







HIGH EFFICIENCY, LOW POWER THERMOELECTRIC COOLERS



FINAL REPORT CONTRACT NO. DAAK79-78-C9016

MERADCOM PROCUREMENT & PRODUCTION DIRECTORATE FORT BELVOIR, VIRGINIA 22060

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I. \ ABSTRACT

This report describes the work performed on a 15 month development program. The purpose of this program was the development of a high performance, multi-stage thermoelectric (TE) cooler which would operate satisfactorily under extreme military environments. Five coolers each of three types (various heat loads), were designed and fabricated. Detailed environmental and performance tests were performed on these coolers. The electrical tests included measurement of COP/stage and performance under various heat loads on each type of cooler. The effect of adding radiation shielding to each type was also determined. All of the coolers performed satisfactorily after environmental and performance testing. Performance data, predicted from design calculations and computerized material parameter measurements, showed excellent correlation with measured test data.

A TE material development and evaluation program was conducted in conjunction with the cooler design and fabrication effort. P-type TE material was produced which was comparable to that commercially available. Advanced computerized TE material evaluation techniques developed by Marlow Industries were used to evaluate α , ρ , κ , and Z for a particular lot of material toward the end of the program.

- alpha, nho, Kappa,

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II. PURPOSE

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The purpose of this program was the development of a family of ruggedized, high efficiency TE coolers. These coolers were to have low power inputs and various heat load capacities to meet different requirements for the second generation FLIRS. These objectives were to be achieved through improved design techniques, better fabrication methods and/or improved low temperature TE materials.

III. NARRATIVE

A. Cooler Design, Fabrication and Testing

1. Stress Testing of GFE Cooler

In the early phases of the development program, a GFE cooler manufactured in 1975 by Marlow Industries for the Night Vision Laboratory was subjected to a shock and vibration testing sequence. The purpose of these series of tests was to evaluate the stress limitations of the <u>present</u> <u>cooler design and fabrication process</u>. Results obtained were to be used in determining what changes in design and construction might be needed to improve the cooler mechanical environmental performance.

Six GFE coolers were received for use in the test program. These units were performance tested to establish reference data prior to environmental testing. One of the units, S/N IA-O18 was found to be open. Test data for the other five units is shown in Table 1 of the Appendix.

One cooler, S/N IA-034, was selected at random from the GFE units and subjected to the following series of stress tests:

a. The unit was vibration stressed per MIL-STD-810C, method 514.2, over a frequency range from 10 hertz to 3500 hertz. A resonant search sweep was made over the frequency range with each sweep having 20 minutes duration. Nine sweeps were made in the horizontal axis for a total stress period of six hours. During the testing period, an AC milliohmeter was used to monitor an intermittent or permanent change in the cooler resistance. No resonances or changes in the cooler characteristics were noted during the testing and no damage to the cooler was found after test completion.

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The cooler was then restressed in the same manner with an applied force of 50 g's for two hours and, again no change in cooler characteristics or physical damage was noted.

b. The cooler was then shock-tested per MIL-STD-883A, method 2002.1, test condition A (Half sine wave 500 g's, 1 ms duration pulse) with the exception that the number of impacts was six in each of 3 mutually perpendicular axes for a total of 36 impacts. An AC milliohmmeter was used to monitor the cooler resistance during the shock testing. No variation in the cooler resistance or physical damage to the cooler was noted.

The cooler was then shock-tested per MIL-STD-810C, method 516.2, procedure IV with a shock pulse of 125 g's amplitude and 10 ms duration. No damage to the cooler or change in resistance was noted.

The cooler was then shock-tested in the same manner as the first shock test at a level of 3300 g's for a duration of 0.5 millisecond. Two shocks were applied in each direction along each of 3 mutually perpendicular axes for a total of 12 shocks. The above shock level represented the maximum level available from the test equipment. No variation in the cooler resistance or physical damage was detected after the completion of this test. The vendor's Component Evaluation Report is listed in the Appendix. After the completion of the vibration and shock testing, shown in Table 1 the test cooler was given a resistance check and a performance test with the results listed in Table 2. No measureable change was noted in the operating characteristics of the test cooler.

TABLE 1 Shock and Vibration Levels

	SHO)CK		VIBRATION
	AMPLITUDE	DURATION	AMPLITUDE	FREQUENCY RANGE
Specification	500 g's	0.3 ms	2.5 g's	10Hz to 3500Hz
Test	3300 g's	0.5 ms	50 g's	10Hz to 3500Hz

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TÆ	ABLE	2
Cooler	S/N	IA-034

Measurements Before and After Shock and Vibration Stress Testing

Performance Test	Before	After
Voltage (Volts)	6.0	6.0
Current (Amps)	.864	.864
т _н (°с)	26.9	26.4
Tc (°C)	-78.7	-79.1
Resistance Check		
First Stage (Ω)	5.8	6.0
Second Stage (Ω)	2.37	2.38
Third Stage (Ω)	.95	.95
Fourth Stage (Ω)	. 32	. 32

The results of the shock and vibration testing as shown in Table 2 demonstrates that TE coolers which have been manufactured using these materials and fabrication methods can be stressed at levels much higher than that specified in the contract with no degradation in performance or physical damage to the coolers.

The GFE multi-stage coolers, that were furnished for this environmental testing were fabricated from the basic "unitary" design, i.e., with only one ceramic between each stage. No high-stress environmental test data is available for "modular" multi-stage TE coolers which are assembled by cascading individual single-stage cooler units. Thus, in view of the satisfactory test results described above, <u>no change in the basic</u> "unitary" multi-stage cooler design is recommended.

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2. Cooler Designs

2.1 The coolers to be delivered under this program were to have physical and electrical characteristics as shown in Table 3.

	TABLE 3		
Coo	ler Specificatio	on Data <u>Cooler Type</u>	
<u>Characteristic</u>	<u>I - A</u>	<u>I – B</u>	<u>11</u>
Top Stage (in.)	0.20 X 0.20	0.20 X 0.20	0.40 X 0.40
Centering (in.)	0.010	0.010	0.010
Parallelism (in.)	0.005	0.005	0.005
Input Voltage (Volts)	6.00 <u>+</u> 0.01	6.00 <u>+</u> 0.01	6.00 <u>+</u> 0.01
Input Power (watts)	3.5	7.0	10.5
Heat Load (mw)	30	60	150
т _н (°к)	298	298	298
Tc (°K)	193	193	193
Cool-down Time (min.)	2	2	2

2.2 The development of thermoelectric materials with improved low temperature characteristics was undertaken in order to achieve the goal of high cooler efficiencies at temperatures of 193°K. Additionally, the incorporation of radiation shielding and similar techniques together with further cooler design effort was studied to fully exploit these non-power consuming factors.

The characteristics of commercially available TE materials are generally optimized for operation at room temperature (27°C). This fact is illustrated in Figure 1 where the figure of merit (Z) is substantially lower at 193°K than at 300°K. The material characteristics shown in Figure 1 are the average of the n- and p-type parameters. During the materials effort

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portion of the program, substantial quantities of p-type and n-type thermoelectric materials were produced and evaluated. The thermoelectric characteristics of the p-type material were comparable with those of commercially available TE material. However, the TE characteristics of the n-type material, while substantially more uniform with temperature than commercially available TE material, were lower in overall performance. For this reason, the TE elements in the coolers, were processed from commercial material.

2.3 Thermoelectric material ingot Nos. N295 and P298 were used to fabricate the TE coolers which were built and tested for delivery on this program. The parameter values for the TE material which were used in the cooler design calculations were based on published data for the material. As mentioned previously the average TE material parameters for an n-p couple are plotted over the cooler operating temperature range in Figure 1. This data was utilized by the computer program design technology developed at Marlow Industries to obtain the cooler design information. From this, the number of stages, the number of couples per stage, the TE element dimensions and the overall cooler dimensions for each type of cooler were determined. Table 4 lists the Specification data was obtained from the Contract. The Design data was calculated using data published for commercially available material. The Predicted Performance was calculated using material parameters (of commercially available material) measured by Marlow Industries.

It should be noted that the theoretical COP is independent of cooler heat load. The specified COP for the Type I coolers was .0086 which is achievable with commercial material. The specified COP for Type II was .0143 which was not achievable with commercially available materials.

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Consequently, the power consumption for the design and predicted performance values for the Type II cooler was increased somewhat over that given in the specification. This was done in order to achieve the specified cold side temperature defined in the specification.

The design curves for each type of cooler together with the effects of active heat loading are shown in Figures 2 to 4. It should be noted that the design point exceeds the cold side temperature specification for each of the three cooler types using the published data for commercial material.

At the time that the fabrication of the coolers was essentially completed, a capability was developed at Marlow Industries which made possible for the direct measurement of α , ρ , κ and Z over a wide temperature range for samples from TE material ingots. A sample of material, is cut from a wafer located at a representative section of an ingot. End caps containing thermocouples are attached to the ends of the sample. One end cap of the sample is attached to a TE cooler which, in vacuum, is capable of maintaining any desired temperature under heat load from approximately 190°K to 345°K. Through computer derived measurements α , ρ , κ and Z are determined for the number of points required to define these parameters over the above temperature range. This data is curve-fit using a second order polynomial least squares process

Measurements of α , ρ , κ and Z were made on samples from ingot Nos. N295 and P298 and are shown in Tables 2 and 3 in the Appendix. Resultant curve-fit parameters together with extrapolated values from 60°K to 400°K are shown in Tables 4 and 5 in the Appendix.

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The measured TE material parameters over the cooler operating temperature range are plotted in Figures 5 and 6 for p-type and n-type respectively. This data was utilized by the computer design technology to obtain calculated cooler performance data for the TE coolers which had been fabricated under the program. These predicted performance characteristics based on these measurements are listed in Table 4. It can be seen that the measured material parameters in Figures 5 and 6 differ substantially from those used in the design (Figure 1) and the result is the predicted performance is not as good as the design. It will be noted under the section "Discussion of Results" that the cooler performance test data and the predicted performance data calculated from measured parameters agree well within testing error. The predicted performance curves determined from measured material parameters for each type of cooler are shown in Figures 7 to 9. The performance calculations from which the above curves are derived are given in Tables 6-8 in the Appendix.

Two thermocouples were attached to each cooler assembly. A .001" copper-constantan thermocouple was attached to the cold plate and a .003" copper-constantan thermocouple was attached to the cooler base. This type of thermocouple was used rather than that originally specified because of its greater accuracy over the temperature range. The use of .001" wires attached to the cold plate reduced the conductive heat loss to a negligible value as compared to .003" thermocouple wire.

Radiation shields were designed and fabricated for each of the three cooler types. The material used was gold-plated bronze. Drawings of the radiation shields are shown in MI Dwg. Nos. 2798, 2799, and 2800 in the Appendix.

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Bottom Plate		.52. 1.68"			.52" × .68"			. SG * X / 1/	
Height		. 270 "			" OIF .			.673"	
Centering	,010.	.010.		,0/0'	.010.		,010.	, 0/0 .	
Persilelism	.005"	.006		.500	.005		.005"	.900.	
Element, L/A		168.5/in		B1.3 / in.				320/in.	
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The dimensions of the copper test bases are 1-1/2" X 1-3/4" X 1/4". Each test base has 8 insulated standoff terminals. The size of the test base was increased slightly to allow space for the terminals without requiring the power leads to be bent sharply. The thickness of the base was made 1/4" in order to accomodate the terminal mounting studs.

3.0 Cooler Fabrication

Five TE coolers of each of Type I-A, Type I-B and Type II were fabricated in accordance with Marlow Industries standard process specifications. Detailed product specifications for these coolers, which are designated Models SP 1102, SP 1103 and SP 1104 respectively, are shown in the Appendix. No problems were encountered in assembling the coolers.

After assembly, each cooler was mounted onto a copper test base using 96°C solder. The copper test base had eight insulated terminals to accommadate the two power leads, four thermocouple leads and two heat load resistor leads. Four through-holes were provided for mounting the coolertest base assembly on the cooler vacuum test fixture.

A .001" copper-constantan thermocouple was mounted onto the cold plate of each cooler and connected to a pair of copper test base terminals. A .003" copper-constantan thermocouple was mounted onto the copper test base and connected to another pair of terminals. The cooler input power leads were attached to a third pair of terminals. After completion of the vibration and shock tests, a heat load resistor was mounted onto each cooler with 96°C solder. The power leads to the resistor were attached to the fourth pair of terminals. A radiation shield was mounted onto one cooler of each type with 96°C solder.

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## 4.0 Cooler Testing

Each of the cooler-test base assemblies was subjected to performance and environmental testing. Selected units were given additional testing to further evaluate detailed cooler characteristics. Details of the testing operations are given in Operational and Environmental Test Procedure MI Dwg. No. 2898 in the Appendix.

The AC resistance and the Hy-Pot resistance of each cooler was measured and recorded. Each unit was then performance tested in the test console vacuum. The initial test data is given in Table 5.

The units were mounted on an adapter test plate and tested under the following conditions:

Vibration Test

Frequency Range	- 10 - 3000 Hz
Amplitude	- 2.5 g peak
Sweep Rate	<ul> <li>Logarithmic sweep from 10 Hz to 3000 Hz and back to 10 Hz within a 30 minute period</li> </ul>
Axes	- 3 orthogonal, one sweep per minute
Shock Test	
No. of Impacts	- 6 in each direction and amplitude
Axes	- 3 orthogonal, both directions
Amplitudes	<ul> <li>half sine wave pulses, of 500 g's peak value with a minimum duration of 0.3 millisecond measured between the 10% value of peak amplitude. Half sine wave pulses of 140 ± 10 g's peak value with a duration of 9 milliseconds ± 10%.</li> </ul>

A component evaluation report is shown in the Appendix.

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After completion of the vibration and shock testing, the units were tested again. The post test data (Table 6) indicated that two of the Type II units had degraded somewhat in performance. However, the units were cleaned prior to the 85°C bake tests, subjected to the bake cycle and tested again. These test results indicated that the apparent subpar performance was the result of oil on the cooler that was received during the shock and vibration testing. The equipment used to conduct the shock and vibration testing has a considerable amount of oil associated with it and it is believed that the oil got onto the coolers during the handling and/or testing operations.

The component evaluation report is shown in the Appendix. No degradation in operating characteristics occurred during the 48 hour, 85°C bake cycle as shown by the test results in Table 7.

Three coolers of each type were subjected to storage at  $-60^{\circ}$ C for 24 hours. A heat load resistor was mounted on one unit of each type. These units were operated with the normal heat load at  $-40^{\circ}$ C during the cold stress test. No degradation of cooler performance was noted as a result of either of these tests. Test data is given in Tables 8 and 9.

Heat load resistors were mounted on the cold plates of two coolers of each type. Voltage leads and thermocouples were mounted on each stage of one cooler of each type equipped with a heat load resistor. These coolers were operated at nominal voltage and heat load, and data was recorded to determine the power input and temperature for each stage. Similar data was taken for conditions of zero heat load and 200% nominal heat load. The test data and calculations for the COP's for each type of cooler was given in Tables 10-13.

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One cooler of each type with heat load resistor was tested to determine  $\Delta T$  at a nominal voltage and heat loads of zero, nominal, and 200% nominal values. The variation of  $\Delta T$  with voltage at nominal heat load was also determined. Radiation shields were mounted onto one each of the above coolers. One of the coolers, SP 1102-2, was damaged while mounting the radiation shield. This cooler was not repaired since it was felt that the cooler performance might be affected. Instead, a radiation shield was mounted on cooler, SP 1102-1, which had comparable performance characteristics (refer to Table 5) and this unit was used for subsequent testing with the radiation shield. The measurement of  $\Delta T$  at nominal voltage and heat load was repeated for each cooler after installation of the radiation shields. Data for these tests is given in Tables 14-16.

The radiation (passive) heat load listed in Table 2 was calculated using the equation:

 $Q_r = \sigma \in A (T_A^4 - Tc^4)$   $\sigma = 3.549 \times 10^{-11} (watts/in^2 \circ K^4)$   $\epsilon = effective emissivity, 0.8$   $A = surface area (in^2)$   $T_A^= ambient temperature (°K)$ Tc= cold surface temperature (°K)

The final tabulated test data for each cooler is given in Table 17. Thermoelectric Material Development

1.0 Introduction

Β.

The operating range of multi-stage thermoelectric coolers has been extended to temperatures of 193°K and lower. Consequently, a need exists for thermoelectric materials with a high figure of merit, Z, particularly at these lower temperatures. The figure of merit is defined by the equation,  $Z = \alpha^2/\rho\kappa$ , where  $\alpha$  is the Seebeck coefficient,  $\rho$  is the electrical resistivity and  $\kappa$  is the thermal conductivity.

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		·										
			1.00	.053	23.7	296.7	6.7	279.7	30		7	2190
			2.00	011.	23.5	296.5	-18.4	2546			7	253.1
			3.00	.176	23.6	2966	-376	235.4			7	232
			2.00	-249	23.8	296.8	- 52.2	120.8			7	2182
			5.00	.327	24.0	297.0	-61.7	211.3			7	208.4
			6.00	40B	24.2	297.2	- 67.0	2060			3	2020
			2.00	.498	24.3	297.3	- 69.1	2039			7	200.8
			00.0	561	24.6	297.6	- 68.4	2016	-		1	20102
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 In examining the parameters for presently available TE materials, the electrical resistivity ( $\rho$ ) decreases with decreasing temperature while the thermal conductivity ( $\kappa$ ) increases. TE materials can be produced for which the product of  $\rho$  and  $\kappa$  decreases as the temperature is reduced, thus tending to improve the figure of merit. However, the Seebeck coefficient ( $\alpha$ ) invariably decreases with a decrease in temperature for high Z materials. The net effect is a reduction in the figure of merit, particularly at temperatures below about 225°K. The investigation of TE material characteristics at lower temperatures was one of the objectives of this program.

2.0 Process

In the early phases of the program, an extensive literature survey was conducted in the field of thermoelectric materials. Most of the present TE materials are composed of  $Bi_2 Te_3 - Sb_2 Te_3 - Sb_2 Se_3$  alloys in various concentrations with added dopants to form p-type or n-type material. The TE material is composed of crystallographically oriented polycrystals usually in the form of an ingot 10-20 mm in diameter and several centimeters long.

A common method for forming the polycrystalline material is the utilization of the vertical Bridgeman technique. This technique produces the TE material by the slow solidification of a molten ingot of the material over a sharp temperature gradient. Other methods employ zone leveling and sintering techniques but these methods have not been evaluated under the present program.

The detailed Thermoelectric Material Growth Procedure, MI Dwg. No. 2454 is listed in the Appendix. A description of the materials, equipment, processing methods and evaluation techniques follows.

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The raw meterials needed for the process are bismuth, tellurium, antimony, selenium, and the n-type dopant, antimony iodide. High purity material was purchased in shot or powder form. The source and purity of all starting materials is listed in Table 9 of the Appendix. The materials were carefully weighed on a Model 1202 Sartarious balance, to the corresponding stoicometric values required for the particular type of material. The molar ratio of the starting materials for some of the p- and n-type ingots produced under this program are listed in Table 10 and 11 of the Appendix.After the materials have been weighed they are placed in a cone tipped quartz tube.The tube is prepared by washing with hydroflouric acid,rinsing with deionized water, and sealing one end so that the sides of the tube converge at an approximate thirty degree angle. A small diameter tube is used to conserve material and allow for a more orderly formation of the crystals. These quartz tubes are cleaned one at a time and used immediately when dry to minimize exposure to atmosphere and surface contaminants.

The tube containing the raw materials is connected to the vacuum system and evacuated for an hour. The raw material is then compacted by slowly melting the material with a torch. This compacting serves to reduce surface area and thus decrease oxidation. The tube is evacuated for an extended period of time and then sealed with the torch under a vacuum. The vacuum of the sealed tube is checked by visual examination of the tube. The quartz capsule is then placed in the alloy oven and melted again. This time the molten material is gently rocked to help in the mixing process, and increase the homogeneity of the material. It is removed from the oven and allowed to solidify. The material at this point is a mass conglomerate of the pseudo-ternary alloys bismuth telluride, antimony telluride, and antimony selenide.

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The next step in the process involves the Bridgeman Technique. The ingot of material is slowly passed through a two zone resistance furnace.

The first zone the material passes through operates at an elevated temperature which causes the material to become molten. The second zone is substantially cooler therefore, causing a steep temperature gradient for solidification. The cone-shaped end is the first section of the quartz crucible to leave the molten zone and initiate the solidification process. The cone acts as a seed to start the orderly formation of the crystalline structure. The temperatures the ingot experiences are recorded by a Omegaline Model # 0-5237-55 recorder. Two thermocouples at different positions on the ingot allow for dual temperature measurement which are simultaneously measured by the recorder. This generates a full furnace profile on every production run as well as data for thermoelectric material characterization. A typical furnace profile is shown in the TE Material Growth Procedure MI Dwg. No. 2454 in the Appendix.

After removal from the furnace the quartz tube contains the final thermoelectric material. The tube is carefully broken from around the material. The ingot is now ready for characterization. With a Keithly 503 milliohmmeter the resistivity is measured down the length of the ingot at evenly spaced intervals using a special two point probe. This procedure is repeated on three sides of the ingot. An average resistivity is calculated from this data using the equation

$$\rho = \frac{RA}{L}$$

where  $\rho$  is the electrical resistivity, R is the measured resistance, A is the cross-sectional area, and L is the **distance** between the probes of the special two point probe. All data is

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recorded on the electrical resistivity record shown at the back of the Thermoelectric Growth Procedure, Dwg. No. 2454.

The Seebeck coefficient is calculated by using a simple galvanometer, a hot probe, and a cold probe. The measurements are made in much the same manner as the resistivity measurement. Galvanometer readings are taken at evenly spaced intervals down the length of the ingot on three different sides. Calculations use the equation

$$V_{\alpha\alpha} = \alpha \Delta T$$

where  $V_{oc}$  is the open circuit voltage,  $\alpha$  is the Seebeck coefficient, and  $\Delta T$  is the difference in temperature between the hot and cold probes. The calculations depend on the very important assumption that  $\Delta T$  remains constant throughout the test and the use of a standard ingot of material for comparison. The standard ingot of material was an ingot selected from commercially available n- and p-type material. The parameters of the standard ingots were determined by building test coolers and calculating the value of  $\sigma$  from AC resistance and cooler performance measurements. The data is recorded on the Seebeck coefficient record shown in the Material Growth Procedure, Dwg. No. 2454. These two measurements, electrical resistivity and Seebeck coefficient give a quick reference as to the quality of the thermoelectric material.

To further test the material, selected ingots were cut in one inch pieces. Single couple coolers were built from these one inch sections and tested. A typical set of material parameters for a single couple is presented in Table 12 of the Appendix and Figures 1 and 2.

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When a particular set of ingots exhibits good material properties, the one inch sections are set aside for MI 1060 cooler fabrication. A combination of the ingot with good parameters and the commercial ingot are used for cooler construction. The combinations are listed in Table 18.

> TABLE 18 MI 1060 Material Combinations

Cooler	Ρ	N
#1	MI	MI
#2	MI	Std.
#3	Std.	MI
#4	Std.	Std.

A typical set of MI 1060 material parameters derived from test data are given in Figures 3, 4 and 5 of the Appendix. Typical performance of an MI 1060 is shown in Table 13 of the Appendix.

There are two disadvantages associated with this system of material analysis. The process of building special coolers for material evaluation is costly and involves a period of several days. Thus, considerable time is required to generate data by analysis of the test coolers and to feed back this information into the material production process. In addition, this data is based on the cooler performance rather than the basic material parameters and includes production and testing variables. At best, the data reflects the average parameters for the n-type and p-type materials used in constructing the test coolers.

For these reasons two different approaches to material evaluation were considered. Both of these characterization techniques are still under development. One type of analysis is compositional characterization. The electron microprobe was the instrument chosen for the analysis. An adequate

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analysis was not obtained with this technique during initial trials. Verification of the basic chemical components was required to be done at NVL on the samples tested since the values generated by a local vendor did not correspond with the expected values. The results of the initial composition analysis by the local vendor compared with the NVL analysis are shown in Table 19. Later analysis done by a local laboratory using the electron microprobe technique confirmed the measurements made by NVL and expected by Marlow Industries. The analysis from the local laboratory is shown in Tables 20 and 21. Although the quantity of the dopant (Iodine) was not evaluated, the major compositional quantities correlated very closely and the analysis supported our hypothesis of hydrocarbon contamination due to oil backstreaming associated with mechanical pumps. Further work on the development of the methods to measure halogen concentration is desired in order to measure n-type dopant levels. A typical data analysis summary is shown for several samples in Table 14 of the Appendix.

The other type of evaluation required is  $\alpha$ ,  $\rho$ ,  $\kappa$  characterization. The capability to obtain  $\alpha$ ,  $\rho$ ,  $\kappa$  characterization of entire ingots was still being developed at program end. However, samples of thermoelectric material ingots were analyzed over a temperature range of 180°K to 350°K using a computerized characterization method unique to Marlow Industries. These results were extrapolated to cover the range from 60°K to 400°K by computer statisticallized data analysis.

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4.4

## Comparison of Material Analysis using Electron Microprobe

## Atomic Percent

<u>P</u>	-Type Mater	<u>ial</u>		N-Typ	e Material	
	Raw Material	Local Vendor	NVL	Raw Material	Local Vendor	NVL
Bismuth	8.558	52.83	9.62	35.630	54.38	38.87
Tellurium	50.661	43.74	59.05	56.385	42.67	55.83
Antimony	39.738		28.51	1.923		1.40
Selenium	1.014	3.41	2.82	6.046	2.95	3.90

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# P-Type Material Composition (from local laboratory) Electron Microprobe Analysis

Sample	Bi	Те	Sb	<u>Se</u>	
XP015	9.662	58.518	29.848	1.972	
XP022	10.150	59,067	28.750	2.032	
XP024	6.203	58.498	32.772	2.527	
XP027	10.197	58.655	29.257	1.891	
XP028	9.222	61,190	27.711	1.876	

## Molar ratio of final compounds

These values conform to expected results very closely.

# N-Type Material Composition (from local laboratory) Electron Microprobe Analysis

<u>Sample</u>	<u>Bi</u>	Te	Sb	Se
XN008	37.772	49.875	2.012	10.341
XN012	35.384	56.345	4.327	3.944
XN021	35.501	56.983	3.762	3.754
XN022	34.438	57.025	4.570	3.967
XN029	36.312	55.645	4.240	3.803
XNO30B	35.443	56.443	4.229	3.885
XNO3OT	37.525	54.878	3.824	3.773
XN032	35.970	56.567	3.774	3.688

Molar ratio of final compounds

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The computerized material characterization technique produced absolute values for the material parameters as compared to previous systems where standards were required and the data obtained is only relative to these standards. This system of  $\alpha$ ,  $\rho$ ,  $\kappa$  measurement is invaluable to a materials research program. Data generated by this technique is shown in Tables 15-18 in the Appendix and Figures 6 and 7 of the Appendix.

#### IV. DISCUSSION OF RESULTS

#### A. Thermoelectric Coolers

A GFE multi-stage cooler was subjected to severe mechanical stress testing as described in Section III - A. l. of this report. A performance test made after completion of the stress tests showed no measureable change in the operating characteristics of the cooler. Also, no visible damage to the cooler was noted. As a result of this testing, it is felt that the basic "unitary" design configuration, i.e., with only one ceramic between each stage, provides a completely satisfactory cooler that will meet all military environmental requirements.

Three types of coolers were designed and fabricated for use with 30, 60 and 150 mw active heat loads and designated SP 1102, SP 1103 and SP 1104 respectively.

Five coolers of each of the three types were acceptance tested with satisfactory results. All of the coolers were subjected to the shock and vibration testing as called out in the "Operational and Environmental Test Procedure". No performance degradation or mechanical damage resulted from this testing. The Component Evaluation Report is given in the Appendix and

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the test data are listed in Table 2 . All of the units were baked at 85°C for 48 hours. The post-bake test data listed in Table 7 showed no change in operating characteristics.

Three TE coolers of each type were stored at -60°C for over 24 hours. One unit of each type was operated at -40°C under nominal heat load conditions. The test data recorded for the tests is shown in Tables 8 and 9 and indicated all operations were normal.

The Coefficient of Performance calculated for each cooler based on test data is shown in Tables 10-13. A summary of the COP data for nominal heat loading is given in Table 22. Performance curves for the three cooler types are shown in Figure 10.

As discussed previously the overall COP design values for the SP 1102 and SP 1103 type coolers were selected to achieve the goal of reduced power consumptions. Based on published material parameters, it was originally felt that the performance could be achieved using the selected COP value. Later, the actual material parameters were measured using computer techniques. These measured quantities were used to determine the coolers predicted performance and the calculated values for Tc with nominal heat loads are listed in the "predicted performance" column of Table 4. However, the cooler designs had been frozen and fabrication of the test coolers was essentially completed by the time the measurements were made. If the measured material parameter values had been available, a lower design figure for COP would have been chosen in order to meet the 193°K specification.

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Because of the higher COP specification for the SP 1104 type cooler, a somewhat lower design value was used in order to fill the 193°K performance as specified. As above, it was later determined that the measured material parameters, when used to calculate the cooler predicted performance, indicated that the coolers predicted performance would be somewhat lower than that determined from published TE material parameters.

It can be seen that the coolers predicted performance calculations that were based on measured material parameters using the computer technique agree very closely with the measured performance on the test coolers. The comparison is shown in Table 23. The variations are well within those to be expected from manufacturing tolerances and test measurement errors.

A cooler of each type was tested to determine its heat load characteristics and voltage-performance data. The data from these tests is shown in Tables 14 and 15. A radiation shield was mounted onto each of these coolers and the heat load characteristics of the cooler/radiation shield assembly was measured. The data from these tests is given in Table 16.

The measured performance data and the heat load characteristics for the coolers without and with the heat shields are plotted in Figures 11-13. It can be seen that <u>excellent correlation</u> exists between the measured performance data and the predicted performance values determined from computer characterized material parameters and thermoelectric computer modeling techniques.

The measured performance of the SP 1102 and SP 1104 coolers showed an improvement after installation of radiation shielding. However, the improvement was less than anticipated and the SP 1103 cooler showed very little measured performance improvement after the radiation shield was mounted. Only one radiation shield design was investigated.

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# Comparison of Cooler Calculations and Measured Performance

	SPEC.	DESIGN	PREDICTED PERFORMANCE	MEASURED PERFORMANCE
1102-4	193	192.0	202.5	200.9
1103-5	193	191.7	195.9	197.6
1104-5	193	192.5	197.6	196.3



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Although beyond the scope of this program, it is felt that further investigation of various shield designs, measurement of the emissivity of materials and finishes, etc., will result in substantial improvement in performance through the use of radiation shielding.

B. Thermoelectric Materials

A total of 48 ingots of TE material were grown during the course of the program. There were 21 p-type ingots and 27 n-type ingots produced. The evaluation of the p-type TE material by parameter measurements and sample cooler performance indicates that it is comparable to commercially available material. However, the n-type material evaluation based on similar measurements shows that it is of somewhat lower quality in performance.

There were a number of problems which arose during the course of the program. All of these problem areas were resolved or solutions developed during the latter phases of the work.

The initial procedure for evaluating the material parameters consisted of the following steps:

- Measurement of the resistivity at 3 equally spaced radii at intervals along the surface of the ingot and averaging the individual values.
- (2) Measurement of the Seebeck coefficient  $(\alpha)$  with a heated probe and galvanometer. The output of the sample is compared with that of a standard ingot for which  $\alpha$  has been determined.
- (3) Measurement of the characteristics ( $\alpha$ ,  $\rho$  and Z) of a single couple cooler with 1" long elements of n-type and p-type ingots.

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(4) Fabrication of a seven-couple cooler from these materials and determination of  $\alpha$ ,  $\rho$  and Z from cooler performance test data.

With the exception of the resistivity measurements, the techniques described are subject to appreciable measurement error, involve a considerable amount of effort and do not directly measure thermal conductivity ( $\kappa$ ). In addition the resulting time delays prevent quick feedback of information for constructive changes in material processing factors.

Computerized measurement techniques were recently developed at Matlow Industries to provide the simultaneous evaluation of the individual quantities  $\alpha$ ,  $\rho$ , and  $\kappa$  on a TE material sample .15" square and .19" long. These quantities can be measured over a temperature range from 190°K to 340°K. Thus, it is possible to provide the basic information for use in material evaluation studies or TE cooler design within a matter of hours after the material is available. In addition, all parameters including thermal conductivity are measured with an accuracy equal to or greater than the previous methods.

Early in the program, arrangements were made with a local vendor to perform chemical analyses on the TE material ingots. The test results appeared to be subject to considerable error and this assumption was verified by NVL laboratory testing. Later, a local laboratory was used to perform the chemical analyses. The analysis data furnished by the latter laboratory and the data from NVL both agreed very closely with that expected from the material quantities used in preparing the TE material samples.

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The TE material chemical analyses and parameter measurements indicate the following factors contributed to lower material quality, particularly in the n-type.

(1) Evidence was obtained indicating lack of control of the dopant which was present in the ingot. This was indicated by the wide range of resistivity measurements shown in Figure 14.

(2) Contaminants such as carbon and chlorine were detected through the chemical analyses. These degrade both p-type and n-type TE material.

(3) Measurements indicated that the material was non-uniform from one section of an ingot to the other. These factors have been studied and proposed solutions to each problem area have been generated. These solutions will be evaluated in future experimentation.

#### V. CONCLUSIONS

#### A. Thermoelectric Coolers

1. Test thermoelectric coolers have been fabricated using design and assembly procedures developed at Marlow Industries. Tests on these coolers demonstrate their ability to satisfactorily meet military environmental requirements.

2. Using computer techniques developed by Marlow Industries,  $\alpha$ ,  $\rho$ ,  $\kappa$ and Z can now be measured over a temperature range from 190°K to 350°K and will be of major importance on future programs. Extrapolated values of these parameters can be determined over the temperature range from 60°K to 400°K with this technique.

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Figure 14

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3. The material parameter data, as determined in 2. above, can be utilized by the computer program design technology developed at Marlow Industries to determine all essential cooler design information.

4. The results of testing the coolers designed and fabricated on this program show that the correlation between the measured performance data and the coolers predicted performance is well within manufacturing tolerance and testing variances.

5. The use of radiation shielding enhances thermoelectric cooler performance.

6. P-type TE material was produced which is comparable to commercially available material.

7. Computerized techniques have been developed that will measure directly and simultaneously the TE material parameters ( $\alpha$ ,  $\rho$ , and  $\kappa$ ) of a small bulk sample over a wide temperature range.

8. Chemical analyses have confirmed that ingot compositions conform to that of the basic ingredients and that some contaminants were present.

9. Improved processing techniques have been determined that will improve the characteristics of both material types.

#### VI. RECOMMENDATIONS FOR FURTHER DEVELOPMENT

1. Implement the process improvements that will bring the quality of the n-type material up to that of the p-type and will further improve both types of material.

2. Evaluate TE material processing methods which utilize zone leveling techniques and sintered materials.

3. Investigate the properties  $(\alpha, \rho \text{ and } \kappa)$  of selected materials at temperatures below 240°K using the recently developed, computerized parameter measurement techniques described previously. This information will be used to optimize thermoelectric materials specifically for use at low temperatures.

4. Utilize a recently expanded facility which provides room for a laboratory specifically designed for thermoelectric material production.

5. Utilize a transient thermal model program that was developed for designing coolers, determining performance capabilities, and overall analysis of integrated systems variables.

6. Utilize Marlow Industries capability to produce its own patterned ceramics which allows full flexibility to utilize to the fullest extent design and material improvements.

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# APPENDIX

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APPENDIX

Table 1 GFE Cooler Test Data Table 2 TE Material Data, N295 Table 3 TE Material Data, P298 TE Material Curve-Fitted Data, N295 Table 4 Table 5 TE Material Curve-Fitted Data, P298 Performance Calculations, SP 1102 Table 6 Table 7 Performance Calculations, SP 1103 Performance Calculations, SP 1104 Table 8 Table 9 Source and Purity of all Starting Materials Table 10 P-Type Material Analysis Table 11 N-Type Material Analysis Table 12 TE Materials For Single Cooler Table 13 TE Single Couple Cooler Test Data Table 14 Microprobe Analysis of Several Ingots Table 15 TE Material Data, BTXP027 Table 16 TE Material Data, XN008 Table 17 TE Material Curve-Fitted Data, BTXP027 Table 18 TE Material Curve-Fitted Data, XN008 Figure 1 TE Material Characteristics, XN008 Figure 2 TE Material Characteristics, XP027 Figure 3 TE Cooler Performance, No. 2 Figure 4 TE Cooler Performance, No. 3 Figure 5 TE Cooler Performance, No. 4 Figure 6 TE Material Characteristics, BTXP027 Figure 7 TE Material Characteristics, XN008 Shield, MI Dwg. No. 2798 Shield, MI Dwg. No. 2799 Shield, MI Dwg. No. 2800 Product Specification for SP 1102 Product Specification for SP 1103 Product Specification for SP 1104 Operational and Environmental Test Procedure, Dwg. NO. 2898 Component Evaluation Reports (2) Thermoelectric Material Growth Procedure, MI Dwg. No. 2454

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SAMPLE NAME	SER #	FILE # 1 3.0	DATA POINTS 12.12	DATE TESTED 11:08:78/19:44
SO,KO, 2.7922E 3.5086E -3.4896E	RO S1,K1, -06 -1.0324E -02 -1.2231E -05 2.5718E	R1 S2,K2,R2 -06 1.2490E- -04 1.8359E- -06 2.5809E-	2 A,K,R,Z 09 -194.535 07 14.918 09 9.689 2.618	D,C,V 69.2648 -50.9797 -0.1099
TEMP K	ALPHA	KAPPA	RHO	Z
	(V/DEG C)	(W/CM*DEG C)	(OHM/CM)	(/DEG К)
345.24 Lbd	-2.0433E-04	1.4944E-02	1.1565E-03	2.4157E-03
Calc	-2.0478E-04	1.4744E-02	1.1606E-03	2.4508E-03
A %	-0.223	1.357	-0.352	-1.432
331.82 -	-2.0317E-04	1.4619E-02	1.1049E-03	2.5558E-03
	-2.0228E-04	1.4717E-02	1.1026E-03	2.5214E-03
	0.443	-0.668	0.202	1.362
318.41	-1.9792E-04	1.4518E-02	1.0456E-03	2.5806E-03
	-1.9932E-04	1.4756E-02	1.0456E-03	2.5749E-03
	-0.704	-1.615	-0.005	0.221
302.73	-1.9665E-04	1.4963E-02	9.8399E-04	2.6264E-03
	-1.9530E-04	1.4886E-02	9.8018E-04	2.6141E-03
	0.690	0.516	0.389	0.474
288.28	-1.8994E-04	1.4895E-02	9.2069E-04	2.6307E-03
	-1.9105E-04	1.5085E-02	9.2098E-04	2.6271E-03
	-0.579	-1.262	-0.032	0.139
272.28	-1.8740E-04	1.5724E-02	8.5639E-04	2.6080E-03
	-1.8573E-04	1.5396E-02	8.5670E-04	2.6154E-03
	0.900	2.134	-0.036	-0.284
258.04	-1.8162E-04	1.6143E-02	7.9968E-04	2.5552E-0
	-1.8046E-04	1.5751E-02	8.0056E-04	2.5825E-0
	0.645	2.489	-0.109	-1.057
24 3.92	-1.7241E-04	1.5573E-02	7.4655E-04	2.5569E-0
	-1.7473E-04	1.6176E-02	7.4598E-04	2.5301E-0
	-1.327	-3.731	0.077	1.059
227.09	-1.6802E-04	1.7147E-02	6.8194E-04	2.4144E-0:
	-1.6726E-04	1.6780E-02	6.8224E-04	2.4438E-0:
	0.457	2.190	-0.043	-1.203
210.14	-1.5705E-04	1.7271E-02	6.1783E-04	2.3115E-0
	-1.5901E-04	1.7492E-02	6.1952E-04	2.3334E-0
	-1.235	-1.262	-0.273	-0.938
198.57	-1.5551E-04	1.8454E-02	5.7679E-04	2.2720E-0
	-1.5298E-04	1.8039E-02	5.7756E-04	2.2461E-0
	1.656	2.301	-0.133	1.149
192.85	-1.4906E-04	1.8046E-02	5.5889E-04	2.2031E-02
	-1.4987E-04	1.8327E-02	5.5707E-04	2.1999E-03
	-C.537	-1.536	0.327	0.146

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SAMPLE NAME	SER #	FILE #	DATA POINTS	DATE TESTED
P298 BOT	2.1	7.0	12.12	11:13:78/13:56
SO, KO, RO	S1,K1	,R1 S2,K2	,R2 A,K,R	D,C,V   36 67.8562
-2.6875E-0	5 1.12271	E-06 -1.2956	E-09 193.3	
5.5395E-0	27-22321 5 9.78761	E-05-6.9207 E-07 7.5521	E-08 14.0 E-09 10.2 2.5	28 49.1680 287 0.1081 590
TEMP K	ALPHA	KAPPA	RHO	Z
	(V/DEG C)	(W/CM*DEG C	) (OHM/CM	(/DEGK)
342.28	2.0682E-04	1.2929E-02	1.2713E-	03 2.6024 E-03
	2.0562E-04	1.2853E-02	1.2752E-	03 2.5796E-03
	0.583	0.590	-0.307	0.885
328.99	2.0149E-04 2 <del>.</del> 0226E-04	1.3421E-02 1.3196E-02	1.1953E- - 1.1948E- 0.026	03 2.5309E-03 03 2.5947E-03
	L-9799E-04		1.1177E- 1.1180E- -0.029	-2.439 03 - 2.6348E-03 03 2.5991E-03
300.56	L.9184E-04	1.3772E-02	1.0335E-	03 2.5857E-03
	L.9353E-04	1.4011E-02	1.0318E-	03 2.5908E-03
	-0.873	-1.706	0.163	-0.197
286.57	L.8960E-04	1.4416E-02	9.5822E-	04 2.6025E-03
	L.8846E-04	1.4453E-02	9.5606E-	04 2.5705E-03
	0.605	-0.257	0.226	1.245
270.60	L.8287E-04	1.4976E-02	8.7584E-	04 2.5496E-03
	L.8206E-04	1.4991E-02	8.7322E-	04 2.5321E-03
	0.445	-0.096	0.299	0.689
256.35	L.7486E-04	1.5411E-02	8.0454E-	04 2.4660E-03
	L.7579E-04	1.5500E-02	8.0258E-	04 2.4841E-03
	-0.529	-0.574	0.244	0.727
242.02	L.6930E-04	1.6486E-02	7.3246E-	04 2.3736E-03
	L.6896E-04	1.6041E-02	7.3462E-	04 2.4224E-03
	0.201	2.771	-0.295	-2.016
224.99	6318E-04	1.7232E-02	6.5409E-	04 2.3623E-03
	6014E-04	1.6721E-02	6.5789E-	04 2.3313E-03
	1.896	3.057	-0.578	1.333
206.87	.4960E-04	1.7602E-02	5.8023E-	04 2.1912E-03
	.4994E-04	1.7488E-02	5.8107E-	04 2.2123E-03
	.227	0.650	-0.145	-0.953
194.76 1 1	3507E-04 4264E-04 .5.310	1.6406E-02 1.8026E-02 -8.987	5.3510E- 5.3247E- 0.494	04 2.0780E-03 04 2.1197E-03 -1.968
188.97 1 1	.4602E-04 .3902E-04 5.038	1.9805E-02 1.8291E-02 8.277	5.0959E- 5.1004E- -0.088	04 2.1128E-03 04 2.0717E-03 1.986
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2.79226	-06 $-1.0324$	E-06 1.2490	E-09 -194.5	
3.50806	-02 $-1.2231-05$ $2.5719$	E-04 1.8339 E-06 2.5900	E-01 14.9 E-09 0.6	
-3.40305	-03 2.3718		2.6	
TEMP K	ALPHA	KAPPA	RHO	Z
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400.00	-2.1035E-04	1.5538E-02	1.4068E-	-03 2.024 3E-03
390.00	-2.0989E-04	1.5311E-02	1.3607E-	·03 2.1147E-03
380.00	-2.0919E-04	1.5120E-02	1.3151E-	·03 2.2007E-03
370.00	-2.0823E-04	1.4967E-02	1.2700E-	<u>-03</u> 2.2812E-03
360.00	-2.0702E-04	1.4849E-02	1.2254E-	·03 2.3553E-03
350.00	-2.0557E-04	1.4769E-02	1.1814E-	·03 2.4219E-03
340.00	-2.0386E-04	1.4725E-02	1.1379E-	03 2.4803E-03
330.00	-2.0190E-04	1.4718E-02	1.0949E-	·03 2.5297E-03
320.00	-1.9970E-04	1.4748E-02	1.0524E-	·03 2.5695E-03
310.00	-1.9724E-04	1.4814E-02	1.0104E-	·03 2.5991E-03
300 00	-1 94535-04	1 4918F-02	9 68 925-	04 2 61825-03
290.00	-1 9158E-04	1.49100 02	9.27976-	-04 2.01020 03
280.00	-1.8837E-04	1.5234E-02	8,87556-	-04 2.6244E-03
270.00	-1.8492E-04	1.5447E-02	8.4763E-	-04 2.6115E-03
260.00	-1.8121E-04	1.5697E-02	8.0824E-	-04 2.5883E-03
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230.00	-1.720E-04	1.59846-02	7.09305-	-04 2.5550E-03
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230.00		1.000000-02	0.73146-	-04 2.4604E-03
210 00	-1.5390E-04	1.70056-02	6.3301E-	
210.00	-1.30346-04	1.74706-02	0.19006-	.04 5.33525-03
200.00	-1.5374E-04	1.7969E-02	5.8270E-	04 2.2574E-03
190.00	-1.4828E-04	1.8476E-02	5.4691E-	-04 2.1761E-03
180.00	-1.4258E-04	1.9020E-02	5.1165E-	·04 2.0891E-03
170.00	<u>-1.3663E-04</u>	1.9600E-02	4.7689E-	·04 1.9971E-03
160.00	-1.3043E-04	2.0217E-02	4.4266E-	•04 1.9008E-03
150.00	-1.2397E-04	2.0871E-02	4.0894E-	-04 1.8007E-03
140.00	-1.1727E-04	2.1562E-02	3.7574E-	-04 1.6975E-03
130.00	-1.1032E-04	2.2289E-02	3.4305E-	-04 1.5916E-03
120.00	-1.0312E-04	2.3053E-02	3.1088E-	·04 1.4836E-03
110.00	-9.5664E-05	2.3854E-02	2.7923E-	-04 1.3740E-03
100-00	-8.79638-05	2-46925-02	2.4809F-	-04 1.26315-03
90.00	-8,0011E-05	2.5566E=02	2.17475-	-04 1,1514E-03
80.00	-7.1810E-05	2.6477E-02	1.87365-	-04 1,0395E-03
70-00	-6.3359E-05	2.74755-02	1.57776-	-04 9.2777E-04
60.00	-5.4658E-05	2.8409E-02	1.2870E-	·04 8.1709E-04
			2020.00	

TE MoterBI Carre Fitted Gote ق ہ ، کا تے MARLOW INDUSTRIES DATE 12:09:78 SAMPLE NAME DATE TESTED DATA POINTS SER # FILE # ---- -7.0 ----- 12.12 11:13:78/13:56 -P298BOT--2-1--S1,K1,R1 D,C,V SO,KO,RO S2,K2,R2 A ,K ,R ,Z -1-1227E-06 ----1.2956E-09 -2-68-75E-05--193.336 67.8562 -7.2232E-05 6.9207E-08 2.9469E-02 14.028 49.1680 7.5521E-09 9.7876E-07 10.287 0.1081 5.5395E-05 -2.590 ---TEMP K KAPPA ALPHA RHO 7 (V/DEG-C)-- (W/CM*DEG C) (OHM/CM) (/DEG K) 400-00--2-1492E-04 1.1649E-02 1.6552E-03 -2.3954E-03 390.00 2.1392E-04 1.1825E-02 1.5858E-03 2.4405E-03 1.2014E-02 2.4803E-03 380.00 1.5179E-03 2.1267E-04 370.00 1:4514E-03 " -2-11-16E-04 1.2218E-02 2:5145E-03 1.2435E-02 1.3865E-03 2.5431E-03 360.00 2.0939E-04 350.00 2.0736E-04 1.2666E-02_ 1.3231E-03340.00 2.0508E-04 1.2910E-02 1.2612E-03 2.5829E-03 330.00 2.0253E-04 1.3169E-021.2008E-03 2.5939E-03 320.00 1.9973E-04 1.3442E - 021.1419E-03 2.5988E-03 310.00 1.9666E - 041.3728E-02 1.0846E-03 2.5976E-03 1.9334E-04 300.00 1.4028E-021.0287E-03 2.5902E-03 290.00 1.8975E-041.4342E - 029.7437E-04 2.5766E-03 280.00 1.8591E-04 1.4670E-02 9.2154E-04 2.5567E-03 270.00 1.8181E-04 8.7021E-04 1.5012E-02 2.5304E-03 260.00 1.7745E-04 1.5367E-02 8.2040E-04 2.4977E-03 250.00 1.7283E - 041.5736E-02 7.7209E-04 2.4584E-03 2.4126E-03 240.00 1-6795E-04 1.6120E-02 7.2530E-04 230.00 1.6281E - 041.6517E-02 6.8002E - 042.3601E-03

220.00 1.5742E-04 6.3625E-04 1.6928E-02 2.3008E-03 210.00 1-5176E-04 1.7352E-02 5.9398E-04 2.2345E-03 200.00 1.4584E - 041.7791E-02 5.5323E-04 2.1611E-03 2.0804E-03 190.00 1-3967E=04 1.8243E-02 5.1399E-04 180.00 1.3324E - 041.8710 = 024.7626E-041.9922E-03 1.9190E-02 4.4004E-04 170.00 1.2654E-041.8964E-03 160.00 1-1959E-04 1.9684E-02 4.0533E-04 1.7927E-03 150.00 1.1238E - 042.0191E-02 3.7213E-04 1.6809E-03 140.00 1.0491E - 042.0713E-02 .3.4044E-04 1.5609E-03 130.00 9.7183E-05 2.1248E-02 3.1026E-041.4326E-03 120.00 8.9195E-05 2.1798 E-02 2.8160E-C4 1.2961E-03 2.2361E-02 110.00 8.0947E-05 2.5444E-04 1.1517E-03 100.00 7.2441E-05 2.2879E-04 9.9994E-04 2.2938E-02 90.00 6.3676E-05 2.3529E-02 2.0466E-04 ---8.4202E-04 80.00 5.4651E-05 2.4133E-02 1.8203E - 046.7988E-04 70.00 4.5367E-05 2.4752E-02 1.6091E-045.1675E-04 60.00 3.5824E-05 _2.5384E-02 1.4131E - 043.5778E-04

SP02T, DAT (50541, 1)

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PAGE 1

Table 6

P 1102		13-JAN	•79 16159			
TH	TC	Q	I	V	P	COP
298,0	267,1	0,000	0,050	1,000	0,050	0,00000
298,0	281.1	0,030	0.054	1,000	0,054	0,56049
298.0	295.4	0.060	0,057	1,000	0,057	1.05649
298,0	240,9	0.000	0,110	2,000	0,219	0,00000
298,0	254,3	0,030	0,112	2.000	0,225	0,13342
298,0	268.2	0,060	0,116	2.000	0,231	0,25964
298.0	220,4	0.000	0,178	3,000	0,533	0,00000
298.0	233,1	0.030	0,180	3,000	0,541	0,05545
298,0	246,3	0,060	0,183	3,000	0,549	0,10920
298,0	205.5	0,000	0,255	4,000	1,018	0,00000
298.0	217.5	0,030	0,257	4,000	1,027	0,02922
298.0	230,1	0,060	0.259	4,000	1,036	0.05790
298,0	196,0	0,000	0,338	5,000	1,690	0.00000
298,0	207.4	0,030	0,340	5,000	1,698	0.01766
298,0	219,5	0,060	0,342	5,000	1,708	0.03513
298,0	191.4	0,000	0,425	6,000	2,553	0.00000
298,0	202.4	0,030	0,427	6,000	2,560	0.01172
298.0	214,0	0,060	0,429	6,000	2,568	0,02336
298,0	191.4	0,000	0,514	7,000	3,595	0,00000
298,0	202.0	0,030	0,514	7,000	3,600	0.00833
298,0	213.2	0,060	0.515	7,000	3,605	0,01664
298.0	195,3	0.000	0,599	8,000	4,791	0.00000
298,0	205,8	0,030	0,599	8,000	4,793	0,00626
298,0	216,7	0,060	0,599	8,000	4,794	0,01251
298,0	202,6	0,000	0.678	9,000	6,106	0.00000
298.0	213,3	0.030	0,678	9,000	6,103	0,00492
298.0	224.0	0.060	0.678	9,000	6,100	0.00984

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8P03T, DAT (50541, 1]

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Table 7

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1M 200 0	TU 267 A		^0	*	P	CUP
200 0	203.4	0,000	0 106	1,000	0,099	0,0000
200.0	294.2	0.000	0.113	1,000	0 113	0,30075
298.0	235.2	0 000	0.217	2.000	0.434	0.00000
298.0	249.5	0.060	0.223	2.000	0.447	0.13437
298.0	264.1	0.120	0.230	2.000	0.460	0.26079
298.0	213.8	0.000	0.355	3,000	1.064	0.00000
298.0	227.1	0.060	0.360	3.000	1.081	0.05549
298.0	240.8	0.120	0.366	3.000	1.099	0.10916
298.0	198.5	0.000	0.511	4.000	2.044	0.00000
298.0	211.0	0.060	0.516	4.000	2.063	0.02908
298.0	223.9	0.120	0.521	4.000	2.083	0.05760
298.0	188.9	0.000	0.692	5.000	3,409	0.00000
298.0	200.8	0,060	0.685	5.000	3.427	0,01751
298.0	213,0	0,120	0,689	5,000	3,447	0,03481
298,0	184,5	0,000	0,861	6,000	5,167	0.00000
298,0	195,9	0,060	0,864	6,000	5,182	0,01158
298.0	207.6	0,120	0.866	6,000	5,199	0,02308
298,0	_ 184,8	0,000	1,042	7,000	7,292	0.00000
298.0	195,8	0.060	1,043	7.000	7,302	0.00822
298,0	207.1	0,120	1,045	7.000	7,313	0,01641
298,0	189,2	0,000_	1,216	8,000	9,732	0.00000
298.0	200,0	0.060	1,217	8,000	9,734	0.00616
298,0	211,1	0,120	1,21/	8.000	9,737	0.01232
298.0	197.3	0,000	1,379	9,000	12,409	0,00000
298.0	208,1	0,000	1.3/0	9.000	12,401	0.00444
230.0	21791	0,120	1.3/1	9,000	12,393	0.00308
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SP04T, DAT[50541,1]

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PAGE 1

PERFORMANCE CALCULATIONS GENERATED 13-JAN-79 17:25 Thermal model generated 13-JAN-79 17:23 SP 1104 NO Shield 13-JAN-79 17:20

	TH	TC	Q	I	V	P	COP	
	298,0	264,5	0.000	0,241	1,000	0,241	0.00000	
	298,0	279,5	0,150	0,257	1,000	0,257	0,58340	
	298,0	295.1	0,300	0,275	1,000	0,275	1,09142	
	298,0	236,8	0,000	0,527	2,000	1,053	0.00000	
	298.0	250,9	0,150	0,542	2,000	1,094	0,13839	
	298.0	265.8	0,300	0,559	2,000	1,117	0,26852	
	298.0	215,5	0,000	0,860	3,000	2,581	0.00000	
	298,0	228,8	0,150	0,874	3,000	2,622	0.05721	
	298,0	242.7	0,300	0,889	3,000	2,666	0,11251	
	298.0	200,3	0,000	1,238	4.000	4,954	0.00000	
	298.0	212,8	0,150	1,250	4,000	4,999	0.03001	
	298,0	226,0	0,300	1,262	4,000	5,049	0,05942	
	298.0	190.7	0,000	1.651	5,000	8,256	0.00000	
	298,0	202,6	0,150	1,660	5,000	8,299	0,01807	
	298,0	215,1	0,300	1,670	5,000	8,349	0.03593	
	298,0	186,3	0,000	2.084	6,000	12,506	0,00000	
	298.0	197,6	0,150	2,090	6,000	12,543	0.01196	
	298.0	209.7	0,300	2,097	6,000	12,584	0.02384	
	298.0	186,5	0,000	2,521	7.000	17,646	0,00000	
	298,0	197.5	0,150	2,524	7,000	17,670	0.00849	
	298,0	209,1	0,300	2.528	7,000	17,697	0,01695	
	298.0	190.8	0.000	2,943	8,000	23,542	0.00000	
	298.0	201.6	0,150	2,944	8,000	23,550	0.00637	•
	298.0	213.0	0.300	2,945	8,000	23,557	0.01273	
	298,0	198,5	0.000	3,335	9,000	30,017	0.00000	
-	298,0	209.7	0.150	3,333	······································	29,998	0,00500	-
	298.0	220.9	0.300	3,331	9,000	29,982	0,01001	

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13-JAN-79

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## SOURCE AND PURITY OF ALL STARTING RAW MATERIALS

Source:	Kawecki-Berylco Industries										
	P.O. Box 5	67									
	Boyertown,	Pennsy	lvania	19512							
Purity:	Bismuth	5 9's	i.e. 9	9.999							

- Tellurium 59's Antimony 59's Selenium 59's
- Source: Alfa Division Ventron Corporation 16207 So. Carmenita Road Cerritos, California 90701
- Purity: Antimony Iodide 99%

## P-Type Material Analysis

Molar ratio of starting compounds (Calculated from weight data on material growth record)

16

* These values basically conform to the formula:

 $(Sb_2Se_3)_3$   $(Sb_2Te_3)_{72}$   $(Bi_2Te_3)_{25}$ + 3.08 weight %Te

Except sample XPO24, which fits the formula:

 $(Sb_2Se_3)_5$   $(Sb_2Te_3)_{79}$   $(Bi_2Te_3)_{16}$ +10.54 weight %Te

#### N-Type Material Analysis

Molar ratio of starting compounds (Calculated from weight data on material growth record)

Sample	Bi	Те	Sb	Se	Excess Te
XN008	35.630	56.385	6.046	1.923	
XN012	35.994	57.009	3.977	3.088	
XN021	35.905	56.890	4.008	3.001	
XN022	35.948	56.923	3.997	3.033	
XN029	35.912	56.912	4.009	2.982	
XNO30B	35.912	56.901	4.009	2.892	
XN030T	35.912	56.901	4.009	2.982	
XN032	35.968	56.970	4.014	3.049	

These values conform to the formula: (Sb₂Se₃)₅ (Sb₂Te₃)5 (Bi₂Te₃)₉₀

Except sample SN008, the corresponding formula is:

 $(Sb_2Se_3)_{3.3}$   $(Sb_2Te_3)_{12.3}$   $(Bi_2Te_3)_{84.4}$ 

+3.92 weight % Bismuth

0

## TE MATERIAL PARAMETERS FOR SINGLE COUPLE

(Elements: 12mm diameter, 1 inch long)

XP007			
Position	a X 10 ⁻⁴	ρ X 10 ⁻⁴	z x 10 ⁻³
	V/°C	ohm cm	°K-1
I	1.675	19.5	0.959
2	2.050	18.0	1.556
3	1.675	17.0	1.100
4	1.675	17.0	1.100

XN008			
Position	∝ X 10 ⁻⁴ V/°C	ρ X 10 ⁻⁴ ohm cm	z x 10 ⁻³ °K ⁻¹
1	0.775	9.0	0.444
2	1.25	7.5	1.388
3	0.575	7.75	0.284
4	0.250	6.75	0.062

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							┥													Ļ		-		-		-			
(0)	×.	Em T WO	1.365	1.443	1869		1.588	1.682	1.820	1 980	0200	1.768	1.594																
MI DE	2	D-cm/d	7.61	9.696	10.82		2.932	8.712	9.677	2007	20,00	4.06	10.26														-		
(ctal)	, <i>د</i>	Kex o'	1.656	2.051	2.306		1661	1.748	1924	2136	0116	61117	2.167																
10.55	2	2-1203	2.64	2.641	2.630		2.191	2.084	2.045	1 < 11	1010	×-604	2.738																
Joo/er	IL	х.	245.5	261.8	280.5		2.46.5	270	2895	1.1.1		1.407	2,82.8																
and a	27	ŗ	61	68.4	21		53	60	67	202		0.0	80.5																
Ne Cou	72	<b>پ</b>	-58	- 45,4	-31		- 53	58 -	-17	102-	101	- 45.6	- 30.5																
518	3	ů.	£	23	46		0	27	50	11	20		50																
	F	Same	68	2.0	25		6.7	0%	24	21	100	2.3	2.1																
	7	cha	0.64	.85	607.		0.635	134	.87	1.87	00	281	,98																
		Coaler No.	MT 1060 - 2	X POD 8	N Standerd		NI 1060-3	P Standard	X NOOR	15 10/0 1		Merner ( )	N Shandend											-					

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made by Texas Instrumints Incorporated. Electron Mirroprobe Hnalysis

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riuni*			Zdiff		0.715	0.258	7.954	0.642	5.004				lide *													-			· · · ·	!
is Tellu	Inant-	Hinalusis	wt 70		1.191	2,705 -	2.590 -	1.324 -	7.967	-			201 Unon	-														•		 - - - - -
Exce	Raus	platerial	al 44		0.476	2.963	10.544	1,572	2.963				Antin	Raw	Material	w1 %		0.040	0,040	0.634	0.321	0,634	0.634	0.634	0,321					
4m			2 diff.		0.198	0.281	-0.271	0.122	0.125									8.418	0,736	0.753	0,934	2.821	0.903	15210	0.639			•		
Seleniu	Inco+	Eisulanh	Mohr 7.		1.912	2,032	2,527	1.891	9/8/									145.01	3.944	3.754	3.967	3,803	3.885	3.773	3.688					
	Gain	Material	Molar %		1.794	1211	2.748	1,769	1221									1.923	3.008	3,001	3,033	2,982	2.982	238.6	3.049					
nu			2 diff		0.048	-0.350	2,511	-0,136	-1.389									-4,034	0,350	- Di 246	0.573	0,231	0120	-0.185	OPIID-					
Antimo	Ingot.	Inglusis	Moker %		29.848	28.750	32,172	29.257	27.711									21018	4.327	3.762	4.570	4.240	4.229	3.624	3.111					
	haw	nlaterial	Illolar 20		29,800	29,100	30.261	29,393	29.100									94019	3.971	4.008	3.997	4.009	4.007	4.007	4.014					
m			20 diff		0,034	-0.382	-2,568	-0.381	1.241					-				-4.510	199.0-	0.093	0.102	-1.256	-0.458	-21023	-0,403					
elluri	Ingot	Inalysic	Malar 2.		58,518	54.067	58.478	529.85	61.19									49.875	56.345	56.483	520.72	243.22	56.443	54.878	56,567					
	Kaw	Material	Malar 23		58.484	59.449	61,066	59,038	59.449									56.385	57.009	56.590	56.123	156.901	106.12	101 .01	56.910					
h	•		2 diff.		-0,280	0.449	0.329	0.396	H.H.O-									2112	-0.610	-0.404	-1151D	01400	69410-	1.613	D100L					
(Smut	Ingot	Hinalysis	Piolar So		9,662	10,150	6.203	79.97	22201									31.772	35.384	35.501	34.438	36.312	35.443	31.525	25.310					
Ø	Kaw	Waterial	Nohr 20		9.742	2701	11.815	2,801	9.701									35,630	35.494	201.52	35.748	32:912	35.212	31.912						
Sample					X1,015	X1'022	XĽOZY	XĽQ27	X 10.28									XNDES	XNU12	XN021	210NX	10:21	X11630 P	AILesu L	- SERIN			1		

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	:	ARLOW INDUST	RIES Tobb 15	DATE 12:09:78
SAMPLE NAME	SER #	FILE #	DATA POINTS	DATE TESTED
BTXP027	16.0	17.0	12.12	12:01:02:04:41
SO,KO,R -1.4317E- 8.2479E- 5.8306E-	O S1,K1 C4 1.8840E O3 7.9572E O5 4.9752E	,R1  S2,1    2-06  -2.56    2-05  -1.87    2-07  9.23	K2, R2  A,K,    17E-09  191.    44E-07 15.    21E-09  10.    2.	R;Z C,C,V 485 63.9368 250 49.0889 384 0.1067 315
ТЕМР К	ALPHA	KAPPA	RHO	Z
	(V/DEG C)	(W/CM*DEG	C) (OHM/C	M) (/DEG K)
340.33	2.0278E-04	1.3740E-	02 1.2957E	-03 2.3096E-03
	2.0131E-04	1.3619E-	02 1.2970E	-03 2.2945E-03
	0.727	0.890	0.095_	-0.659
329.18	2.0027E-04	1.4342E-0	02 1.2206E	-03 2.2912E-03
	0943E-04	1.4131E=0	021.2224E	-03 2.3024E-03
317.13	1.9470E=04 1.9668E=04 -1.004	1.432 1.4286E-0 1.4632E-0 -2.366	021.1465E 021.1465E 0.1446E 0.170	-03 2.3146E-03 -03 2.3098E-03 0.207
303.35	1.8959E-04	1.4907E-	02 1.0603E	-03 2.2742E-03
	1.9262E-04	1.5138E-	02 1.0588E	-03 2.3149E-03
	-1.570	1.526	0.145	-1.757 -
290.17	1.8970E-04	1.5688E-(	02 9.8122E	-04 2.3377E-03
	_1_8783E-04 _	1.5555E-(	02 9.8003E	-04 2.3142E-03
	0.997	0.855	0.122	1.015
275_22	_1_8143E-04 1.8132E-04 0.065		C2	-04 2.3264E-03 -04 2.3041E-03 0.966
261.54	1.7095E-04	1.5607E-0	02 8.1836E	-04 2.2820E-03
	1.7435E-04	1.6238E-0	02 8.1995E	-04 2.2832E-03
	1.953	-3.887	-0.193	0.213
247.54	1.7315E-04	1.8171E-0	02 7.4659E	-04 2.21C1E-03
	1.6623E-04	1.6460E-0	02 7.4715E	-04 2.2469E-03
	4.165	10.395	-C.075	-1.640
_231.26	1.5532-04	1.7182E-0	02 6.7058E	-04 2.1503E-03
	1.5553E-04	1.6625E-0	02 6.6712E	-04 2.1810E-03
	1.216	3.347	0.518	-1.383
214.63	1.3995E-04	1.5866E-0	C2 5.8635E	-04 2.1053E-03
	1.4319E-04	1.6692E-0	D2 5.9036E	-04 2.0806E-03
	-2.263	-4.948	-C.679	1.186
203.24	1.2911E-04	1.5557E-(	02 5.4200E	-04 1.9769E-C3
	1.3392E-04	1.6678E-(	02 5.4076E	-04 1.9887E-03
	-3.597	-6.722	0.229	-C.595
197.54	1.3422E-04	1.8115E-0	02 5.1729E	-04 1.9226E-03
	1.2904E-04	1.6652E-0	02 5.1684E	-04 1.9347E-03
	4.018	8.782	0.087	-0.624

	•	waanii a						Table 16
- i 9			MARLO	W INDUSTR	IES		DATE	12:09:78
	SAMPLE NAME XN008	SER #	F	ILE # 14.0	DATA	POINTS 2.12	1:	DATE TESTED 17:78/08:14
	50.80.	RO 51.1	(].R]	52.5	2.R2	A.K.1	R.Z	D.C.V
	-5.1643E	-05 -5.251	4E-07	4,999	9E-10	-164	185	48.7533
	3.4656E	-02 -8.840	)4E-05	9.995	9E-08	17.2	131	-43.8167
•	6.9721E	-05 2.144	6E-06	3.257	3E-09 .	10.0	063 564	-0.0950
	TEND V			אססגע		DUO		7
		<u>(V/DEG_C)</u>	()	W/CM*DEG	C)	_ (OHM/CI	u)	(/DEG_K)
					 ?			1 40155-02
	240.11	-1.7241E-04		1.6152E-0	2	1.1759E-	-03	1.4915E-03
*****		0.221		5.641		-0.229		-4.702
	327.83	-1.6754E-04		1.5213E-0	2	1.1266E:	-03	1,6377E-03
,		-1.7006E-04		1.6418E-0	2	1.1228E-	-03	1.5689E-03
		-1.485		-7.336		0.333		4.387
<u>.</u>	315.36	-1.7073E-04		1.7200E-0	2	1.0669E-	-03	1.5885E-03
		-1.6753E-04		1.6718E-0	2	1.0700E-	-03	1.5689E-03
		1.915		2.883	·	-0.292		1.252
	301.36	-1.6290E-04		1.6887 = 0	2	1.0156E-	-03	1-5473E-03
	301.30	-1.6449E-04		1.7093E-0	2	1.0118E-	-03	1.5644E-03
		-0.967		-1.205	-	0.370		-1.094
	288 49	-1 61885-04		1 7604F-0	2	9 5872F-	- 04	1 55278-03
	200.32	-1.6153E-04		1 7472F-0	2 -	9 5950F.	- 04	1.5563E-03
		0.220		0.756		-0.081	01	-0.232
	273 79	-1 5977E-04			· -	9 02008-	- 04	 1 5479E-03
	2/31/3	-1.5794E-04		1.7945E-0	2	9.0105E-	-04	1.5477E-03
		1.159		.1.882 -	-	0.105	•••	0.337
	260.89	-1.5483E-04		1.8661E-0	2	8.4920E	-04	1.5127E-03
		-1.5461E-04		1.8396E-0	2 .	8.5093E-	-04	1.5272E-03
		0.136		1.439	-	-0.204		-0.948
	247.63	=1.5036E-04		1.8710E-0	2	8.0008E-	- 04	1.5104E-03
		-1.5102E-04		1.8894E-0	2 8	8.0C [1E-	- 04	1.5079E-03
		-0.436		-0.978	•	-0.054		0.162
	231.87	-1.4438E-04		1.9031E-0	2	7.4251E	- 04	1.47525-03
	232.07	-1.4652E-04		1.9532E-0	2 ·	7.4211E-	-04	1 48125-03
		-1.461		-2.565	-	0.055	•••	-0.400
	216.10	-1.4264E-04		2.05555-0	2	6.8440E-	- 04	1.4463E-03
		-1.4178E-04		2.0220E-0	2 1	6.8530E-	- 04	1.4506E-03
		9.606		1.655	-	-0.132	••	-0.301
		-1.38535-04		2.1577E-0	2	6.5081E	-04	1.36875-03
	•	-1.3854E-04		2.06965-0	2	6.4907E-	- 04	1.4289E-03
		0.067		4.256		0.268		-4.211
		-1.3722E-04		2.0206E-0	2	6.3039E-	- 04	1.4782E-03
		-1.36835-04		2.0942E-0	2	6.3119E-	-04	1.4175E-03
		0.245		-3.515		-0.128		4.294

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I			MARLOW INDUSTRI	ES	DATE 12:09:78
	<b>.</b>				
· <b>Ì</b>	SAMPLE NAME	SER #	FILE #	DATA_POINTS	DATE_TESTED_
	BTXP027	16.0	17.0	12.12	12:01:02:04:41
	SO, KO,	RO 51,K	1,R1S2,K2	<u>, R2</u> A,K,	R,ZD,C,V
	-1.4317E	-04 1.884	0E-06 -2.5617	E-09 191.	485 63.9368
<b>V</b>	8.2479E	-03 7 <b>.</b> 957	2E-05 -1.8744	E-07 15.	250 <b>49.0889</b>
	<u>5.8306</u> E	-05 4.975	2 <u>E-07</u> 9.2321	E-0910.	3840.1067
1				2.	315
	TEMP K	ALPHA	КАРРА	RHO	Z
i		(V/DEG C)	(W/CM*DEG C	(OHM/C	M) (/DEG K)
•					
	400.00	2.0057E-04	1.0087E-02	1.7344E	-03 2.2994E-03
1	390.00	2.0197E-04	1.0772E-02	1.6565E	-03 2.2859E-03
		2.0285E-04	<u>1.1419E-02</u>	<u>1.5805E</u>	<u>-U3</u> 2.2799E-03
	370.00	2.0322E-04	1.2029E-02	1.5063E	2.2/93E-03
ļ	360.00	2.0308E-04	1.26022-02	1.43398	2.2824E-03
	350.00	2.0243E-04	1.3137E-02	1.3634E	-03 2.2879E-03
	340.00	2.0127E-04	1.3635E-02	1.2947E	-03 2.2947E-03
	330.00	<u>1.9959E-04</u>	1.4095E-02	<u>1.2279</u> E	-032.3018E=03
	320.00	1.9740E-04	1.4517E-02	1.1629E	-03 2.3082E-03
	310.00	1.9470E-04	1.4903E-02	1.0997E	2.3130E-03
	300.00	1.9149E-04	1.5250E-02	1.0384E	-03 2.3153E-03
	290.00	1.8775E-04	1.5560E-02	9.7900E	-04 2.31425-03
<b></b>	280.00	1.8352E-04	1.5833E-02	9.2141E	-04 _ 2.3086E-03
	270.00	1.7877E-04	1.6068 E-02	8.6565E	-04 2.2976E-03
	260.00	1.7351E-04	1.6266E-02	8.1175E	2.2800E-03
	250.00	1.6773E-04	1.6426E-02	7.5969E	-04 2.2545E-03
	240.00	1.6144E-04	1.6549E-02	7.0948E	-04 2.2199E-03
	230_00	1.5464E-04	1.6634E-02	6.6111E	-04 2.17465-03
	220.00	1.4733E-04	1.6682E-02	6.1459E	-04 2.11725-03
	210.00	1.3951E-04	1.6692E-02	5.6992E	-04 2.0458E-03
	200.00	1.3117E-04	1,6665E-02	5.27095	-04 1.95875-03
	190.00	1.2232E-04	1.6600E-02	4.86115	-04 1.8541E-03
	180.00	1.12962-04	1.6498E-02	4.4698E	-04 1.7302E-03
	170.00	1.03082-04	1.6358E-02	4.0969E	-04 1.5855E-03
	160.00	9.2696E-05	1.6181E-02	3.7425E	-04 1.4189E-03
-	150.00	8.1797F-05	1.59665-07	3.40665	-04 1 23015-03
	140.00	7.0386E-05	1.5714E-02	3.0891E	-04 1.0206F-03
	130.00	5.8462E-05	1.5425E-02	2.7901E	-04 7_94185-04
	120.00	4.6026E-05	1.5097 5-02	2.5095E	-04 5.5913E-04
	110.00	3.3078E-05	1.4733E-02	2.2474E	-04 3.3045E-04
	100.00	1.9617E-05	1.4331E-02	2.0038E	-04 1.3402E-04
	90.00	5.6443E-06	1.3891E-02	1.7786E	-04 1.2894E-05
	80.00	-8.8409E-06	1.3414E-02	1.5719E	-04 3.7068E-05
	70.00	-2.3839E-05	1.2899E-02	1.3837D	-04 3.1838E-04
	60.00	-3.9348E-05	1.2347E-02	1.2139E	-04 1.0330E-03

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		MA	RLOW INDUSTRIE	S	DAT	E 12:09:78
SAMPLI	E NAME	SER #	FILE #	DATA	POINTS	DATE TESTED
——————————————————————————————————————	N008	- 13:0		]	1-2-12	17:78/08:14
	CO XO DO	<u> </u>	1 63 23	<b>D1</b> ·		
<b>-</b> _	SU,KU,RO	SI,KI,R		KZ	A,K,K,Z	D,C,V
	3.4656E-02	-8 8404E-	05 9,99595	2-10 2-08	17.131	-43-8167
	6.9721E-05	2.1446E-	06 3.25731	C-09	10.063	-0.0950
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TEM	PK	ALPHA	KAPPA		RHU	
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	-00	-81-70E-04			-1-4487E-03-	
390.	.00 -1	.8040E-04	1.5382E-02		1.4016E-03	1.5095E-03
. 380.	.00 -1	.7900E-04	1.5497E-02		1.3550E-03	1.5258E-03
	-00 <u>-</u> 1-	-/-/49E-04			1 26295-03-	1 55075-03
	.00 -1	./3092-04	1.57852-02		1,20396-03	1.33072-03
	-00	<del>.7419E-04</del>			-1.2194E-03-	
340.	.00 -1	.7239E-04	1.6154E-02		1.1754E-03	1.5651E-03
330.	.00 -1	.7049E-04	1.6368E-02		1.1322E-03	1.5685E-03
	-00	.5849E-04	1.6603E-02-		1:0895E-03	1.5693E-03
310.	-1 -1	.6539E-04	1.685/E-02		1.04/66-03	1.56//8-03
	-00	.64-18E-04			1.0063E-03	1.5637E-03
290	.00 -1	.6188E-04	1.7426E-02		9.6559E-04	1.5575E-03
280.	.00 -1	.5948E-04	1.7740E-02		9.2558E-04	1.5490E-03
270.	.001	.5698E-04			8-8622E-04	1.5385E-03
250.	.00 -1	.54388-04	1.8428E-02		8.4/51E-04	1.5260E-03
250	-00	-5168E-04			8.0945E-04	1.5116E-03
240	.00 -1	.4888E-04	1.9197E-02		7.7205E-04	1.4955E-03
230	.00 -1	.4598E-04	1.9611E-02		7.3529E-04	1.4777E-03
220-	-00	-4297E-04	2.0045E-02		6.9919E-04	- 1.4585E-03
210	.00 -1	.3987E-04	2.0500E-02		6.6373E-04	1.4379E-03
200	.00 -1	3667E-04	2:0974E-02		6-2893E-04-	1.4160E-03
190.	.00 -1	.33372-04	2.1468E-02		5.9478E-04	1.3930E-03
130.	.00 -1	.2997E-04	2.1982E-02		5.6129E-04	1.3690E-03
170.	:00 -1	.2647E-C4	2.2516E-02		5.2844E-04	1.3442E-03
150.	.00 -1	.2287E-04	2.3071E-02		4.9624E-04	1.3186E-03
150	00 -1	19165-04	2 36455-02		A 6470F-04	- 1-2924F-03
140.	.00 -1	.1536E-04	2.4239E-02		4.3381E-04	1.2657E-03
130	.00 -1	.1145E-04	2.4853E-02		4.0357E-04	1.2387E-03
120.	.00 -1	.0746E-C4	2.5487E-02		3.7398E-04	1.2115E-03
110.	.00 -1	.0336E-04	2.6141E-02		3.4504E-04	1.1844E-03
100	.1009	91575-05	2.68155-02		3.16758-04	1 15765-03
90.	.00 -9	.4855E-05	2.7509E-02		2.8912E - 04	1.1313E-03
80.	.00 -9	.0454E-05	2.8224 E-02		2.6214E-04	1.1059E-03
70	.00 -3	.59532-05	2.8958E-02		2.3580E-04	1.0819E-03
60.	.00 -3	.1351E-05	2.9712E-02		2.1012E-04	1.0601E-03

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Figura 1

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Figure 2

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	-05	-04	-03	-02	-01	ITEM	PART NUMBER	DESCRIPTION
					$\geq$		2761.01	COOLER, SPILOZ
<b>J</b>							8791 01	Acoustic BACE
<b>}</b> −−−+−							217 -01	CERAMIC, BASE
1						2	2772-01	LERAMIC, 22
4						4	2765-01	CERAMIC ATH
}						5	2766-01	AEPAMIC, TOP
					211	4	1181-02	TAR
1					14	7	1826-02	TAB , SPECIAL
					106	B	2789-01	ELEMENT P'
					106	9	2789-02	ELEMENT 'N'
					AR	10	1260-07	SOLDER, 138°C
					AR	11	1781-01	FLUX, ELEM ASSY
*					AR	12	1781-06	FLUX, TINNING
					AR	13	1774-03	WIRE, BUSS, 26 GA
					1	14	2336-02	MATRIX, IST STG
						15	2337-02	MATRIX, 220 STG
					1	16	2338-02	MATRIX, 3 RD STG
L						17	2793-02	MATRIX, 4TH STG
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ΔΤ TEST I V TH TC A.C. RESIS HI-POT TES	: = = T: ST:						SIZE CODE IDENT W A 55688 SCALE: N/A	SP 1102 2761 NEV. SHEET 2 OF 4





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4	-05	-04	-03	- 02	-01	ITEM	PART NUMBER	DESCRIPTION
					$\sim$		2762-01	COOLER, SP 1103
1								
"					1		1225-01	CERAMIC, BASE
					1	2	1228-01	CERAMIC, 2ND
						3	2764-01	CERAMIC, 3RD
						4	2765-01	CERAMIC, 4TH
						5	2766-01	CERAMIC, TOP
• •					211	6	1181-02	TAB
					14	7	1826-02	TAB, SPECIAL
					106	8	1990-01	ELEMENT \P'
·					106	9	1990-02	ELEMENT 'N'
					AR	10	1260-07	SOLDER, 138°C
					AR	11	1781-01	FLUX, ELEM ASSY
· •					AR	12	1781-06	FLUX, TINNING
					AR	13	1774-03	WIRE, BUSS, 26 GA
					1	14	1825-02	MATRIX, IST STG
·					1	15	1828-02	MATRIX, 200 STG
					1	16	1828-02	MATRIX, 300 STG
						17	2795-02	MATRIX, 4TH STG

# NOTES:

I. FABRICATE PER ASSY. PROCESS 1823

2. TEST PER TEST PROC. 1486 REF TEST SPEC.

3. INSPECT PER WORKMANSHIP STD'S. 1023







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SIZE	CODE IDENT #			2762
SCAL	NONE	REV :	A	SHEET: 40F4




			Ν	ΛΑΤ	ERIA	AL	LIST	
	-05	-04	-03	- 02	-01	ITEM	PART NUMBER	DESCRIPTION
					$\ge$		2763-01	COOLER, SPIIO4
						1	1126-01	CERAMIC, BASE
1					1	2	1127-01.	CERAMIC, ZND
					1	3	2776-01	CERAMIC, 3RD
						4	2777-01	CERAMIC, 4 TH
1						5	2778-01	CERAMIC, TOP
					215	6	1009-02	TAB
					12	7	1111-02	TAB, SPECIAL
					106	8	2790-01	ELEMENT 'P'
ſ					106	9	2790-02	ELEMENT
					AR	10	1260-07	SOLDER, 138°C
!					AR	11	1781-01	FLUX, ELEM ASSY
Ĺ					AR	12	1781-06	FLUX, TINNING
					AZ	13	1774-04	WIRE, BUSS 26 GA.
						14	2796-02	MATRIX
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# NOTES:

I. FABRICATE PER ASSY. PROCESS 1823

1. TEST PER TEST PROC. 1486 REF TEST SPEC.

3. INSPECT PER WORKMANSHIP STD'S. 1023





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# OPERATIONAL AND ENVIRONMENTAL TEST PROCEDURE

SP 1102, SP 1103, SP 1104

- 1.0 The purposes of the test procedure are to determine the internal and external performance characteristics of the three types of coolers designed and fabricated under this program and to conduct environmental testing of these coolers which were designed for operation in rugged environmental conditions.
- 2.0 Electrical Performance Testing
- 2.1 Mounting Cooler(s)
- 2.1.1 Mount the cooler(s) to be tested on a copper test base using 96°C solder (MI Dwg. No. 1260-05) and Alpha 200L flux (MI Dwg. No. 1781-06).
- 2.1.2 Attach the power leads to the test base terminals using 138°C solder,MI Dwg. No. 1260-07 and Alpha 200L flux MI Dwg. No. 1781-06.
- 2.1.3 Mount a .001" Cu-constantan thermocouple on the top surface of the cooler using the solder and flux referenced in Section 2.1 above. Attach the thermocouple leads to the test terminals using the same materials.
- 2.1.4 Mount a .003" Cu-constantan thermocouple on the test base adjacent to the cooler base and attach the leads to the test base terminals using the same materials referenced in Section 2.1.2.
- 2.1.5 Thoroughly rinse the cooler-test base (CTB) assembly in hot water (60°C) and dry with compressed air.
- 2.2 AC Resistance/Hy-Pot Testing
- 2.2.1 Measure the AC Resistance of the cooler and record the reading on the traveler.

- 2.2.2 Measure the Hy-Pot resistance from the leads to the test base (>100 megohms at 500 volts).
- 2.3 Thermal Performance Test
- 2.3.1 Mount the cooler-test base (CTB) assembly(s) on a cooler test jig per MI Dwg. No. 2248 and connect the power and thermocouple leads to the CTB terminals.
- 2.3.2 Mount the cooler test jig on the test console and mount the heat exchanger on the cooler test jig.
- 2.3.3 Connect the power thermocouple electrical plug, turn ON the vacuum system and set CTB temperature at  $25 + 2^{\circ}$ C.
- 2.3.4 Turn ON power to cooler and set the input voltage of the first test cooler to  $6.00 \pm .01$  volts.
- 2.3.5 Allow the cooler cold plate temperature to stabilize and record the following data on the traveler:
  - a. Serial number of cooler assembly
  - b. Base temperature  $(25 \pm 2^{\circ}C)$
  - c. Cold side temperature
  - d. Input voltage
  - e. Input current
  - f. Vacuum (<10-4 torr)
- 2.3.6 Repeat steps 2.3.4 and 2.3.5 for all cooler assemblies on test jig.
- 2.3.7 Turn OFF power supply, allow vacuum chamber to reach atmosphere pressure, disconnect electrical plug and remove cooler test base (CTB) assemblies from test jig.
- 3.0 VIBRATION AND SHOCK TESTS
- 3.1 Mount ehe CTB assemblies on the shock and vibration test adapter plate.
- 3.2 Bolt the adapter plate to the vibration test stand.

- 3.3 Attach an acceleration sensor to the adapter plate.
- 3.4 **Perform the vibration test under the following conditions:** 
  - Frequency Range 10 300 Hz
  - Amplitude 2.5g peak
  - Sweep Rate Logarithmic sweep from 10 Hz to 3000Hz and back to 10 Hz within a 30-minute period
  - Axes 3 orthogonal, one sweep per minute
- 3.5 Remove the adapter plate from the vibration test stand and bolt to the shock test stand.
- 3.6 Perform the mechanical shock test under the following conditions

No. of impacts	-	6 in each direction and amplitude
Axes	-	3 orthogonal, both directions
Amplitudes	-	half sine wave pulses, of 500 g's peak value with a duration of 1.0 millisecond measured between the $10\%$ values of peak amplitude. Half sine wave pulses of $140 \pm 10$ g's peak value with a duration of 9 milliseconds $\pm 10\%$ .

- 3.7 Remove the adapter plate from the vibration test stand and remove the CTB assemblies from the adapter plate.
- 4.0 THERMAL PERFORMANCE TEST
- 4.1 Repeat Section 2.3 above.
- 5.0 HIGH TEMPERATURE TESTS
- 5.1 Place the CTB assemblies in an oven with the temperature stabilized at 85°C and allow to remain for 48 hours.
- 5.2 Remove from the oven and repeat Section 2.3 for each cooler.

# 6.0 LOW TEMPERATURE TEST

- 6.1 Select one CTB assembly of each type and mount the assembly in a test vacuum chamber, ST 1018 Modified.
- 6.2 Connect power and thermocouple leads to the CTB assembly.
- 6.3 Coat the O-ring gasket for the test vacuum chamber with a thin layer of vacuum grease, install in annular groove and mount the cover on the test vacuum chamber.
- 6.4 Place the test vacuum chamber in a cold chamber, exhaust the system and back fill with argon.
- 6.5 Stabilize the cold chamber at -60°C and allow to remain for 24 hours.
- 6.6 Raise the cold chamber temperature to -40°C and evacuate the test chamber.
- 6.7 Turn ON the power supplies to the cooler and adjust for proper values.
- 6.8 As soon as operating conditions stabilize, read and record  $T_{H}$ , Tc,  $I_{n}$ , and V.
- 6.9 Turn OFF power and vacuum and remove test vacuum chamber from cold chamber.
  6.10 Remove CTB assembly(s) from test vacuum chamber(s).
- 7.0 TEMPERATURE SHOCK TESTS
- 7.1 Stabilize oven temperature at 70°C and cold chamber temperature at -60°C.
- 7.2 Place CTB assemblies in cold chamber and allow to remain for four hours.
- 7.3 Remove assemblies from cold chamber, place in oven within five minutes and allow to remain for four hours.
- 7.4 Remove assemblies from oven and place in cold chamber within five minutes.
- 7.5 Repeat steps 7.2 and 7.3 for a total of three cycles.
- 7.6 Remove CTB assemblies from oven and repeat Section 2.3 for each cooler.

## 8.0 TE COOLER TESTS

8.1 COP Tests

- 8.1.1 Take two CTB assemblies of each type of cooler and mount a heat load resistor on the cold plate of each cooler.
- 8.1.2 Mount a .001" Cu-constantan thermocouple on a metallized pad on each of the intermediate ceramics of one cooler of each type and attach voltage measurement leads to each stage of the cooler.
- 8.1.3 Mount one of the CTB assemblies from Section 8.1.2 on a vacuum test fixture and connect the power, voltage and thermocouple leads from the CTB terminals to the test fixture feed throughs.
- 8.1.4 Attach the heat exchanger to the vacuum test fixture and mount the assembly on the test console.
- 8.1.5 Connect the power, voltage and thermoccuple leads from the test fixture to the test equipment and attach the temperature controller water lines to the heat exchanger.
- 8.1.6 Tunr ON the vacuum system and set the CTB base temperature at  $25 \pm 2^{\circ}$ C.
- 8.1.7 Set the cooler power voltage at  $6.00 \pm .01$  volts and the resistor heat load at the nominal value.
- 8.1.8 Turn ON the power supply and allow the cooler cold plate temperature to stabilize.
- 8.1.9 Measure and record the following data:
  - a. Base temperature
  - b. Temperature for each intermediate stage
  - c. Cold side temperature
  - d. Input Voltage
  - e. Voltage for each stage
  - f. Input current
  - g. Heat load

8.1.10	Set the resistor heat load at zero and repeat Sections 8.8 and 8.9.
8.1.11	Set the resistor heat load to 200% of nominal value and repeat Sections
	8.8 and 8.9.
8.1.12	Turn OFF power supplies, disconnect leads, remove vacuum and remove
	test fixture from test console.
8.1.13	Repeat Sections 8.1.3 to 8.1.12 for each of the other two cooler types.
8.2	Cooler characteristics with heat load
8.2.1	Mount a CTB assembly with heat load resistor on a vacuum test fixture
	and connect the power and thermocouple leads from the CTB terminals to
	the test fixture feed throughs
922	Attach the beat exchanges to the vacuum test fixture and mount the assembly
0.2.2	Actach the heat exchanger to the vacuum test fixture and mount the assembly
	on the test console.
8.2.3	Connect the power and thermocouple leads from the test fixture to the
	test equipment and attach the temperature controller water lines to the
	heat exchanger.
8.2.4	Turn ON the vacuum system and set the CTB base temperature at 25 $\pm$ 2°C.
8.2.5	Set the cooler power supply voltage at 6.00 $\pm$ .01 volts and resistor heat
	load at nominal value.
8.2.6	Turn ON power supply and record time for cold plate temperature to reach
	193°K.
8.2.7	Allow the cooler cold plate temperature to stabilize and record the
	following data on the traveler.
	a. Base temperature
	b. Cold side temperature
	c. Input voltage
	d. Input current
	e. Heat load
	f. Vacuum (<10 ⁻⁴ torr)

- 8.2.8 Set the resistor heat load at zero and repeat Section 8.9.
- 8.2.9 Set the resistor heat load to 200% of nominal value and repeat Section 8.9.
- 8.2.10 Set the resistor heat load at the nominal value and set the cooler input voltage to  $1 \pm .01$  volt and repeat Section 8.9.
- 8.2.11 Increase the cooler volts in steps of 1 volts, repeating Section 8.9 at each step until the cooler cold plate temperature increases with higher voltage.
- 8.2.12 Turn OFF power supplies, allow vacuum chamber to reach atmosphere pressure, disconnect electrical connections and remove cooler test base assembly from test jig.
- 8.2.13 Repeat Section 8.2.1 to 8.2.12 for each of the other two cooler test base assemblies.
- 8.3 Cooler performance with radiation shield and heat load.
- 8.3.1 Install the proper radiation shield on each of the test coolers used in Section 8.2.
- 8.3.2 Perform the tests listed in Sections 8.2.1 to 8.2.8 and 8.2.12 on each of the three cooler types with the radiation shield installed.
- 8.4 Calculate radiation heat load per unit area vs. cold surface temperature.
- 9.0 INSPECTION (Q.C.)

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9.1 Inspect per MI Workmanship Standards, Dwg. No. 1023 and the cooler Product Specification.

	COMPONENT REPO	EVALUATION ORT	XXX Crair Rapids, Iowa Dallas, Texas Newport Beach. Calif. Toronto, Ontario COLLINS PART NOS.
DATE RECEIVED	DATE COMPLETED	PURPOSE OF TEST	None REV
REPORT WRITTEN BY		ENGINEERING INFORMATIO	DN TR NO.
A. C. Wallier	walk	MANUFACTURER QUALIFIC	ATION ET #805028
B. T. Ward	Ballitan	ADDITIONAL SOURCE	INCE PART NAME COOler Assy.
EST REQUESTED BY	provide dates	X OTHER Contract	MFG'R & CODE IDENT NO.
Marlow, Ind.			Marlow, Ind.
One_(1)		P0#3309	MFG'RS PART NO.
NFGR DATE CODE		EP 26, 2014	- INK.
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A <b>PPR</b> OVAL STATUS			
APPROVAL STATUS	DN ENGIN EERING	EERING INFO	REFERENCE:
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ORD OF TEST	TEST PERFORMED	NO IN TEST	NO OF DISC	REMARKS OR TYPE OF DISCREPANCY
1.	Vibration: As per MIL-STD-810C; Method 514.2, except frequ- ency range is 10 to 3500 hertz with a constant 2.5G input. Scanning speed is up and back down in the above mentioned frequency range in twenty (20) minutes. Nine sweeps in each of two axes, (vertical and major horz), for a total of eighteen (18) sweeps or six (6) hours.	]	0	
1.1	Vibration: As in Test No. 1, except input is 0.10 inc. DA or 50G's (which ever is the lesser), for a total of six (6) sweeps or two (2) hours.	T	0	

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ORD OF EST	TRIT PERFORMED	NO IN TEST	NO OF DISC	REMARKS OR TYPE OF DISCREPANCY
2.2	Shock: As in Test No.2, except number of impacts is two (2) in each direction of three (3) mutually perpen- dicular axes for a total of twelve (12). See shock photo under remarks for impact level and pulse duration.			MARLOW INC. 4-28-75

CO	MPONENT E REPO	VALUA RT	TION	Cedar Rapids, Iowa XX Dallas, Texas — Newport Beach, Calif. — Toronto, Ontario COLLINS PART NOS.
DATE RECEIVED	DATE COMPLETED	PURPO	SE OF TEST	None REV
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B. T. Ward	sal Ward	ADDITION	AL SOURCE	COOLER COOLER
Marlow Ind		CONTRACT NO		Marlow Ind.
15 (5 ea. PN)		PO # 43	00	MFG'RS PART NO.
NONE		36-20	14	SP 1102/1103/1104
1 2	Vibration Shock		15	
APPROVAL STATUS		<u> </u>		
APPROVED DIS	AGINEERING	RING INFO	PENDING	PO # 4300
CAL STATUS			IDEP	
REMARKS				EQUIPMENT CHECKOUT: APPROVED
4				
				QUALITY INSPECTION ACCEPTABLE Q.A. ENGR SIGNATURE
COMP. APPL. ENGINEER			DATE	QUALITY INSPECTION ACCEPTABLE Q.A. ENGR SIGNATURE

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REPORT NO _ET #901013_____



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### Drawing No. 2454

## THERMOELECTRIC MATERIAL GROWTH PROCEDURE

## 1.0 Scope

The following describes the procedure to be followed to produce thermoelectric material in the form of rods 12.5 millimeters in diameter.

# 2.0 References

- 2.1 The following process, tests, and control documents are included in this specification by reference. Drawing 1454 - Wafers - Procedure for sawing Drawing 1455 - Elements - Procedure for dicing Drawing MI 1060 - Product Specification Drawing 1486 - Procedure for testing thermoelectric coolers Drawing 1110 - Diffusion Barrier - Procedure for coating thermoelectric material.
- 3.0 Procedures
- 3.1 Cleaning of quartz tubes.
- 3.1.1 Caution: Gloves must be worn when using hydrofloric acid
- 3.1.2 Prepare a cleaning solution of 10% hydrofloric acid and deionized water by volume, and carefully pour the solution in the squirt bottle labeled 10% HF.
- 3.1.3 While rotating the quartz tube, pour a portion of the 10% HF through the tube for several revolutions.
- 3.1.4 Rinse the tube for approximately 2 minutes with deionized water.

- 3.1.4 Dry the tube with the HG 501 Heater.
- 3.2 Preparing quartz tube.
- 3.2.1 <u>Caution</u>: Protective dark glasses must be worn when using the torch.
- 3.2.2 Fasten hydrogen-oxygen torch to bench. Light and adjust flame. Light hydrogen first, then add oxygen as needed.
- 3.2.3 Heat the center of the quartz tube by rotating the tube over the flame.
- 3.2.4 As the quartz gets soft, simultaneously twist and gently pull the two sections of the tube apart so that there are two tubes with sealed ends.
- 3.2.5 With the torch, form each tube so that the walls converge to form a  $30^{\circ}$  angle at the tip.
- 3.2.6 Allow tube to cool to room temperature.
- 3.3 Measure material and place in tube.
- 3.3.1 Select a sequential ingot serial number, starting with XPOOO for p-type material or XNOOO for n-type material, and all material growth data or comments or traveler 2454-01.
- 3.3.2 The amount of each raw material, in grams, required for each type of material is listed below.*

	P-Type	N-Type
Bismuth	15.70	48.47
Tellurium	55.79	46.86
Antimony	27.44	3.14
Selenium	1.04	1.53

*Typical Composition

- 3.3.3 Turn ON the Model 1202 Sartonies Balance and turn OFF the laminar flow vent hood. Use weighing paper during all weighing operations.
- 3.3.4 Attach the quartz tube to the tube stand and fit the special funnel on the top of tube.

- 3.3.5 The raw material should be added to the tube in the order; dopant, selenium, antimony, tellurium, and then bismuth.
- 3.3.6 Weigh out the pre-specified amount of the raw material and then carefully transfer the material to the quartz tube. Record all required data on traveler 2454-01.
- 3.4 Evacuate tube and seal.
- 3.4.1 Remove tube from weighing station and attach tube to the vacuum station with 0-ring and compression seal.
- 3.4.2 Evacuate tube.
- 3.4.3 Close off vacuum valve after thirty minutes.
- 3.4.4 With the torch, carefully melt the contents of the tube to compact the material into the bottom of the tube.
- 3.4.5 After the material has solidified, re-evacuate the tube for at least 30 min.
- 3.4.6 When the tube of material is cool enough to hold with the hand, close vacuum valve and use the torch to seal the tube about 2 inches above the top of the material. Seal the tube in the same manner as in step 3.2.4.
- 3.4.7 After sealing, inspect the tube and material for cracks or abnormalities. Record all required data on traveler 2454-01.
- 3.5 Alloy the Ingot.
- 3.5.1 Set the rocker furnace temperature at 625°C and set the rocker speed at 15-20 cycles per minute.
- 3.5.2 When the oven is at equilibrium, place the tube in the oven and allow to rock for 2 hours. Record all data on traveler 2454-01.
- 3.5.3 Turn OFF the rocker motor and quickly remove the ingot from the oven. Lay the tube on the asbestos sheet in the horizontal position.
- 3.6 Grow Crystal.

3.6.1	Place the sealed ingot in the ingot holder and attach the ingot holder
	to the crystal drive arm.
3.6.2	Set the furnace temperature so that it is just above the hot zone of the
	furnace.
3.6.3	Lower the ingot into the furnace so that it is just above the hot zone
	of the furnace.
3.6.4	Place covers around the top of the furnace.
3.6.5	With the motor control switch at 28* on the dial, place the directional
	switch in the down position.
3.6.6	Check all thermocouple connections for proper connections with the chart
	recorder.
3.6.7	Let the ingot slowly drop through the furnace undisturbed until the
	temperature of the ingot is below 500°C. This will take approximately
	48 hours.
3.6.8	Record all required data on traveler 2454-01.
3.6.8 3.7	Record all required data on traveler 2454-01. Remove ingot and record weight.
3.6.8 3.7 3.7.1	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove
3.6.8 3.7 3.7.1	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace.
3.6.8 3.7 3.7.1 3.7.2	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace. Remove ingot from tube by breaking glass with pliers.
3.6.8 3.7 3.7.1 3.7.2 3.7.3	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace. Remove ingot from tube by breaking glass with pliers. Weigh the ingot on the balance and record data on traveler 2454-01.
3.6.8 3.7 3.7.1 3.7.2 3.7.3 3.8	<pre>Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace. Remove ingot from tube by breaking glass with pliers. Weigh the ingot on the balance and record data on traveler 2454-01. Calculate Resistivity (p) and Seebeck Coefficient (a).</pre>
3.6.8 3.7 3.7.1 3.7.2 3.7.3 3.8 3.8.1	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace. Remove ingot from tube by breaking glass with pliers. Weigh the ingot on the balance and record data on traveler 2454-01. Calculate Resistivity (p) and Seebeck Coefficient (a). Attach the alligator clips to the current leads of the Keithly 503 HC
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3.6.8 3.7 3.7.1 3.7.2 3.7.3 3.8 3.8.1 3.8.2	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace. Remove ingot from tube by breaking glass with pliers. Weigh the ingot on the balance and record data on traveler 2454-01. Calculate Resistivity (p) and Seebeck Coefficient (α). Attach the alligator clips to the current leads of the Keithly 503 HC milliohmeter to the ends of the ingot. Attach voltage leads to the special two point probe. Measure the resistance at half inch intervals down the length of the
3.6.8 3.7 3.7.1 3.7.2 3.7.3 3.8 3.8.1 3.8.2	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace. Remove ingot from tube by breaking glass with pliers. Weigh the ingot on the balance and record data on traveler 2454-01. Calculate Resistivity (p) and Seebeck Coefficient (α). Attach the alligator clips to the current leads of the Keithly 503 HC milliohmeter to the ends of the ingot. Attach voltage leads to the special two point probe. Measure the resistance at half inch intervals down the length of the ingot. Roll the ingot 120° and repeat step 3.8.2.
3.6.8 3.7 3.7.1 3.7.2 3.7.3 3.8 3.8.1 3.8.2	Record all required data on traveler 2454-01. Remove ingot and record weight. Remove ingot by detaching ingot holder from crystal drive arm and remove holder from furnace. Remove ingot from tube by breaking glass with pliers. Weigh the ingot on the balance and record data on traveler 2454-01. Calculate Resistivity (p) and Seebeck Coefficient ( $\alpha$ ). Attach the alligator clips to the current leads of the Keithly 503 HC milliohmeter to the ends of the ingot. Attach voltage leads to the special two point probe. Measure the resistance at half inch intervals down the length of the ingot. Roll the ingot 120° and repeat step 3.8.2.

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3.8.3 Roll the ingot another 120° and repeat step 3.8.2.

3.8.4 Calculate the average electrical resistivity by the equation:

 $\rho = RA/L$ 

where

 $\boldsymbol{\rho}$  is the electrical resistivity

R is the measured resistance

A is the cross sectional area

L is the distance between the probes

Record all data on traveler 2454-02.

3.8.5 With the galvanometer, check and record the value of the deflection at half inch intervals down the length of the ingot. Calibrate the galvanometer with a standard piece of p or n type thermoelectric material.

α (std p) = 220 Micro volts/°C

 $\alpha$  (std n) = 200 Micro volts/°C

galvanometer reading (standard) = galvanometer reading

220 Micro volts/°C  $\alpha$  (sample)

- 3.8.6 Roll the ingot 120° and repeat step 3.8.5.
- 3.8.7 Roll the ingot another 120° and repeat step 3.8.5.
- 3.8.8 Calculate the average Seebeck Coefficient for the ingot and record all data on traveler 2454-03.
- 3.9 Select and cut ingot for single couple test cooler.
- 3.9.1 Send two ingots, one p-type and one n-type, to the saw room to be cut in 1 inch long 12 millimeter sections.
- 3.10 Build two element coolers using these sections.
- 3.10.1 Tin the ends of each section with 138°C solder, 1260-07 and zinc chloride flux 1781-01.
- 3.10.2 Solder the n-type section to the right side of the special two element cooler ceramic.

- 3.10.3 Solder the p-type section to the left side of the special two element cooler ceramic.
- 3.10.4 Solder the special top copper strip across the top of the two elements.
- 3.10.5 Solder a red lead wire to the n-type side of ceramic.
- 3.10.6 Solder a black lead wire to the p-type side of the ceramic.
- 3.11 Attach the current leads of the Keithly AC Milliohmeter to the leads of the cooler.
- 3.11.2 With the voltage leads, measure and record the resistance of each element of the cooler. Clip the voltage leads to the bottom and top tabs close to the element to be measured. Record the data on traveler 2454-04.
- 3.12 Test cooler for cooling effect.
- 3.12.1 Attach the current leads of the test station to the red and black leads of the cooler and apply maximum current to cooler.
- 3.12.2 Attach a thermocouple to the top of the test cooler and measure cooling effect. Record all data on traveler 2454-04.
- 3.13 Wafer best section for MI 1060 coolers.
- 3.13.1 Select the best element pair of the ingots and send to saw room for wafering per process 1454.
- 3.13.2 Cut the ingot sections into .060 thick wafers.
- 3.14 Coat wafers.

- 3.14.1 Coat wafer per process 1110 with the current adjusted to .3 amps for 10 wafers.
- 3.15 Dice wafers for 1060 coolers.
- 3.15.1 Send wafers to saw room for dicing per process 1455.
- 3.15.2 Dice the wafers into .057 X .057 inch elements.

3.16 Build MI 1060 coolers.

- 3.16.1 Build MI 1060 coolers per product spec MI 1060 with n and p-type elements of grown material lot.
- 3.16.2 Build MI 1060 coolers as in 3.16.1 except use p-type elements from standard stock with the elements of grown n-type.
- 3.16.3 Build MI 1060 cooler as in 3.16.1 except use n-type elements from standard stock with the elements of grown p-type.
- 3.16.4 Build an MI 1060 cooler using standard stock.
- 3.17 Test coolers for maximum cold side temperature.
- 3.17.1 Place MI 1060 coolers in vacuum housing and test per process 1486.
- 3.17.2 While holding the hot side temperature at room temperature (27°C) increase the current by one amp increments until the maximum cold side temperature is passed and record the information.
- 3.17.4 Repeat test procedure with hot side temperature at 0°C.
- 3.17.5 Repeat test procedure with hot side temperature at 50°C.
- 3.18 Analyze data.
- 3.18.1 Using the HP-67 calculator, compute the values of figure-of-merit, alpha, rho, kappa and average temperature.
- 3.18.2 Plot data for figure-of-merit, alpha, rho, and kappa.