; vacuum jacket; column support; liquid level indicator; transfer line; two hydrogen Dewar probes; three hydrogen cylinders; sample line manifold; Richards valve bonnet; silica gel trap and heater; two flow meters; Variac; and two relief valves. The equipment is being reassembled in room R-120.

Control equipment is being assembled for the operation of the calorimeter to be used in the Hot Gas Facility. The water bath has been constructed and is ready for use. This calorimeter can be used to measure the power output of samples as large as 4.5 inches in diameter and about ten inches long and has a precision of about 200 microwatts with an air-filled thermal gradient interspace.

The three-bay drybox to be used for weighing and transferring salts in a dry argon atmosphere is being designed. A flow sheet of the equipment required to maintain a dry, inert atmosphere is being made. Vendors have been consulted on gas pumps, Lectrodryers and commercially-fabricated equipment for the removal and control of hydrogen and oxygen in the drybox.

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 Additional purification of the 100 liters of neon (98 per cent neon-20) which was accumulated during preparation of enriched neon-22 has been started using the four twelve-foot thermal diffusion columns in series cascade. The cascade was operated for about two days with an operating pressure of 350 millimeters of mercury, and with the four center wires at approximately 900°C to permit the system to come to equilibrium. After equilibrium was established, gas was drawn from the top of the cascade at a rate of approximately 300 milliliters per hour and from the bottom of the cascade at zero to 50 milliliters per hour. This variation of bottom draw-off rate was required to keep the system in balance and to maintain gas purity at the top of the cascade. The effect of bottom draw-off was observed from mass spectrometer analysis of gas samples taken eleven feet above the bottom of the cascade. The bottom draw-off rate was varied to prevent these gas samples from attaining a higher neon-22 content than is present in natural neon. The feed gas is contaminated with about 10 per cent nitrogen, and the composition of the gas in the bottom draw-off is greater than 50 per cent nitrogen. Since the isotopic composition of the neon at the bottom is approximately that of natural neon, no efforts were made to collect the bottom draw-off material. Both top and bottom flows were stopped overnight to avoid badly unbalancing the cascade through some unforeseen event. To date 20 liters of neon containing approximately 99.9 per cent neon-20 have been collected.

A total of 118 neon samples were analyzed on the mass spectrometer this month. This work was done in conjunction with the above experimentation.

Liquid Thermal Diffusion Two additional 72-hour runs have been completed with the D_2O-H_2O system. The first run was inconclusive since the densities of the fractions fell short of the density of the feed. The results of the second run are reported in Table 1. The three top fractions were all less dense than the feed, but of the three bottom fractions, only the very bottom one was more dense than the feed.

Duplicate density determinations have indicated a precision of $\pm$ 0.0001 gram per milliliter. The difficulty in attaining a consistent trend among the fractions from runs with this system may be associated with the rather large vertical temperature gradients. Because of the relatively high thermal conductivity of this system, it was decided to operate the columns with a heater voltage in excess of the rated value in order to maintain a more suitable temperature difference. Variacs have been rewired so that the maximum voltage delivered to the heater is now 121 volts instead of 108 volts. The effect of cooling water rate and power input to the column heater on vertical and horizontal temperature gradients is shown in Table 2. It does not appear likely that further increases in voltage will produce improved horizontal temperature differences without entailing excessive vertical hot wall gradients.

Table 1

SEPARATION OF H_2O AND D_2O BY LIQUID THERMAL DIFFUSION

Fraction Number	Density g/ml (25°C)	
Feed	1.0515	
1 (bottom)	1.0521	
2	1.0512	Avg. = 1.0516
3	1.0515	
4	1.0518	
5	1.0516	Avg. = 1.0517
6	1.0516	
7	1.0510	
8	1.0509	Avg. = 1.0510
9 (top)	1.0510	

Feed: 50 50% by volume; avg cold wall temperature, 13°C; avg hot wall temperature, 50°C; avg temperature difference, 37°C; Run time, 72 hr.

Table 2

EFFECT OF POWER INPUT AND COOLING WATER RATE ON HOT AND COLD WALL TEMPERATURES WITH ORDINARY WATER IN ANNULUS (Column 1)

Power Input Watts	Cooling Water Rate liters/min.	Hot Wall Temperature °C Bottom	Middle	Top	Spread	Cold Wall Temperature °C Bottom	Top	Spread	Mean Horizontal Difference °C
427		18.6	20.3	20.2	1.7	16.5	17.0	0.5	2.9
1201	7.2	24.2	28.2	27.2	4.0	15.5	17.6	2.1	9.9
2023		28.7	34.5	34.6	5.9	18.0	22.0	4.0	12.6
205		18.3	19.5	19.5	1.2	15.9	16.9	1.0	2.7
428	3.6	19.4	22.5	22.6	3.2	16.1	18.0	1.9	4.4
1165		25.4	32.5	32.5	7.1	16.0	20.2	4.2	12.0
2069		29.5	40.0	40.0	16.0	19.0	25.4	6.4	12.5
205		18.0	20.4	20.4	2.4	15.8	17.4	1.6	3.0
428	2.4	19.5	23.9	24.1	4.6	15.8	18.3	2.5	5.4
1165		26.0	36.2	36.2	10.2	16.0	22.6	6.6	13.5
2040		29.4	45.8	46.8	17.4	20.0	26.0	6.0	17.7
205		17.8	21.6	21.7	3.9	16.0	17.8	1.8	3.5
428	1.8	19.4	26.4	26.7	7.3	16.0	19.5	3.5	6.4
1184		25.8	41.9	42.2	16.4	15.9	25.5	9.6	15.9
2069		32.4	58.5	57.8	26.1	15.9	30.6	14.6	26.4

Chemical analysis of the fractions from one of the 24-hour runs (Run 33) on the cerium-yttrium-tributyl phosphate system, which was only partially reported last month, has been completed. The cerium results gave a satisfactory material balance, but those for yttrium were unsatisfactorily low.

In the analytical procedure the TBP fractions were stripped several times with dilute nitric acid, and the pooled strip solutions were divided into two aliquots. The first was used for titration of the cerium, while the metals were precipitated as oxalates from the second aliquot and weighed as oxides, the yttrium content being obtained by difference. Application of this procedure to the analysis of TBP samples containing known amounts of yttrium nitrate and cerium nitrate showed that, as the total amount of solute taken was decreased, the per cent of the yttrium recovered in the analysis continually decreased while the cerium result was unaffected. Re-solution of the ignited oxides and titration of the cerium showed that the yttrium, not the cerium, was failing to extract or precipitate completely. Literature references indicated that our extraction conditions were correct and that the difficulty was attributable to incomplete precipitation of yttrium oxalate. Direct evaporation to dryness of the second aliquot in the presence of oxalic acid was therefore substituted for precipitation and filtration. This procedure had led to improved material balances for yttrium although there is still significant loss which is probably confined to the dilute fractions.

Two 24-hour runs and two 48-hour runs on this system have been completed. The feed was a TBP solution approximately 0.1-molar in cerous nitrate and 0.1-molar in yttrium nitrate. The mean temperature difference was 41°C with the hot wall maintained at a nominal value of 60°C.

No yttrium results are available for the 24-hour runs. However, comparison of the cerium results for these runs with those for the 48-hour runs showed that equilibrium was not attained in 24 hours. Concentration profiles from the duplicate 48-hour runs were in substantial agreement, although the yttrium concentrations were thought to be low.

The metals tended to concentrate toward the bottom of the column. However, this tendency appeared more pronounced for cerium than for yttrium so that the mole ratio of cerium to yttrium, which was equal to unity in the feed, considerably exceeded this value in the bottom fractions at the end of 48 hours. These tentative conclusions are being checked.

Several additional runs were needed on the benzene-carbon tetrachloride system in order to complete a study of the effect of time on separation factor, utilizing a nominal temperature difference of 33°C. Since Column 1, which had furnished the earlier data on this system, had been modified for the D_2O-H_2O system, it was desired to determine the performance of Column 2 with the benzene-carbon tetrachloride system. A four-hour run on Column 2 was made for this purpose and was compared with a similar run previously made on Column 1. The two columns gave nearly the same results, so that extension of the study of the system using Column 2 appeared justified. A five hour run was then made on Column 2 and separations consistent with the time trend obtained with Column 1 were obtained.

A laboratory study of the temperature coefficient of refractive index has been made for solutions containing varying relative amounts of benzene and carbon tetrachloride. Data were needed to convert the earlier separation data to a weight basis since refractive index measurements on the earlier runs were made at varying tap water temperatures. The separation factor, defined as the value of the mole ratio (carbon tetrachloride to benzene) for the bottom fraction divided by the same ratio for the top fraction, has now been computed for a series of runs employing various times. The results will be reported after several additional runs have been completed.

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.*

A shipment containing 43.88 grams of ionium was sent to Oak Ridge National Laboratory on October 4, 1957. Complete analytical data are not yet available but will be forwarded to F. M. Case at Oak Ridge when ready.

The remaining ionium is being processed to fulfill a 50-gram commitment to University of California Radiation Laboratory and to obtain 20 grams for retention at Mound. Approximately 40 grams of ionium is contained in about six liters of aqueous solution. This solution cannot be considered "product" as it contains 1.5 kilograms of uranium-238 and other impurities. The removal of the bulk of the uranium is being attempted by contacting the aqueous solution with small amounts of 40 per cent / 60 per cent TBP-Amsco. These operations are being done by hand.

Organic and aqueous process solutions from the pilot plant are being stripped to consolidate the ionium they contain. It is hoped to recover about 20 grams of ionium from these sources.

Chemically-inert solids of apparently high alpha-activity content are being digested with strong caustic solutions in hopes of freeing the thorium they probably contain.

Approximately 250 liters of a nitric acid solution containing 10.6 kilograms of uranium and 120 grams of thorium have been prepared for shipment to Mallinckrodt Chemical Works. These solutions are in a stand-by condition at present, since it is hoped another kilogram or more of uranium can be obtained from the final processing and clean-up operations.

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 partially-digested titanium slurry described in last month's report was initially processed in two batches. To each 10 gallons of slurry, 3.25 gallons of 66° Baumé sulfuric acid was added, and this mixture was heated to 128°C for approximately one hour. After cooling, 3 gallons of water, 16 gallons of concentrated hydrochloric acid, 800 milliliters of 35 per cent hydrogen peroxide, and 20 gallons of 50 per cent diisobutyl carbinol-Amsco mixture were added. The two phases were mixed for 15 minutes. Eight more gallons of organic and 500 milliliter of hydrogen peroxide were added, and the two phases were mixed for five minutes. After separation, the organic phase was stripped of protactinium by mixing with 2-molar phosphoric acid.

The raffinates from these extractions were further treated with 2.5 gallons of 66° Baumé sulfuric acid, three gallons of concentrated hydrochloric acid, and two gallons of 35 per cent hydrogen peroxide for every 15 gallons of raffinate. The treated raffinate was mixed with organic and small additions of hydrogen perodixe were made until no further extraction was noted. The protactinium was stripped from the organic as previously described. The solids remaining in these raffinates were filtered from most of the liquid and were saved as "first-cycle" product titanium precipitate. The thick slurry had a volume of five gallons and contained approximately 300 milligrams of protactinium.

The protactinium and titanium in the phosphoric acid strip solution were precipitated by adding 16 gallons of concentrated hydrochloric acid and heating to boiling for two hours. The solution was allowed to stand for one week after which the supernate was discarded and the precipitate saved as "second-cycle" product titanium precipitate. The precipitate and residual supernate have a volume of three gallons and contain approximately 260 milligrams of protactinium.

Purification Work is continuing on the preliminary homogenization of the protactinium concentrated during FY 1956. An estimated 400 milligrams of protactinium is now in solution in approximately 400 milliliters of diisobutyl carbinol.

Raffinate Disposal Raffinate is being dumped to the river at a rate of three drums per day. The dumping is done by siphoning over a 24-hour period. The highly acid siphoned raffinate is mixed with additional water over the normal flow to the river to reduce corrosion. The river flow is sufficient to dilute the raffinate to the maximum permissible concentration of 4×10^{-9} microcuries per milliliter (as radium). The river flow used in these calculations is 300 cubic feet per second.

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 for 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.*

Repeated experiments with plutonium (III) carbonate formed by addition of carbon dioxide to precipitated plutonium (III) hydroxide showed an initial formation of a white precipitate which, on heating under a carbon dioxide atmosphere, changed to a yellow, crystalline material. The change occurred between 140°C and 220°C. The insolubility of the yellow crystals in the supernate indicated that the material may be plutonium oxide.

A saturated solution of plutonium (VI) (as ammonium plutonate, 5.7 grams per liter at 24°C in 0.5-molar lithium bicarbonate) was sealed in a quartz capillary tube. On immersing it in a bath at 150°C, a small amount of white precipitate formed, but the solution remained a dark brown color. On recooling, the precipitate went back into solution. Several other tubes of this solution which contained, in addition, 140 to 420 psi of carbon dioxide, immediately produced at 150°C a large amount of a light pink precipitate and a colorless solution. This pink precipitate did not go completely back into solution on cooling.

To eliminate the ammonium ions from these solutions, the preparation of lithium plutonate was attempted. Direct precipitation of lithium plutonate by the addition of lithium hydroxide to a solution of plutonyl sulfate resulted in a very small amount of precipitate after long standing. However, when solid lithium hydroxide was added to an acid solution of plutonyl sulfate, the solution heated to almost boiling, and then cooled in an ice bath, excellent yields were obtained. This material showed a solubility of 0.12 gram of plutonium per liter at 24°C. A saturated solution of lithium plutonate in 0.5-molar lithium becarbonate contained 0.69 gram of plutonium per liter at 24°C. It is considerably less soluble than ammonium plutonate.

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.

Tests were continued on the thermoelectric generator described in last month's report (MLM-CF-57-9-41). In run 1 the air was evacuated from the generator, which was insulated with Santocel. The lower efficiency (0.392 per cent vs 0.457 per cent at the same power level at atmospheric pressure) can be attributed either to a larger increase in radiation losses because of the higher operating temperatures, or to the increase in thermal resistance between the cold junctions and the exterior wall. Conduction losses should have decreased.

The generator was removed from the constant-temperature water bath and was operated at the following three different ambient temperatures, 0°C (run 2), -13.2°C (run 3), and 200°C (run 4). Efficiencies decreased at the higher temperatures to some extent because of increased radiation losses, but this decrease amounted to only five per cent at the 200°C temperature.

A fourth ring having thirty-nine junctions was added to the generator. Figure 1 shows the assembled generator with the heater in place. To obtain directly the hot and cold junction temperatures, thermocouples were added by extending both wires of one cold junction and a single wire of the adjacent hot junction. T_1 and T_2 could then be determined without taking the junctions out of the generator circuit. However, only T_2 could be measured under load conditions. By quickly measuring T_2 after removing a load and obtaining a curve of T_2 versus time on semi-log paper, the temperature under load conditions was obtained for several runs. Because of thermal conduction of the heat away from the junction by the leads, it was thought that the temperatures of these junctions might not be representative of the other junctions. However, T_1-T_2 values for these junctions were within a few per cent of the temperatures obtained from $E_0/161$. Runs 5 through 11 in Table 1 give the results of the tests with 161 junctions and give the values of T_1 and T_2.

In Figure 2 the efficiency of the generator is plotted versus the heater input in watts for both three and four rings. If there were no difference in per cent heat loss, and if the thermal resistances were independent of temperature, the efficiency at the same power would be expected to be 1.33 times greater for three rings than for four rings. The ratio of efficiency between the two generators starts near 1.25 at 2 watts, decreases to 1.13 at 10 watts, and falls to 1.05 at 17 watts.

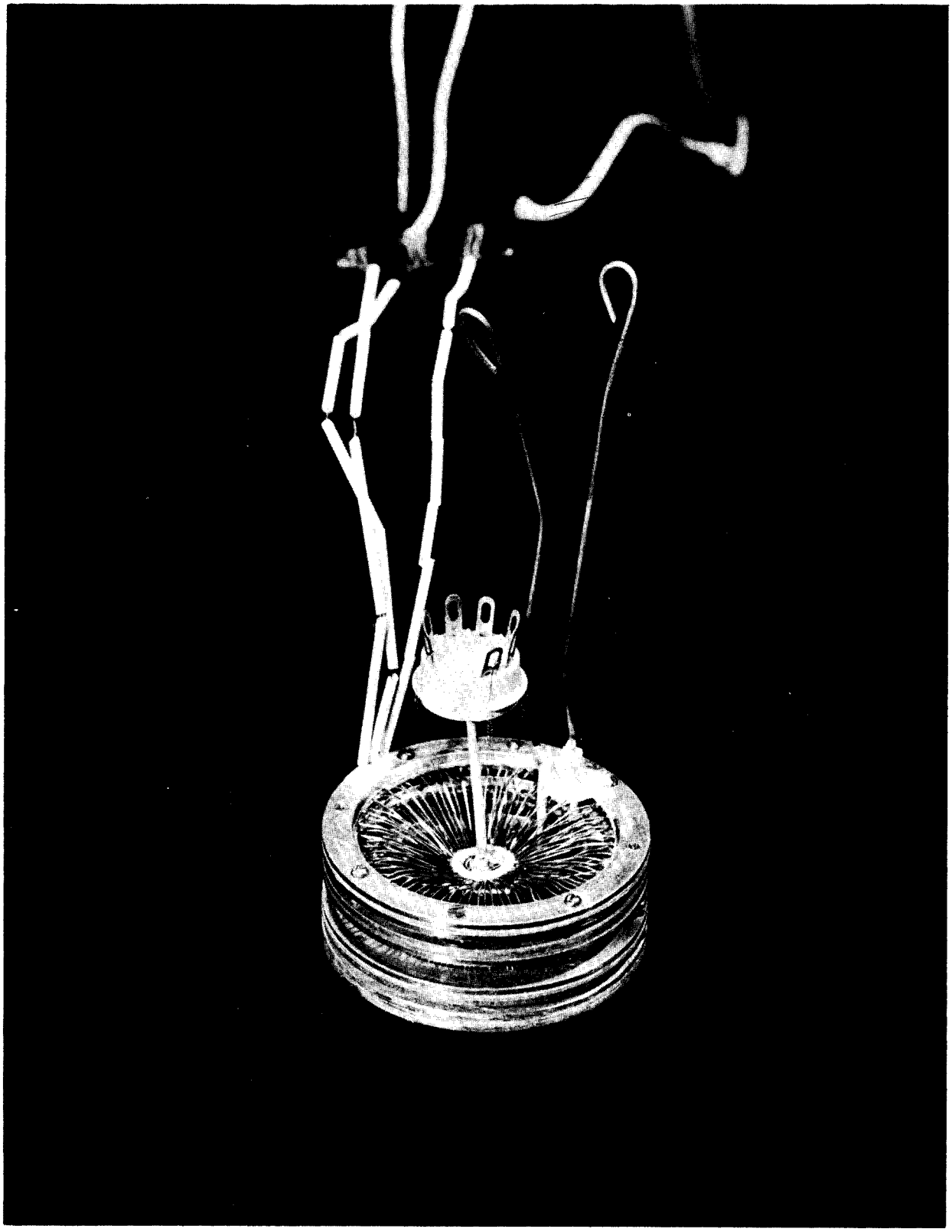

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Figure 1. Thermoelectric Generator No. 2
161 Junctions; Heat Source in Place

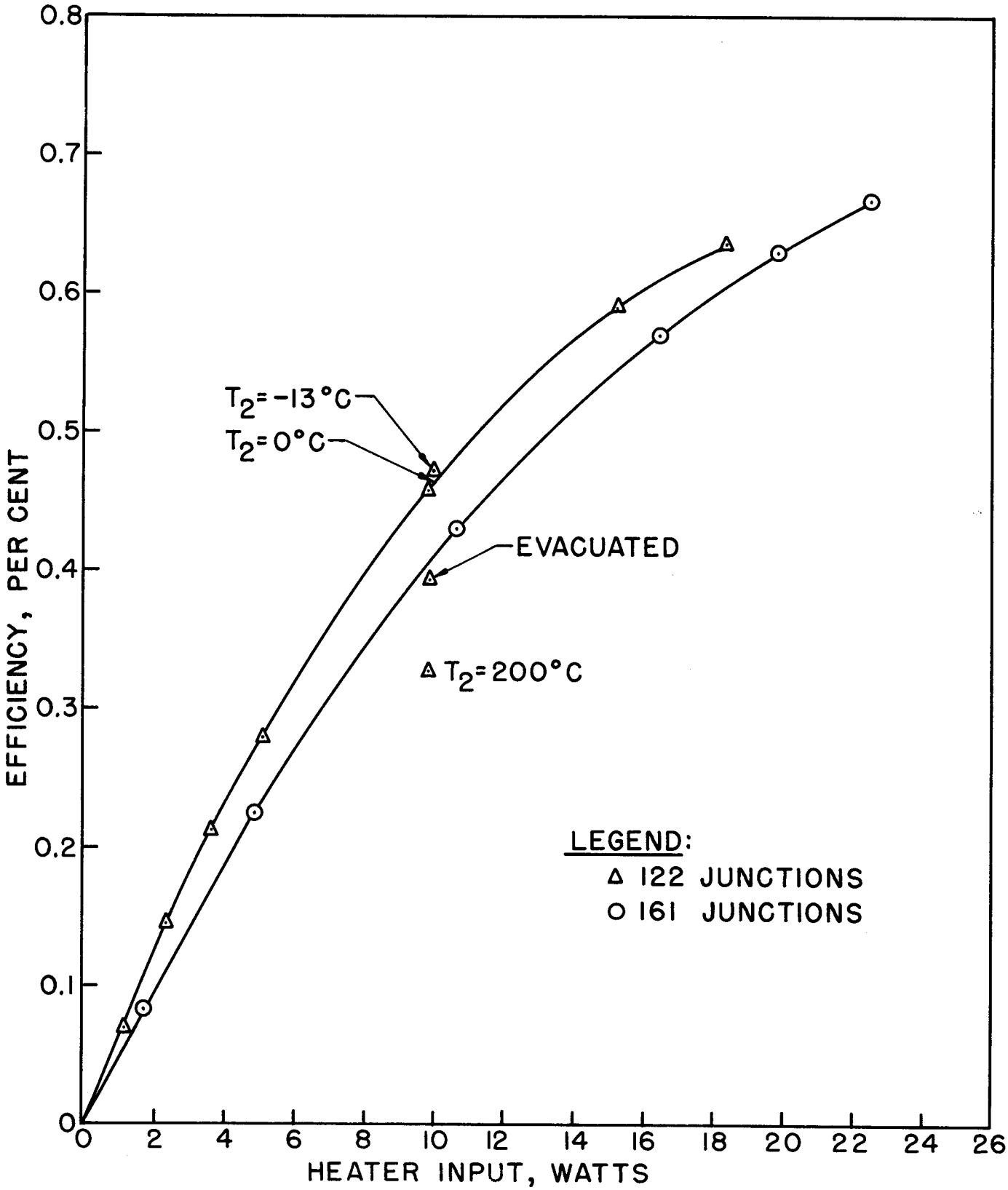

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Figure 2. Efficiency of Generator No. 2 vs Electric Power Input

In Figure 3 the efficiency is plotted versus the temperature difference between the hot and cold junctions. Since the efficiency is greater for four rings than for three rings at all values of $T_1 - T_2$, the per cent loss of heat is less for four rings at all temperatures. From calculations it appears that most of the heat loss occurs from the surface of the wires and not directly from the heater and the surface of the source container. The relative surface areas are three square centimeters for the source container and 56 square centimeters for the wires on the 161-junction generator.

The generators were very stable. No noise could be detected in the fifth decimal place once the load and currents were adjusted.

Table 2 shows the measured parameters as well as some of the calculated parameters for the generator with 161 junctions with a power input of 16.42 watts. The thermal resistance can be considered tentative to the extent of 10 per cent. The magnitude of the thermal resistance from the cold junctions to the bath (about 4°C-watt^{-1}) is larger than expected and deserves more attention in future designs.

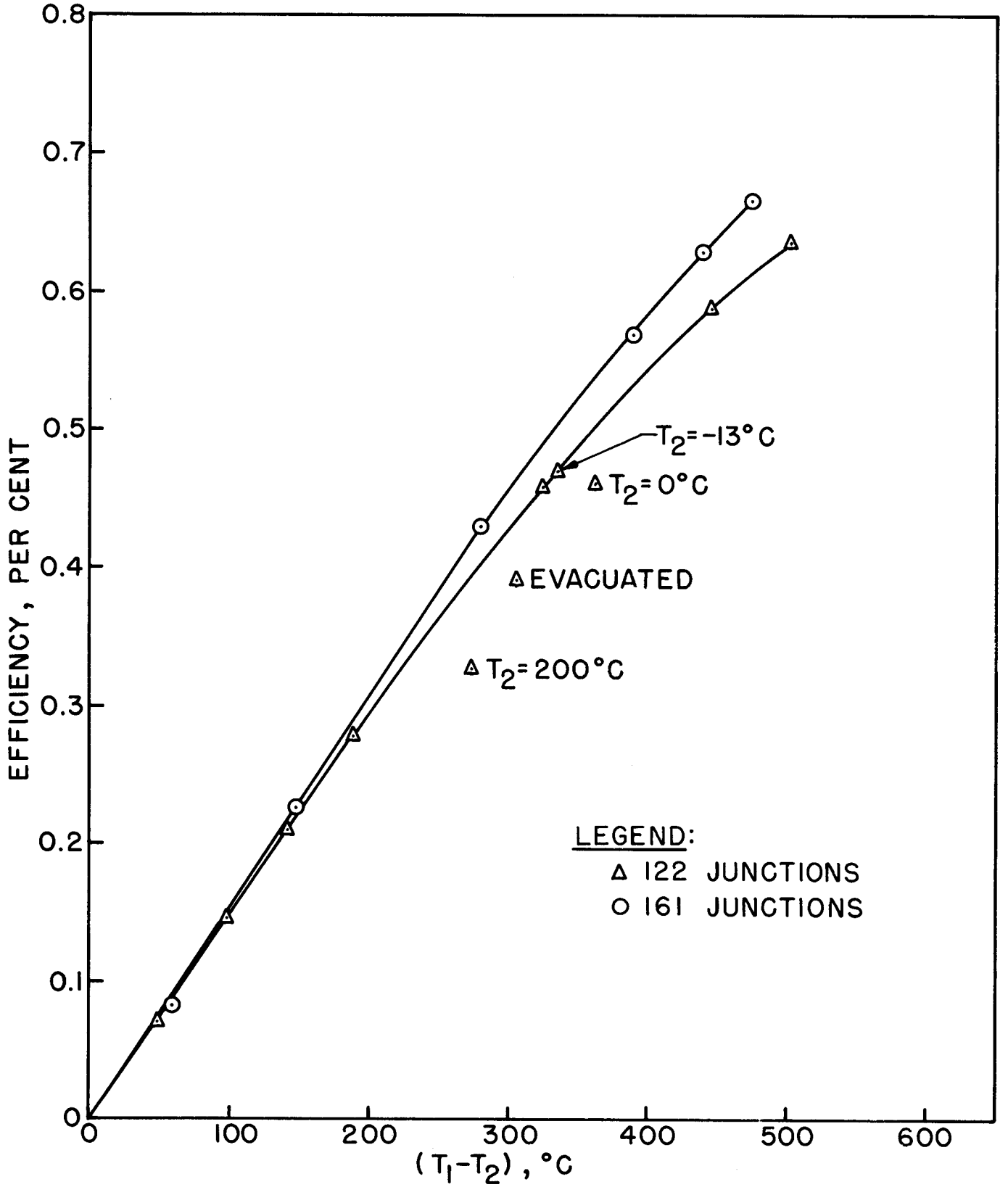

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Figure 3. Efficiency of Generator No. 2 vs Temperature Difference Between Hot and Cold Junctions

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Table 1

PERFORMANCE OF GENERATOR NO. 2

Run	W_s Heater Power watts	T_s Temperature inside heater °C	T_h Hot Junction Temperature °C	T_c Cold Junction Temperature °C	$T_s - T_c$	T_a Ambient Temperature °C	E_o Generator Voltage (No load) volts	W_L Maximum Power To Load milliwatts**	Efficiency %
1	9.856*	832.0			305.1	25.9	2.5841	38.67	0.392
2	9.847	643.5			355.3	0.0	2.7852	45.62	0.463
3	9.851	640.5			330.3	13.2	2.8074	46.43	0.471
4	9.851	727.0			270.3	200	2.3682	32.15	0.326
5	0.0523	34.02	31.73	29.88		29.52	0.016952	0.00135	0.0026
6	1.7337	157.0	94.1	35.9		29.52	0.56379	1.4990	0.0865
7	4.872	340.8	194.1	45.6		29.52	1.5570	11.436	0.2347
8	10.599	595.4	343.0	62.0		29.52	3.1920	45.35	0.4279
9	16.416	779.4	465.6	77.0		29.31	4.5986	93.48	0.5694
10	19.789	854.9	523.5	85.5		29.31	5.3258	124.60	0.6296
11	22.450	911.0	566.5	91.3		29.31	5.8448	149.40	0.6656

* Air evacuated from generator

** Voltage can be approximated by ($E_o/2 \times 0.99$)

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Table 2

CHARACTERISTICS OF GENERATOR NO. 2 WITH 161 JUNCTIONS

W_s (heater input)	= 16.42 watts
T_a (ambient temperature)	= 29.3°C
T_2 (cold junction temperature, no load)	= 77.0°C
T_1 (hot junction temperature, no load)	= 465.6°C
T_s (temperature inside heater, no load)	= 779.0°C
E_o (generator voltage, no load)	= 4.6000 volts
R_i (internal resistance of generator)	= 55.5 ohms
R_L (external load)	= 55.5 ohms
E_L (voltage across external load)	= 2.2778 volts
W_L (power dissipated in external load, E_L^2/R_L)	= 0.09348 watts
L (average length of wire, hot to cold junction)	= 1.90 cm
R_t (thermal resistance, hot to cold junctions, calculated, assuming no loss)	= 33.2°C-watt^{-1}
R_s (thermal resistance to heat losses)	= 82°C-watt^{-1}
(thermal resistance to heat loss by radiation)	= 164°C-watt^{-1}
(thermal resistance to heat loss by conduction)	= 164°C-watt^{-1}
(thermal resistance, cold junctions to bath)	= 4.0°C-watt^{-1}
(thermal resistance, heater to hot junctions)	= 19.1°C-watt^{-1}
f (ratio of R_t/R_s)	= 0.40
f/f+1 (fraction of W_s lost)	= 0.29
β (thermoelectric power, $E_o/n\ (T_1-T_2)$)	= 73.4 μ volts-°C^{-1}
Eff (over-all efficiency, 100 W_L/W_S)	= 0.569 %
Eff(theoretical maximum efficiency, 0.569/(1+f)	= 0.797%