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David R. Smith
Jerome G. Hust
Lambert J. Van Poolen

National Bureau of Standards
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Thermophysical Properties Division
National Engineering Laboratory
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Boulder, Colorado 80303

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Contents

Abbreviations and Symbols Used in This Report

Note: Primed abbreviations distinguish "top" from "bottom" positions of two elements in an identical pair.

k	Thermal Conductivity
A	Area of Metered Section
BAH	Bottom Auxiliary Heater
BCP	Bottom Cold Plate
BP (BP')	Baseplate
BT	Bourdon-tube
CI (CI')	Cold Plate Offset Insulation
DTC	Differential Thermocouple
DVM	Digital Voltmeter
F (F')	Coolant Fluid Lines
G	Gap between Main Heater and Inner Guard
GHP	Guarded Hot Plate
HI (HI')	Heater Offset Insulation
I	Current
LN_2	Liquid Nitrogen
MH/IG	Main Heater/Inner Guard
MS (MS')	Composite Spacers
ND	Null Detector
OD	Outer Diameter
OG	Outer Guard
OGH	Outer Guard Heater
OGS	Outer Guard Supports
OTC	Over-Temperature Control
Q	Heater Power
R	Resistance
S (S')	Specimen
SR	Strain Relief
SS (SS')	Specimen Thickness Spacers
T_1	Temperature of Specimen Cold Surface
T_2	Temperature of Specimen Hot Surface
TAH	Top Auxiliary Heater
TC	Thermocouple
TCA	Thermal Conductivity Apparatus
TCP	Top Cold Plate
V	Voltage
ΔT	Specimen Temperature Difference
ΔX	Specimen Thickness

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A Guarded-Hot-Plate Apparatus for Measuring Effective Thermal Conductivity of Insulations Between 80 K and 360 K*

David R. Smith**, Jerome G. Hust, and Lambert J. Van Poolen***

Thermophysical Properties Division
National Bureau of Standards
Boulder, Colorado 80303

This report describes a guarded-hot-plate apparatus used to determine the effective thermal conductivity of glass fiber insulations. Measurements can be performed at temperatures from 80 K to 360 K, from atmospheric pressure to a vacuum of 10^{-4} Pa (1×10^{-6} torr). Various fill gases such as air, nitrogen, argon, and helium can be utilized. Overall uncertainties of thermal conductivities at atmospheric pressure are 1% at the higher temperatures and 5% at the lower cryogenic temperatures. The modifications of the commercial apparatus described in this report resulted in approximately a four-fold improvement in uncertainty.

Key words: guarded-hot-plate apparatus; insulation; low-temperature; thermal conductivity.

1. INTRODUCTION

In October 1977, NBS/Boulder acquired a surplus commercial guarded-hot-plate apparatus from NASA/Langley Research Center. This report describes the basic operation of this apparatus and modifications performed to obtain the accuracy and precision needed for the establishment of Standard Reference Material certification data at temperatures in the range 80 K to 360 K (-190°C to 90°C).

The layout of the central stack of this guarded-hot-plate apparatus depicted in figure 1 is consistent with the ASTM C-177 specification.[1] This stack consists

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**Permanent address, South Dakota School of Mines and Technology, Rapid City, SD 57701, Dept. of Physics.

***Permanent address, Calvin College, Grand Rapids, MI 49506, Dept. of Engineering.

[1] 1981 Annual Book of ASTM Standards, Part 18, Thermal Insulation, American Society for Testing and Materials, Philadelphia, PA.

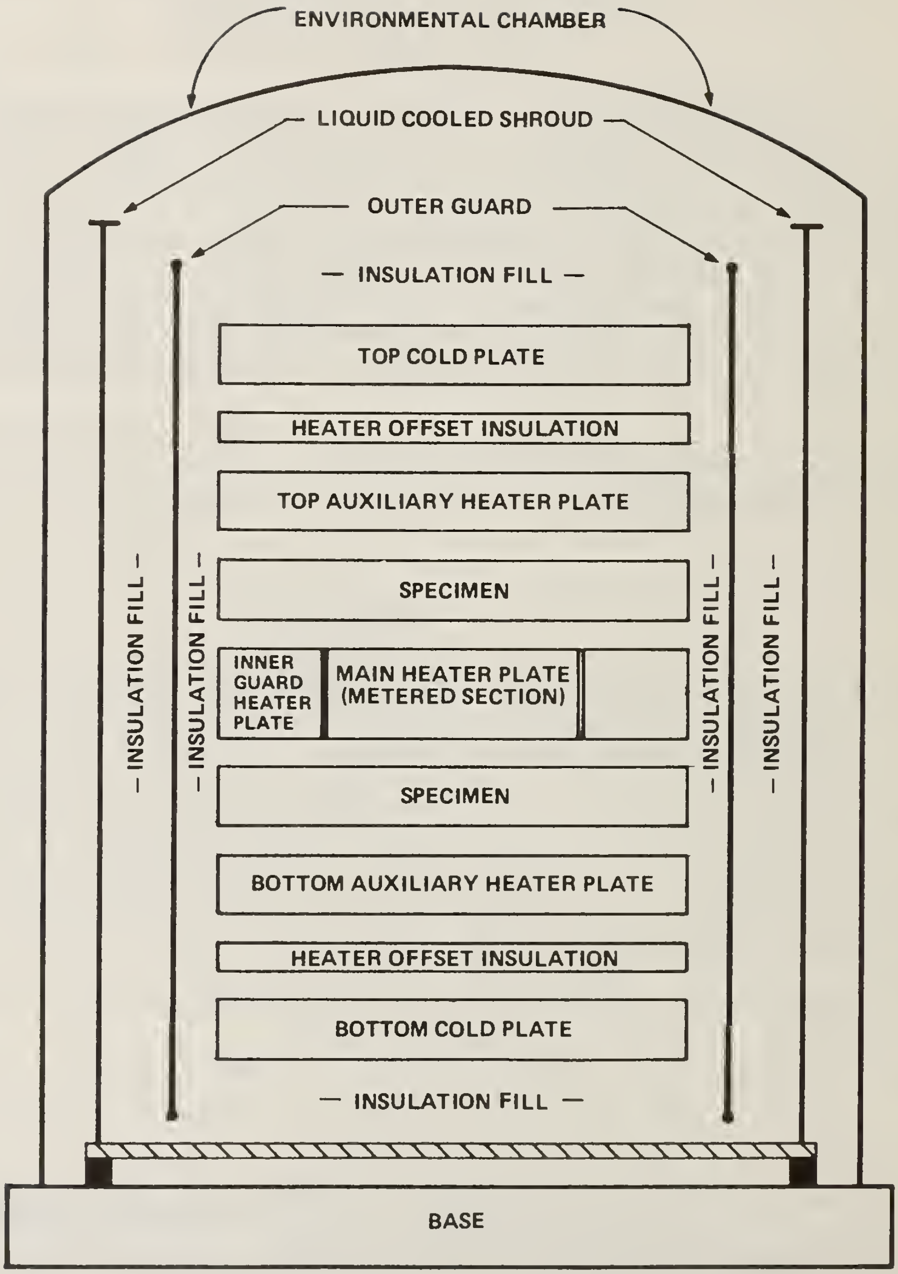


Figure 1. Layout of the Stack, Guards, and Environmental Chamber of the Hot Plate Apparatus Used in this Research.

of a guarded-main-heater plate, two specimens, two cooling plates, and two auxiliary heater plates to provide for additional flexibility in adjusting and controlling the temperatures of the cooler faces of the specimens. The stack is isolated from external influences by the outer guard, a shroud, and an environmental chamber. All of the assembly within the shroud is surrounded by loose-fill insulation. The stack has a circular cross section perpendicular to the vertical axis. The purpose of the stack and associated guarding is to create and measure steady-state one-dimensional heat flow under a set of defined conditions. From these measurements under certain conditions, as specified in the C-177 test method, an effective thermal conductivity may be defined (see section 3.0).

The metered section of the MH/IG plate provides the measured quantity of heat which flows through the specimens to the lower temperature surfaces provided by the auxiliary cold plates, or by the cold plates if the auxiliary plates are not used. The auxiliary plates are thermally offset from the cold plates by an appropriately chosen thermal resistance (the heater offset insulation). The temperatures of the auxiliary plates are electrically controlled to provide stable temperature differences across the specimens. The inner guard, outer guard, shroud, and insulation fill minimize the radial heat flow within the metered area. The temperatures of the guard heaters are electrically controlled to match appropriate temperatures. The cold plates intercept the heat from the heaters and are maintained at a lower temperature by means of various coolants, including liquid nitrogen, chilled alcohol, or water.

A wide range of choices for the specimen temperatures and temperature gradients are provided through the use of the auxiliary cold plates and the various coolants. This flexibility is obtained through the use of the two auxiliary heater plates mentioned above.

The goal of the NBS/Boulder measurement program was to establish thermal transmission data for insulations within ±1% over the temperature range 80 to 360 K. This uncertainty is considered current state-of-the-art. The apparatus as received was designed to measure thermal transmission within ±2% at ambient temperatures and ±5% at liquid nitrogen temperatures in compliance with ASTM C-177 specifications.

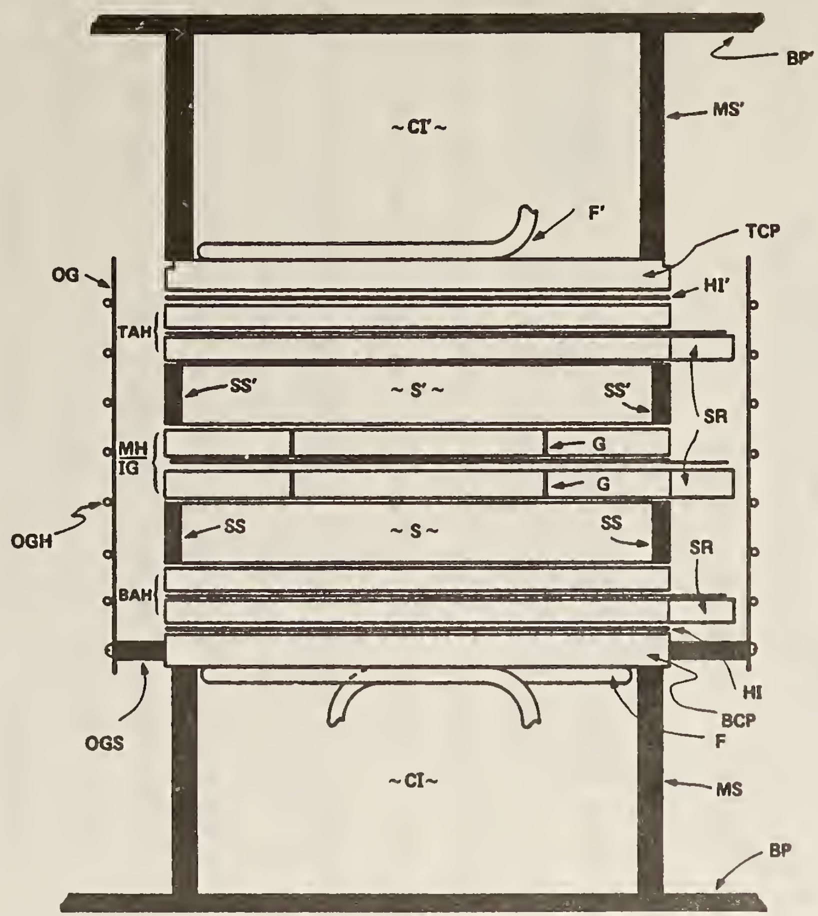
Preliminary tests were conducted to see if this equipment would produce values close to the ±1% uncertainty set as a goal. Results on glass fiberboard indicated that the uncertainties were as high as ±5% at the upper end of the temperature range and as high as ±15% at the low end of the range. These high uncertainties were due primarily to excessive temperature control fluctuations. The above statement applies only to this particular surplus unit and does not necessarily apply to new units produced by this manufacturer. Thus it was decided that the apparatus would have to be modified to achieve our lower uncertainty requirements. The preliminary tests also indicated that modifications could simultaneously improve the efficiency of operation of this particular experimental program.

A detailed description of the stack elements, guarding elements, and other associated equipment is given in the following section. Modifications made to the as-received apparatus are given in detail.

2. DESCRIPTION AND DISCUSSION OF APPARATUS ELEMENTS

2.1 Introduction

Figure 2 shows a more detailed picture of the guarded hot plate (GHP) assembly with heater leads and thermocouples omitted for clarity. Each of the plates facing the specimens are anodized aluminum to achieve a high emissivity. The



BP(BP') = baseplate
CI(CI') = cold plate offset insulation
MS(MS') = composite spacers
F(F') = coolant fluid lines
BCP = bottom cold plate
HI(HI') = heater offset insulation
BAH = bottom auxiliary heater
S(S') = specimen
SS(SS') = specimen thickness spacers
MH/IG = main heater/inner guard
G = gap between MH and IG
SR = strain relief
TAH = top auxiliary heater
TCP = top cold plate
OG = outer guard
OGH = outer guard heater
OGS = outer guard supports

Approximate Overall Dimensions:
BP to BP' = 30cm
Diameter of OG = 24cm

Figure 2. Details of Stack Assembly.

measured emissivity of these plates is 0.83. The components of this stack are individually described below.

2.2 Stack Elements

2.2.1 Main Heater and Inner Guard Assembly

The MH/IG assembly is constructed of a rubberized two-element concentrically arranged heater sandwiched between two circular, black anodized aluminum plates also concentrically split to match the heater arrangement (fig. 3). Each two-piece plate is 9.5 mm (3/8 in.) thick and 203.2 mm (8.00 in.) in total diameter. The anodization increases the emissivity of the surfaces and thereby promotes radiative heat transfer to the specimens during measurements to simulate common in-use conditions. The central disk of both the upper and lower plates, 101.6 mm (4.00 in.) diameter, is separated from the surrounding annulus by a gap of 1.6 mm (1/16 in.) width. Strips of plastic adhesive tape hold the disks in a centered position within the annuli. The plates are bolted together with four flathead screws located as shown in figure 3.

The rubberized heater element (fig. 4) is similarly constructed with a central disk of 101.6 mm diameter separated from the surrounding annulus by a gap of 1.6 mm width. The gap extends for only about 80 percent of the circumference, however. The remaining rubber across the gap provides a bridge for the d-c potential and current leads to the metered heater within the central disk. This heater is powered by a d-c power supply with maximum current and voltage ratings of 2 amps and 55 volts. It was operated in the constant current mode with a stability of better than 0.01%. The annular portion of the MH/IG heater combination contains the Inner Guard heater element (19 ohms) which is powered by a temperature-controller circuit. Details of the controller and the main heater power supply circuit, as modified, are given in a separate section.

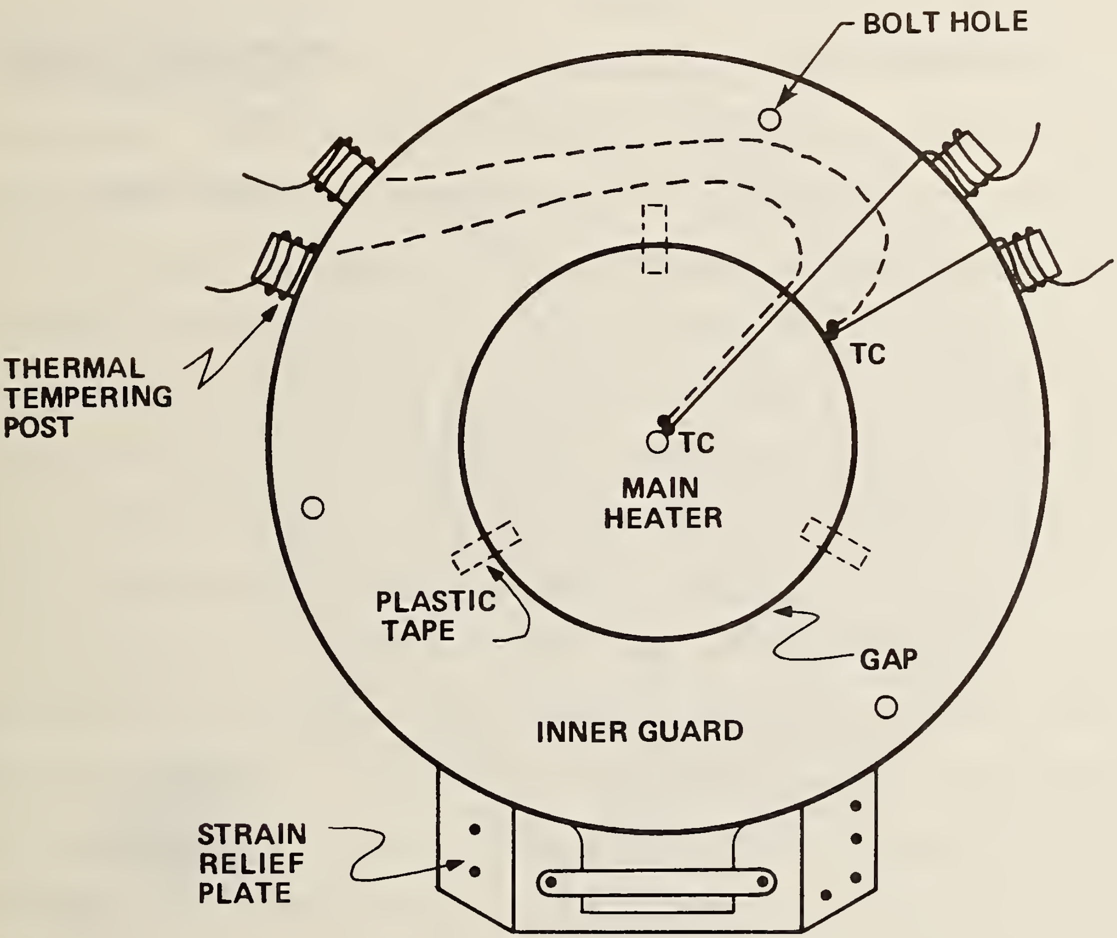


Figure 3. Main Heater/Inner Guard Assembly.

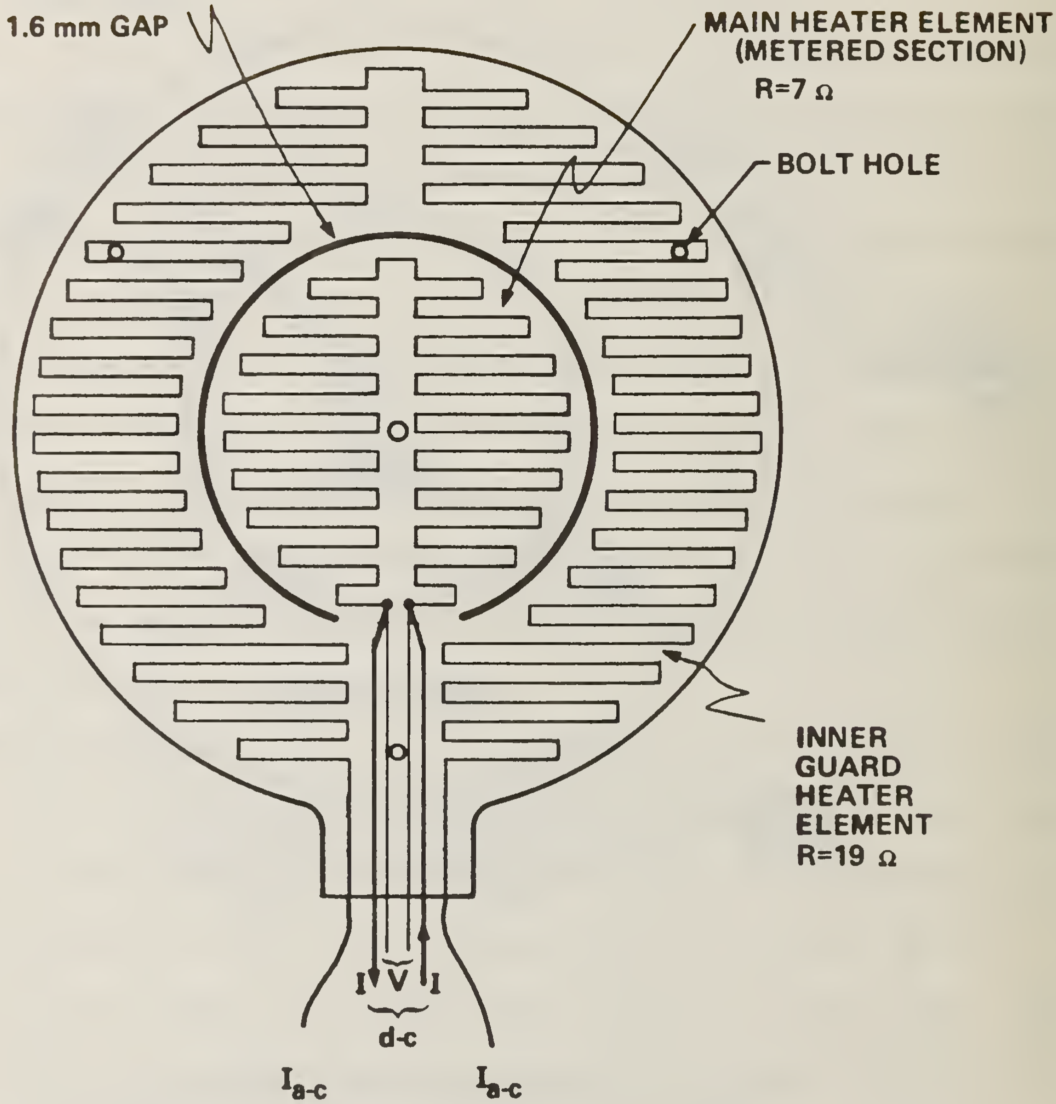


Figure 4. Rubberized Main Heater/Inner Guard Heater Elements.

The unbalance signal to the IG controller is produced by a Type E 20-pair differential thermocouple (DTC), thermopile, illustrated in figure 5. The thermopile elements are constructed of small-diameter (0.20 mm) wires to minimize heat leaks across the gap. The DTC junctions are cemented but electrically isolated, into small grooves in the MH/IG assembly plates. One half of the junctions are on the plates above the heater element and the other half are on the lower plates. This placement is used to average the unbalance of the top and bottom plates to zero. The configuration of this thermopile is non-inductive (i.e., two loops are formed in opposite directions - not illustrated in figure 5) to minimize induced signals. This Type E DTC replaces the 10-pair Type K thermopile originally supplied with the apparatus; a factor of 3 in gap unbalance sensitivity was gained at room temperature, and slightly more than a factor of 3 at 80 K, by this modification. The sensitivity of this thermopile is about 1200 μV/K at room temperature and about 550 μV/K at 80 K.

Originally the MH/IG plate assembly had 2 grooves 1.6 mm wide x 1.6 mm deep machined into each aluminum plate on the side facing the specimens. One crossed the gap and extended to the center of the metered area. The other groove stopped just short of the gap. Type K thermocouples (TC's) in alumina tubes of 1.6 mm OD were fitted snugly into the grooves and were cemented in place with refractory cement. However, at the high vacuum conditions needed occasionally in this research the TC's in the originally supplied ceramic tubes were inadequately anchored (for thermal tempering) to the plate surfaces. To improve the thermal anchoring the following modifications were made: The Type K TC's were cemented with a high conductivity refractory cement into the original grooves along their entire lengths. The wires near the TC beads were raised to be at the upper edge of the grooves, i.e., in the plane of the surface of the plate. Locations of

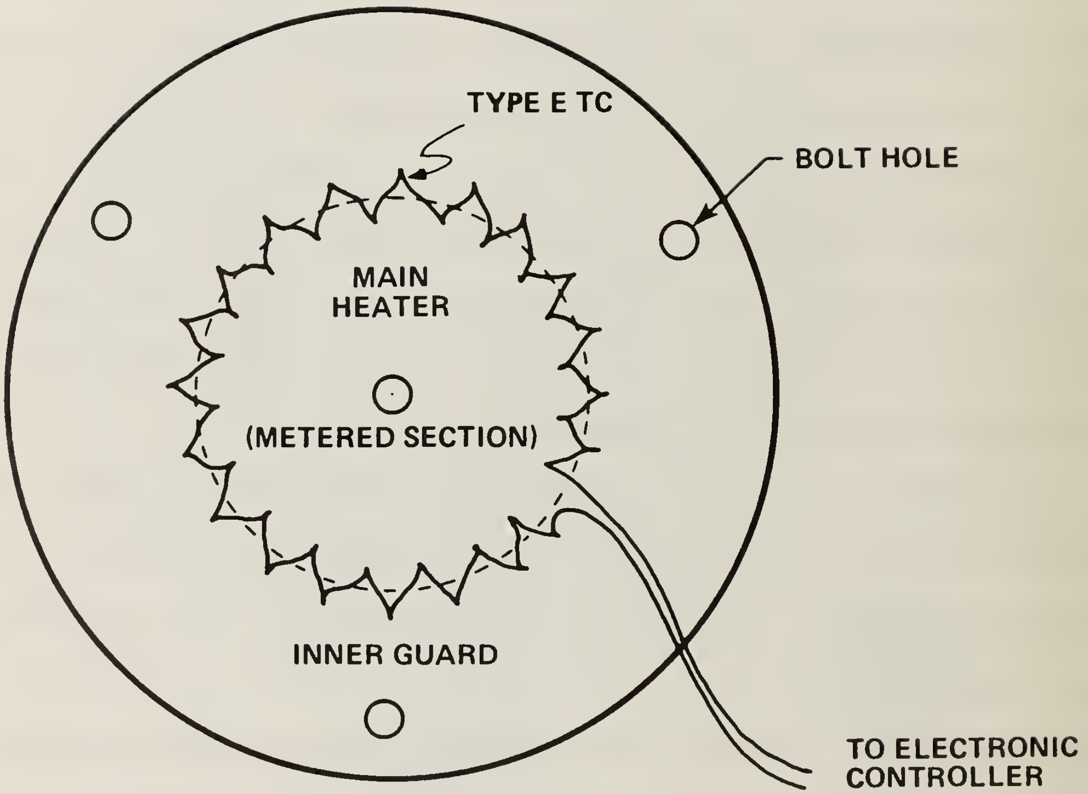


Figure 5. 20-pair Differential Thermocouple (Thermopile).

these thermocouples on the MH/IG assembly are shown in figure 3. Type E thermocouples would yield a higher sensitivity but an extensive rewiring of the entire system would have been required to achieve this moderate improvement in uncertainty.

To provide further thermal tempering of the TC leads to the MH/IG surface, cylindrical copper lugs (13 mm diam. x 13 mm long) were bolted to the edge of the MH/IG plate at the points where the TC's leave the plate. The TC leads were wrapped and cemented around the lugs for a length of 15 cm. Heat conduction along the leads external to the stack should thus be diverted to the IG annulus and not affect the metered area (see fig. 3).

The base plate (BP in fig. 2) equilibrates at a temperature cooler than room temperature but warmer than the bottom cold plate (BCP) temperature when operating at low-temperatures. In the preliminary measurements with LN_2 coolant there was evidence of a significant heat leak from the power terminals on the shroud base plate to the stack via the heavy heater leads. To minimize the effect of this heat leak the heavy leads (2-28 ga a-c leads, 2-28 ga d-c leads, and 2-36 ga potential leads) emerging from the rubberized MH/IG heater pad were replaced with coiled lengths of finer wire of a diameter sufficient to carry the required currents without significant self heating. Their length was great enough to reduce heat leaks into the MH/IG and also sufficient to permit several turns (10 cm) to be wrapped around copper tempering posts mounted on the middle of the OG. The excess length (30 cm) remaining after tempering was coiled to minimize entanglements. The junction between the original heavy leads and the new finer wires were made on fiber-reinforced phenolic blocks mounted on the edge of the strain relief, SR in figure 2, on the MH/IG. The other ends of the new wires were then run to the power terminal strip on the shroud base. These modifications accomplish a diversion of heat leaks to the OG (at points which are heated anyway) and reduce heat leaks to the stack by increasing the lengths of wires running to the stack as well as by decreasing the temperature differences experienced by the portions of such wires immediately adjacent to the stack.

Figure 6 shows the circular MH/IG and annular OG (see section 2.3.1) and the disposition of heater leads from the power terminal strip to the fiber-phenolic blocks on the sides of the MH strain relief. The radial heat leaks across the MH/IG gap and the IG/OG gap are discussed further in sections 2.5.3 and 2.5.4, respectively.

2.2.2 Specimen Thickness Spacers

Specimens involved in the present measurement program are highly compressible. Because of this, rigid spacers are placed between the inner guard and the auxiliary heaters to fix the specimen thickness at a slightly compressed condition to assure good thermal contact between the plates and the specimens.

Matched sets of quartz or stainless steel spacer tubes have been used to maintain a known thickness. Each set of three tubes is cut to the desired length within a tolerance of 0.025 mm. The 7 mm diameter tubes are stuffed with glass fiber to reduce heat transfer along the interior of the tube and are mounted at three equally-spaced points into slots cut into the perimeter of each specimen. After assembly the stack elements are compressed by a screw centered on the upper base plate (see fig. 9) until all the spacers are under light compression, establishing the specimen thickness as that of the spacer tube.

2.2.3 Top Auxiliary Heater

The top auxiliary heater (TAH) assembly, figure 7, is made of two anodized aluminum plates, each 9.5 mm thick and 203.2 mm diameter with a heater (15 Ω) in

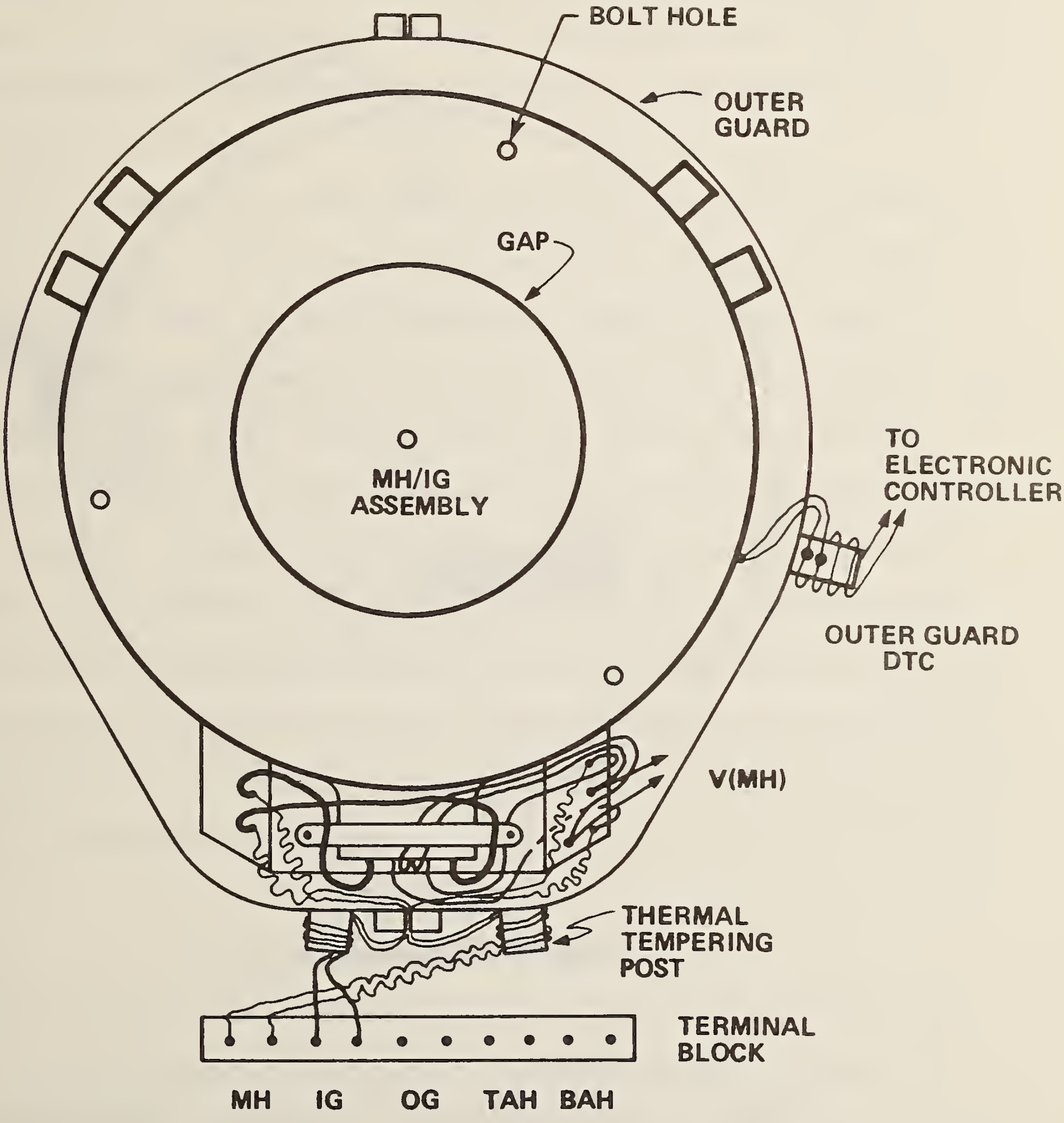


Figure 6. Wiring Details of Main Heater/Inner Guard/Outer Guard Assembly.

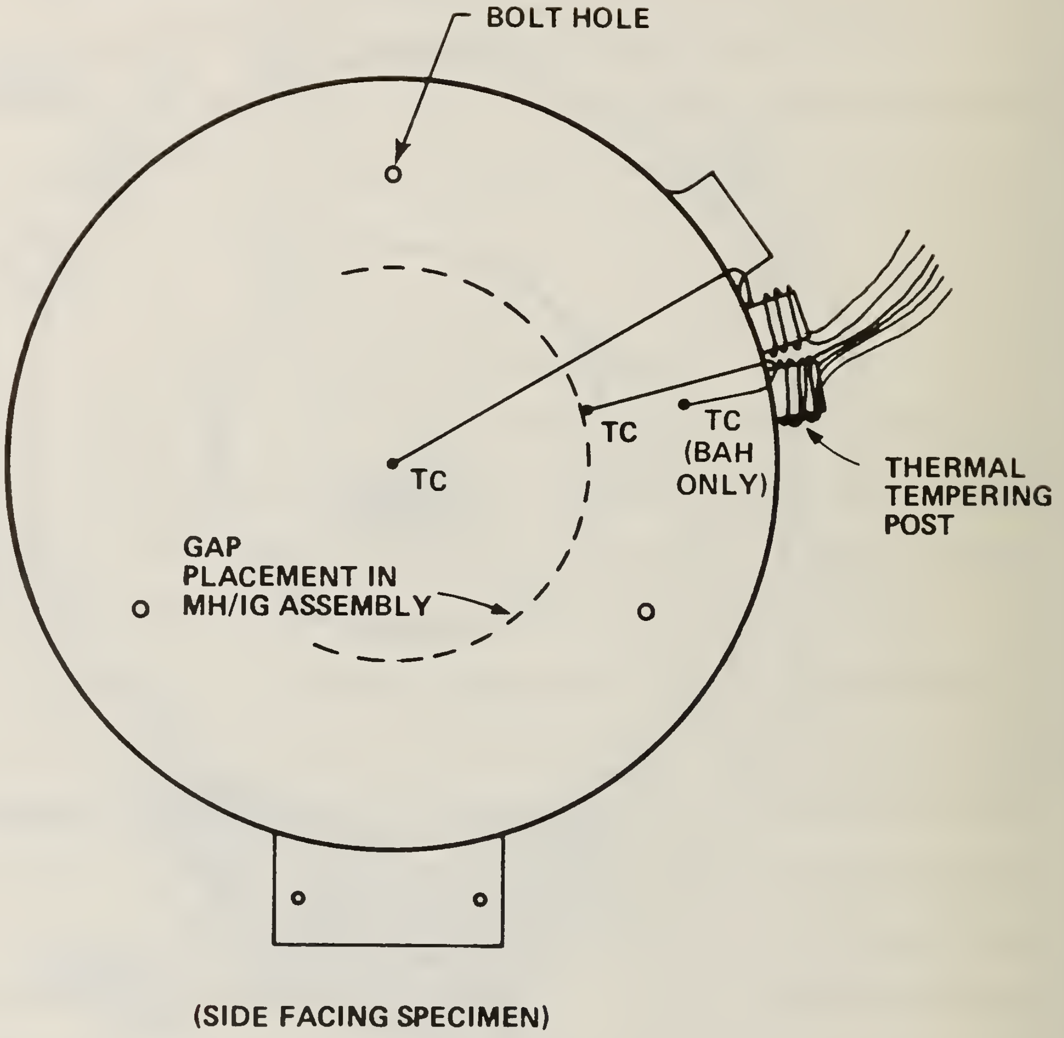


Figure 7. Top and Bottom Auxiliary Heater Thermocouples.

between them. The lower of the two plates had two grooves for TC's on the side facing the specimen and one groove for a control TC on the side facing the heater. All three grooves were filled with cement as described above in the MH/IG section 2.2.1. Two TC's were mounted, one at the center and one half way to the edge of the side facing the specimen, and were tempered on copper posts screwed to the side of the plate, see figure 7.

The TAH, as received, was controlled by a Type K thermocouple referenced to an electronic ice point. To improve temperature control, a resistive temperature sensor was installed in the TAH. This temperature control sensor is made of 30.5 m of 40 gage high resistance alloy wire (37.4 Ω/m at room temperature). The wire was doubled back on itself 200 times into a non-inductive 15 cm length, which was then cemented into a diagonal groove machined into the internal surface of the TAH. Copper leads were soldered to the ends of the resistance wire and flexible cement was placed over the emerging copper leads to serve as a strain-relief. The resulting temperature-sensing resistor has a resistance of 1140 Ω at room temperature and a sensitivity of about 3.8 Ω/K over the entire temperature range. This resistor forms part of the temperature control circuit, described in the section on temperature controllers.

2.2.4 Bottom Auxiliary Heater

The bottom auxiliary heater (BAH) (56 Ω) assembly is similar to the TAH. Modifications to the thermocouple grooves of the BAH assembly are similar to those of the TAH and MH/IG assemblies. The thermocouples (Type K) were also tempered to the surface facing the specimen and to copper lugs screwed to the side of the plate (fig. 7). In the as-received condition, the BAH was also controlled with a TC referenced to an electronic ice point.

In a first modification, the BAH temperature was controlled by a DTC relative to the TAH, however, a more satisfactory result was obtained when the BAH was controlled with an absolute controller rather than a differential controller. The BAH temperature control sensor is of the same type as that described above for the TAH. (Additional modifications of the controller circuitry are given in a later section.)

2.2.5 Cold Plates

The cold plates as manufactured were spaced 5 cm from the upper and lower baseplates and were offset from them by means of two aluminum rings. When water was used as the coolant no problems occurred but when cooling with LN_2 there were some severe thermal link problems. The aluminum standoff rings presented a path of low thermal resistance between the coolant and the baseplates (see fig. 2). The open spaces inside the standoff rings and between the cold plates and baseplates would permit additional heat transfer by radiation and convection. To improve the thermal isolation of the coolant, the aluminum standoff rings were removed and each cold plate was mounted on 3 composite spacer rods. The offset distance was increased to 9 cm. The space between the cold plates and baseplates was packed with glass fiberblanket insulation (see fig. 2).

2.3 Secondary Guarding

2.3.1 Outer Guard

The original thermal conductivity apparatus was equipped with an isothermal shield (outer guard) surrounding the stack. Its purpose was to provide additional guarding against edge heat losses from the stack. The outer guard was provided with a heater element and was designed to be controlled to a temperature equal to that of the IG in the MH/IG assembly.

To improve the guarding of the temperature gradient of the stack a lightweight outer guard was made of copper and stainless steel which could develop a matching temperature gradient in the portions of the guard immediately adjacent to the specimens. The thought was that a guard with a matching temperature gradient would more effectively guard the stack than an isothermal one. Since the new guard could also be operated isothermally, tests were conducted to compare the results from the two modes of operation. No difference was found beyond the imprecision of these measurements ±0.4%.

Therefore, it was decided to revert to the use of a copper outer guard maintained in an isothermal condition at the temperature of the MH/IG. To simplify changing specimens, this guard is made of half shells bolted together and is weakly heat-sinked to the bottom cold plate (see figs. 2 and 8). The half shells form an oval cylinder which surrounds the stack of heaters and insulation specimens with a clearance of about 20 mm from all stack surfaces, including the strain-relief blocks. This clearance is sufficient to provide space for tempering posts fastened to the outer surfaces of the heaters, and clearance for TC and heater leads. The outer guard heater is made of resistance wire (12 Ω). A tempering post is screwed to the shield to provide a mounting for a DTC. This DTC is used in a controller circuit to hold the temperature of the outer guard close to that of the MH/IG region.

2.3.2 Shroud/Insulation Fill

Originally, the bottom baseplate (BP in fig. 2) upon which thermocouple terminal strips were located, was cooled excessively by the LN_2 cooled shroud which was mounted directly on this baseplate with only a thin rubber trim for standoff insulation. The cooling introduced unwanted thermal gradients in the thermocouple terminal strip, hence uncertainties were introduced in temperature

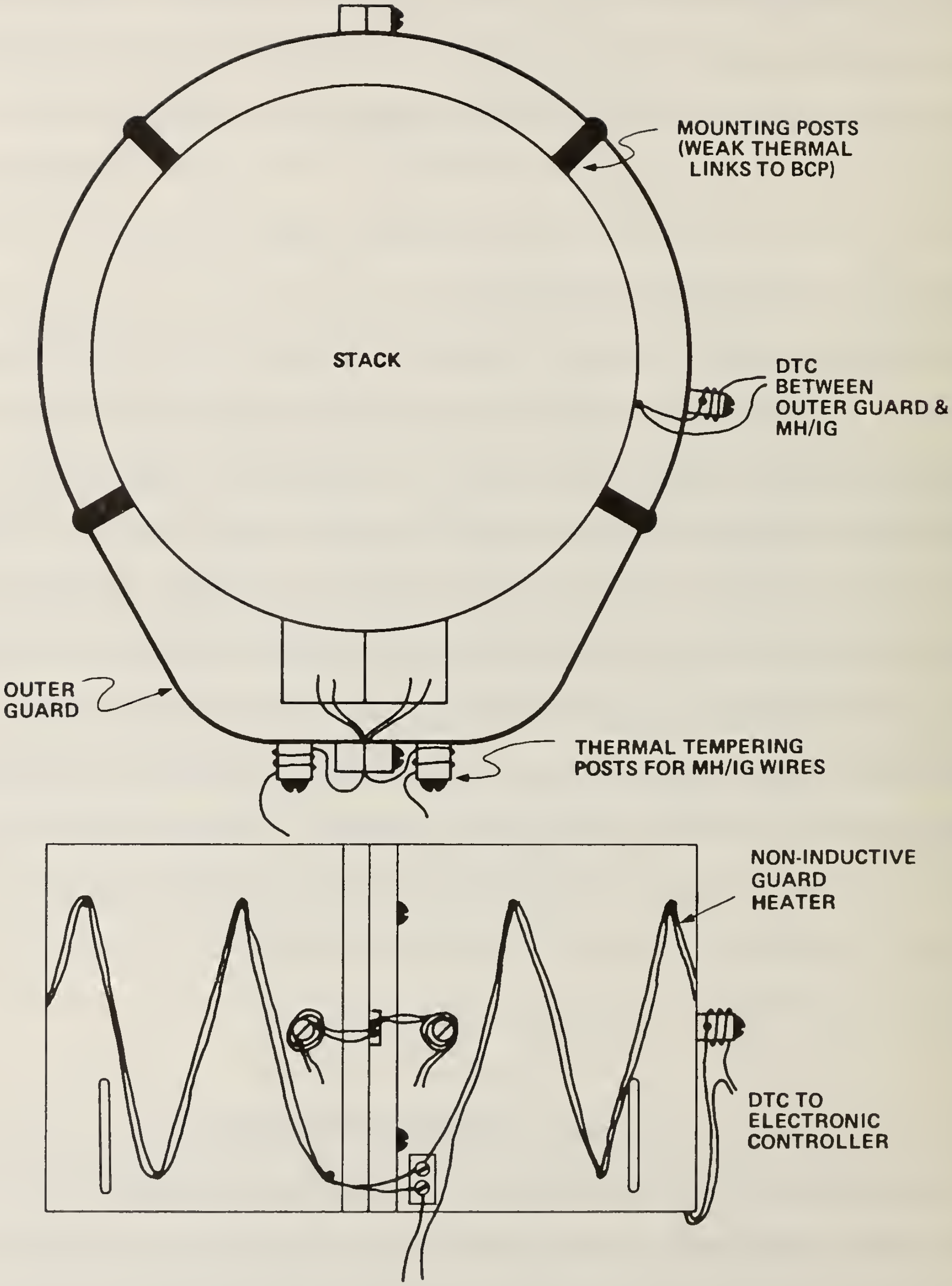


Figure 8. Outer Guard Assembly.

measurement. In addition, the baseplate had fairly direct thermal contact with room temperature through the apparatus support assembly. Thus, LN_2 coolant was wasted maintaining this heat leak. To solve these problems:

1) The thermocouple terminal strips were removed from their original position on the shroud baseplate and mounted on another aluminum plate 10 cm lower (the environmental chamber baseplate) which was directly connected to a massive chamber support ring.

2) The shroud was remounted on a band of epoxy-fiberglass composite to provide thermal isolation between the shroud and its baseplate. The resulting elevation of the shroud (5 cm) permitted the application of a larger quantity of insulation fill within the shroud thereby reducing usage of LN_2 during operation. Sliding doors were constructed in the composite band under the shroud to permit easier removal of the insulation during disassembly.

Expanded mica insulation was used in place of the fine silica gel supplied with the apparatus. The silica gel caused dust problems and was difficult to keep out of the mechanical vacuum pump used to pump on the system. A copper screen topped by 1 inch of glass fiberblanket was used to catch any stray insulation particles penetrating to the vacuum gate valve.

2.3.3 Environmental Chamber

A large metal dewar (environmental chamber) was placed over the entire stack and guard assembly. It provided additional guarding from ambient temperatures and conserved cryogenic coolant. This dewar replaced a glass bell jar supplied with the apparatus. With the use of liquid nitrogen as coolant the metal dewar was deemed safer and eliminated severe frosting which occurred with the glass bell jar.

2.4 Refrigeration

2.4.1 Fluid Transfer Lines

The original uninsulated fluid-transfer lines within the bell jar system of the TCA were designed principally for water as the coolant. Use of uninsulated lines for chilled alcohol or LN_2 as the coolants wastes refrigeration power. Figure 9 illustrates the coolant paths after the following changes were made.

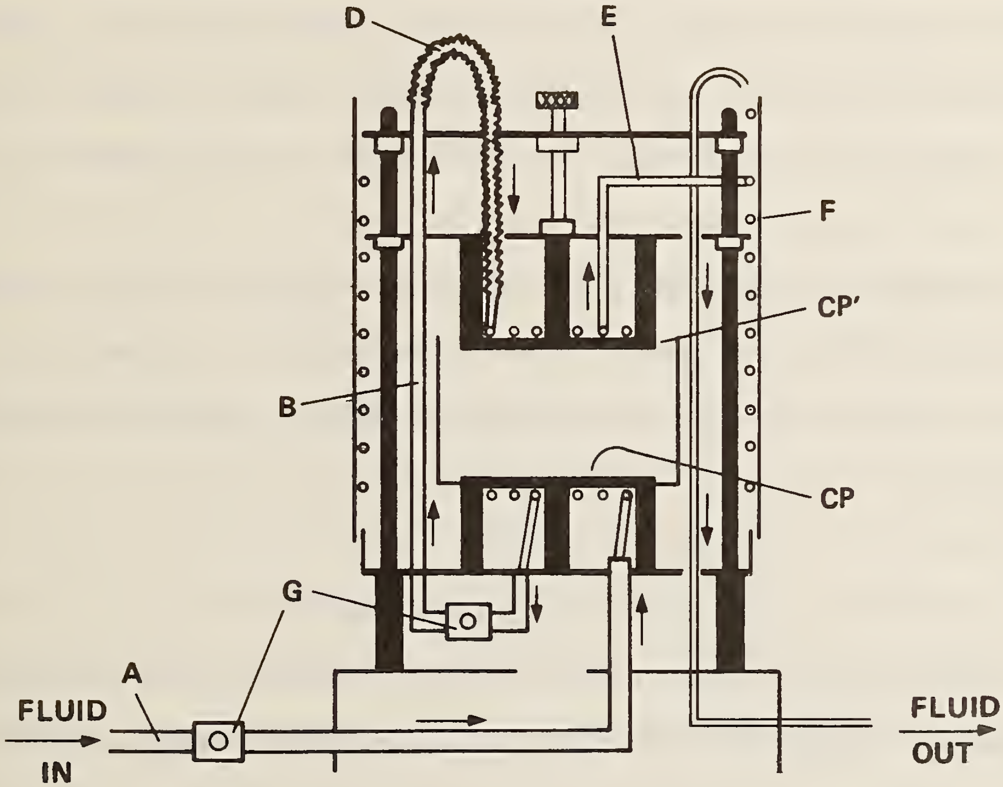
The coolant lines to the two cold plates were originally separate from the shroud coolant lines. The shroud fluid lines were modified to be in series with the cold plate coolant lines. The coolant leaving the stack is then efficiently used to cool the shroud which is the largest cooled element intercepting inflowing heat.

The coolant lines serving the shroud from outside the environmental chamber were disconnected and externally sealed. The cold-plate exit line to the outside was re-routed to meet the old exit point for fluid leaving the shroud. The cold-plate entrance line was removed and was duplicated in shape by a double-walled vacuum-insulated replacement (see fig. 9).

Double-walled vacuum-insulated construction was continued in the fluid line from the BCP to the TCP. Where the line originally consisted of a flexible vacuum-tight stainless steel corrugated hose, a vacuum insulated flexible line was incorporated with internal teflon spacers permitting flexibility and minimizing heat leak to the coolant. The exit line from the top cold plate was rerouted to the entrance point of the shroud coolant line.

A transfer-line pump-out valve was installed in the entrance line external to the vacuum system and another was installed in the interconnecting line between the two cold plates, under the shroud baseplate (see fig. 9).

The external coolant entrance line contains a bayonet-type interconnection permitting rapid change of the LN_2 dewar and minimizes the heat leak into the



A Vacuum-insulated Line for Incoming Fluid
B Rigid Vacuum-insulated Fluid Line from BCP to TCP
CP Bottom cold plate
CP' Top cold plate
D Flexible Vacuum-insulated Line to TCP
E Rigid Fluid Line from TCP to Shroud
F Fluid Lines on Shroud
G Pump-out Ports on Vacuum-insulated Lines

Figure 9. Fluid-Transfer Lines.

transferred LN_2 at the interconnection. The transfer line from the LN_2 storage dewar also has a built-in pump-out valve. All three vacuum-insulated lines are pumped out to an insulating vacuum before beginning LN_2 transfer for operation of the TCA at cryogenic temperatures.

Another advantage of the bayonet-type interconnection is that the female fitting will accept ordinary 9.5 mm (3/8") OD tubing with the O-ring seal. When water cooling is desired, a 9.5 mm (3/8") annealed copper tube joins the female bayonet fitting to the water line. For the temperature range above 200 K a commercial circulating-fluid refrigerator can be used to cool and circulate ethanol into the TCA system via flexible hose and another copper tube mating into the female bayonet fitting. At present these lines from the refrigerator are insulated with a closed-cell insulation and sealed against moisture with polyvinyl electrical tape.

2.4.2 Cryogenic Coolant

When LN_2 coolant is used it is desirable to keep the flow reasonably stable. A gas pressure regulator on a nitrogen gas bottle permits application of a constant gas pressure to the LN_2 storage vessel supplying the coolant to the TCA. A needle valve at the exit line from the outer Dewar baseplate ring is used to control the flow of gas exiting from the system. These controls are used to set the cold plate temperatures only slightly below that of the auxiliary heater plates.

2.5 Temperature Control

Preliminary checks using the four as-received temperature control circuits for the TAH, BAH, IG, and OG, revealed several undesirable features. First, the instability of these circuits was greater than desired for high accuracy measurements and, second, these instabilities were only detectable by observing the stack temperatures for relatively long periods of time, i.e., the controller null indicators were too coarse to exhibit the disturbances. Generally the drifts were about 0.2 K/h, but occasionally a 1-2 K fluctuation would occur for no obvious reason. Other undesirable features were: a) the inability to shut down each controller separately, b) the inability to control the instantaneous power delivered to the heaters, and c) the inability to observe the average power delivered.

After a considerable amount of diagnostic testing, the excessive drift problems were traced to: a) electronic ice-point thermocouple reference devices, b) unstable and temperature-sensitive operational amplifiers in the low signal level circuitry of the temperature controllers due partly to the lack of temperature-control on the controller cabinet, c) excessive spurious emf's generated at TC junction blocks and switches, and d) an excessively high instantaneous power delivery to each of the heaters precluding optimum low power operation.

Extensive changes were made to eliminate the problems. Four on/off power switches were added to provide independent operation of each controller. The temperature control sensor circuitry for the TAH and BAH was modified so that the need for a constant-temperature reference point for the thermocouples was eliminated. A power control circuit was added to allow control of the instantaneous power delivered to the heaters, and a monitor circuit was installed so the operator can qualitatively gauge the average power delivered. The details of these changes are presented below for each control circuit. For a clearer understanding of the need for these changes, the following brief explanation of the controller operation is given.

These temperature controllers employ zero-firing triacs capable of delivering up to 1000 watts of a-c line power to the heaters. Line power is delivered for an integer number of half cycles, the frequency of the delivered half cycles being determined by the magnitude of the error signal fed into the controller. However, the instantaneous peak voltage during the "on" half cycles is fixed at 120 volts, i.e., line voltage. Under conditions of very low average power demand, which occurs at low-temperature, steady-state conditions, a single cycle may be sufficient power for a long period of time. This results in a rapid temperature rise and a slow return to the control point, i.e., the controller acts more like an on/off controller rather than a proportional controller. Proportional controllers, generally, result in better temperature control. The solution is to reduce the peak output voltage so that the temperature rise rate during the "on" period approximately matches the natural cooling rate of the controlled system. This simplistic solution is complicated somewhat by the following design limitations of these controllers: a) they should not be operated with inductive devices on the output and b) the instantaneous output power during the "on" cycle should not go below 250 watts. These limitations dictated the nature of the design and the component sizes shown in the modified circuits, figures 10 and 11.

Figures 10 and 11 illustrate the power dissipation networks which were installed to control the peak power delivered to the heaters while simultaneously allowing the peak power controller output to remain above 250 watts. A high-low power level feature was incorporated to allow large rapid changes of temperature when needed (high power) and to achieve a good match to the system characteristics when at equilibrium (low power). In addition, figures 10 and 11 show the addition of a neon light circuit across the output of each controller. These lights give the operator an immediate, qualitative indication of the frequency at

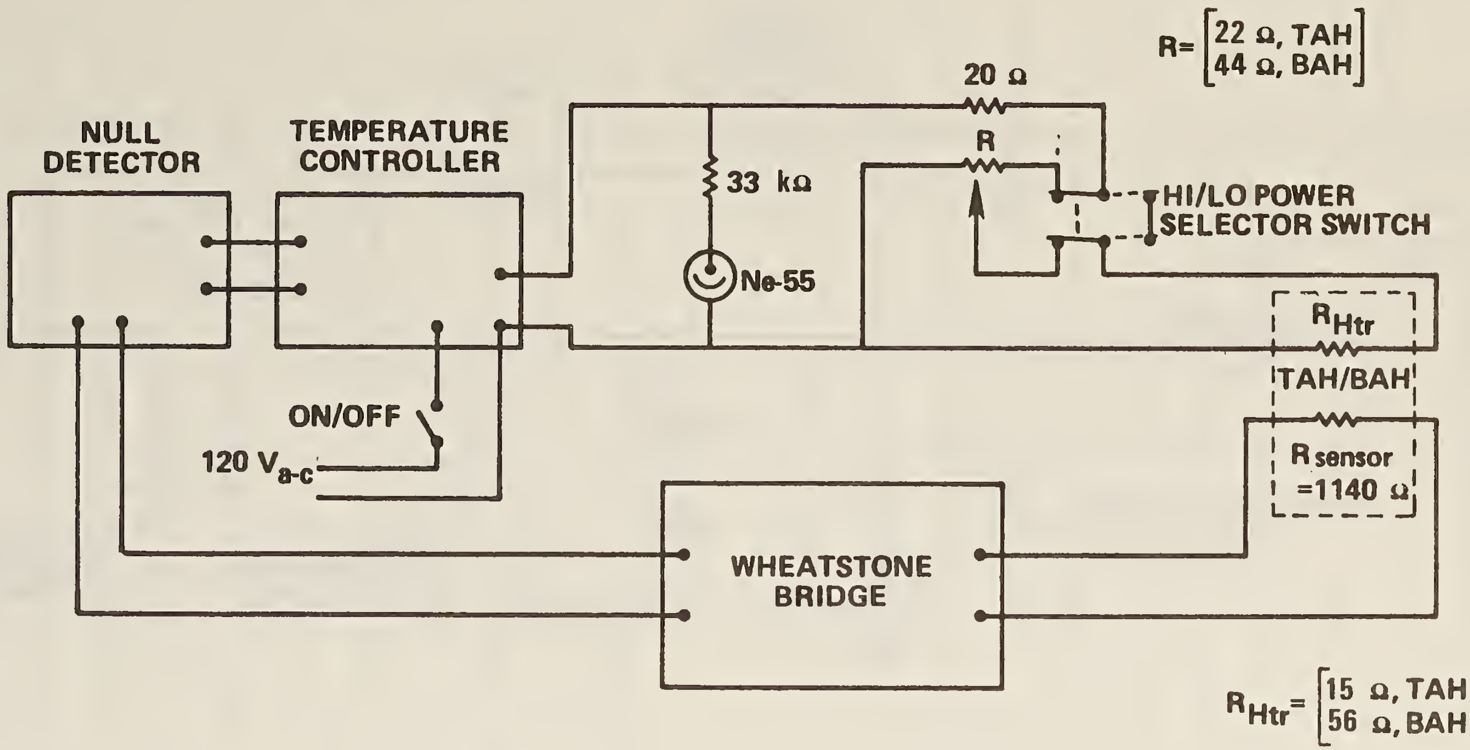


Figure 10. Temperature-Controller Circuit for Top and Bottom Auxiliary Heaters.

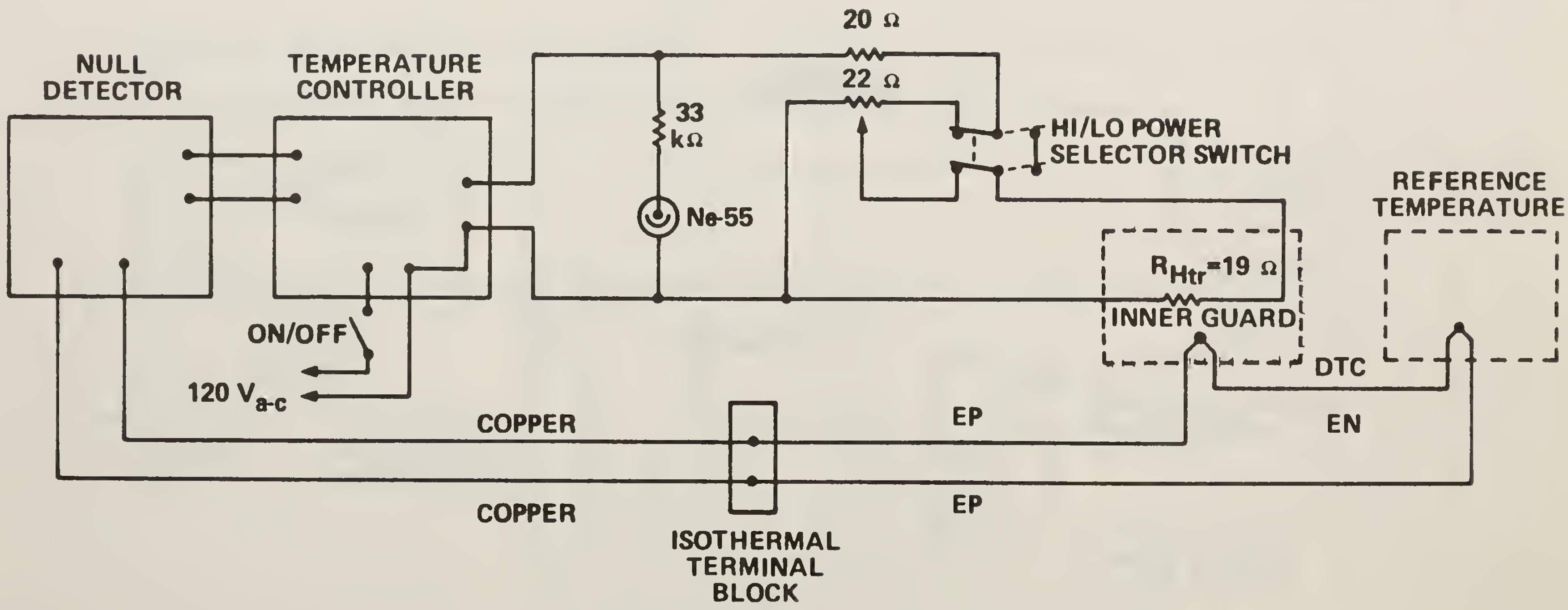


Figure 11. Temperature-Controller Circuit for Inner Guard.

which the half cycles are delivered. Experience has shown that up to about ten pulses per second can be visually resolved.

The front end (low signal level) of the controllers contains the equivalent of a null-detector and a bucking voltage. However, the controllers were not designed for the degree of precision control needed to obtain highly accurate measurements, i.e., the bucking voltage and the operational amplifiers were not sufficiently stable. A relatively simple solution to this problem is to remove the front end and replace it with an external null-detector amplifier with sub-microvolt stability. These units are inexpensive and provided excellent control while simultaneously introducing a sensitive meter to monitor the actual control offset. The front end of the outer guard controller was not modified because it was found that precise control of the outer guard was unnecessary. Specific characteristics of each modified control circuit are given below.

2.5.1 Top Auxiliary Heater Control

The TAH control, figure 10 operates on the basis of a temperature sensitive resistor installed within the plate, as previously described. This resistor is connected as one arm of a Wheatstone bridge circuit. The output (unbalance) of the Wheatstone bridge goes to a microvolt null-detector amplifier whose output, in turn, is the input to the temperature controller. The output of the temperature controller powers the plate heater and, in turn, returns the bridge to balance. The bridge setting determines the temperature control point.

The sensor has a temperature sensitivity of 3.8 Ω/K and the sensor bridge combination has a sensitivity of about 12 μV/mK. During equilibrium operation the null detector range can be as low as 10 μV full scale, but generally the 30 μV range is sufficient for control stability. Experience shows that the TAH control is usually within 1 mK/h.

2.5.2 Bottom Auxiliary Heater Control

This modified control circuit is also illustrated in figure 10. The circuitry is essentially the same as the TAH circuitry. The control of the BAH is similarly within 1 mK/h.

2.5.3 Inner Guard Heater Control

This modified control circuit is illustrated in figure 11. The only notable difference between this and the BAH/TAH control is that the temperature sensor is a thermopile as previously described. The thermopile has a high output per unit temperature unbalance, hence a higher sensitivity to deviations from the control point. This is necessary because the heat flow across the MH/IG gap relative to heater power can be significant even for small gap temperature unbalance.

With this circuit, an average unbalance of less than 5 μV thermopile output is readily maintained. This corresponds to an unbalance of 4 mK at room temperature and about 8 mK near liquid nitrogen temperature. For typical low density insulations with a temperature gradient of 1 K/mm this corresponds to errors of about 0.2% in heater power. The measured and calculated error in thermal conductivity versus gap unbalance for glass fiberboard and fiberblanket at room temperature is 0.04% per microvolt of thermopile output. The approximate relative heat flow components across the MH/IG gap have been calculated and are given below:

Component	Percent of total gap conduction
Gas conduction	40%
Wire conduction	40%
Radiation	15%
Rubber bridge	5%
Gas convection	Negligible

These percentages are applicable for near room temperature with air or nitrogen as fill gas. At lower temperatures the radiation component is considerably smaller. Under vacuum conditions the gas conduction term essentially vanishes.

2.5.4 Outer Guard Heater Control

Although the low signal level portion of this controller is unmodified, a power dissipation and control circuit, similar to figure 11, was added. Adequate control of peak power to the outer guard is obtained by a 20 Ω resistor and 11 Ω rheostat in series with the 12 Ω OGH. The effect on measured thermal conductivity as a function of outer guard unbalance was small, 0.01% change in measured thermal conductivity per °C of outer guard offset. Therefore, accurate control of the outer guard is not necessary and the no analysis of the various heat flow components is given.

2.5.5 Cabinet Temperature Control

To reduce the effects of room temperature changes on the stability of the stack controllers, a temperature controller was installed in the cabinet housing the other four controllers. This controller maintains the temperature of the cabinet within ±0.5 K. It has been found that this was essential with the original controllers but, after modifying the other controllers as described, cabinet control was unnecessary.

2.5.6 Over-Temperature Control

A protective circuit to prevent excessive overheating in the stack (called the over-temperature control, OTC) was furnished with the apparatus. A Type K thermocouple sensing the temperature at one point in the stack is used in a bridge circuit with an optical meter relay. If the temperature at the measured point in the stack exceeds that established by a set-point indicator, the meter relay shuts off all power to the cabinet, including that to an auxiliary power receptacle. The electronic rack containing the d-c power supply for the MH is connected to this receptacle, and so is also under the control of the OTC.

The OTC sensing thermocouple is at the outer edge of the MH/IG assembly, i.e., the hottest point in the stack. Since the TAH and BAH are colder, they are less likely to overheat the specimens or other delicate parts, such as the rubberized heating elements. If either the MH or IG overheats, the sensing thermocouple will detect the fault; the IG is controlled to follow the temperature of the MH and so will follow an overheating fault in the MH. If the TAH or BAH were to overheat a temperature wave diffusing toward the MH/IG would be initiated eventually also raising the MH/IG temperature. So the placement of the single OTC thermocouple at the MH/IG is optimum.

2.5.7 Assessment of Control Stability

The original apparatus, as already stated, performed well below our needs for accurate measurements. After the modifications were made tests showed marked improvement in the temperature stability of the system. The long term (24 h) stability of the current system as measured by the primary thermocouples is 0.1 K. The short term (1 h) stability is 0.02 K. These instabilities include not only the effect of the controller instabilities but also the main heater power supply fluctuations, spurious thermocouple and switch emf's, and read-out instrumentation variation. These variations are used, in part, as the basis for the error analysis given in a later section.

2.6 Measurement

2.6.1 Metered Area Power

The d-c power supply in the as-received apparatus was found to have, for the purposes of this research, insufficient output stability. It was replaced with a commercial high-accuracy Voltage/Current Calibrator with a specified stability of ±0.0025% of setting or 0.0005% of range (whichever is greater) per hour after 2 minutes of constant current output, when operated in the constant current mode.

To reduce the uncertainty in power measurement, the 0.1% resistors supplied with the apparatus for the measurement of heater current and voltage were replaced. In the original equipment a series resistor was used to determine current and a two-resistor voltage dividing network across the heater was used to measure heater voltage. The present system contains a 1 Ω (0.005%) standard resistor in series with the heater for current determination while the heater voltage is measured directly with a high input resistance DVM. With this arrange ment, both the heater voltage and the voltage across the standard resistor are high enough that spurious thermal emf's are negligible and the need for forward and reverse readings is eliminated.

The 1 Ω standard resistor is mounted in the main cabinet controlled to a temperature of 37.5 ±0.5°C. The fractional change in resistance from the calibration temperature of 25°C to 37°C is 0.02%, and is calculated accurately.

Analysis indicated that the measured current supplied to the main heater is accurate to within 0.01%, including power supply variations and all measurement uncertainties. This is equivalent to 0.02% uncertainty in power to the main heater.

It is noted that the d-c power supply can be controlled automatically by a circuit which senses the difference between the actual temperature gradient and the desired equilibrium gradient in the specimens. Power is then fed to the main heater proportional to this difference resulting in a fairly rapid establishment of equilibrium conditions. However, power data for the determination of effective thermal conductivity are taken only when the power supply is operated manually to assure accurate readings of heater current and voltage.

2.6.2 Temperature Measurement

The measurement of all critical apparatus temperatures and temperature differences is performed by spool-calibrated Type K thermocouples as previously mentioned.

A spool of Type KP and one of Type KN thermocouple wire were reserved for use exclusively in this system and a Type K thermocouple made of wire from them was calibrated using liquid He, LN_2, dry-ice-alcohol, water-ice, room temperature water and boiling-water baths. Vapor pressure was used to determine the temperature of the liquid helium bath, while a calibrated platinum resistance thermometer was used to determine the temperatures of the remaining baths. The difference between these calibration data and the standard tables[1] were used to obtain the coefficients of a simple four term polynomial by least square fitting. This calibration contains an estimated uncertainty in temperature of about 20 mK and 50 mK at room temperature and liquid nitrogen temperature, respectively. The corresponding temperature difference uncertainties for a $\Delta T = 25$ K are about 0.1 and 0.2%. Differential thermocouples for the controllers were made from Type E thermocouple wire for greater sensitivity; these DTC's do not have to be calibrated because they operate essentially at zero output. However, the DTC's must be carefully mounted to minimize spurious emf's.

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[1]R. L. Powell, W. J. Hall, C. H. Hyink, L. L. Sparks, G. W. Burns, M. G. Scroger, and H. H. Plumb, Thermocouple Reference Tables Based on the IPTS - 68, National Bureau of Standards Monograph 125, 1974.

A source of uncertainty in temperatures measured by the thermocouples is spurious thermal emf's generated by the lack of isothermal conditions at terminal blocks within the environmental chamber. The repositioning of these blocks to minimize this problem was indicated earlier. The terminal strips were mounted on heavy aluminum angle stock to provide an isothermal region and a vertical orientation for easy access to the terminals.

To minimize reconstruction efforts, portions of the originally supplied TC wire were retained. The original wire is connected to the newly calibrated wire such that both ends of the original wires are at nearly equal temperatures minimizing the contribution of these wires to the measured emf. In addition, a second set of new wires were connected to the ends of the old wires to determine the differences in thermopower between old and new wires. Thermocouples were mounted on the TC blocks at both ends of the original wire to establish the magnitude of the small temperature difference across the wire. From these data, one can correct for the small differences in calibration between the old and new wire. To date, no such corrections have been required.

Additional aluminum blocks are used in conjunction with the TC terminal blocks to temper the TC leads arriving at and leaving from the blocks (see fig. 12). The original TC wire is relatively large (20 gauge) and lies in grooves milled into the opposing surfaces of two blocks. Silicone vacuum grease provides thermal contact between the wires and the blocks. The NBS-calibrated wires (32 gauge) are compressed between 2 layers of electrically insulating film bonded to the blocks with rubber cement. Silicone grease is used to hold the leads in place during assembly as well as for thermal contact between the wires and the blocks.

After the original TC wires pass through the vacuum seals they are brought to a terminal strip which is mounted on a heavy metal plate thermally insulated

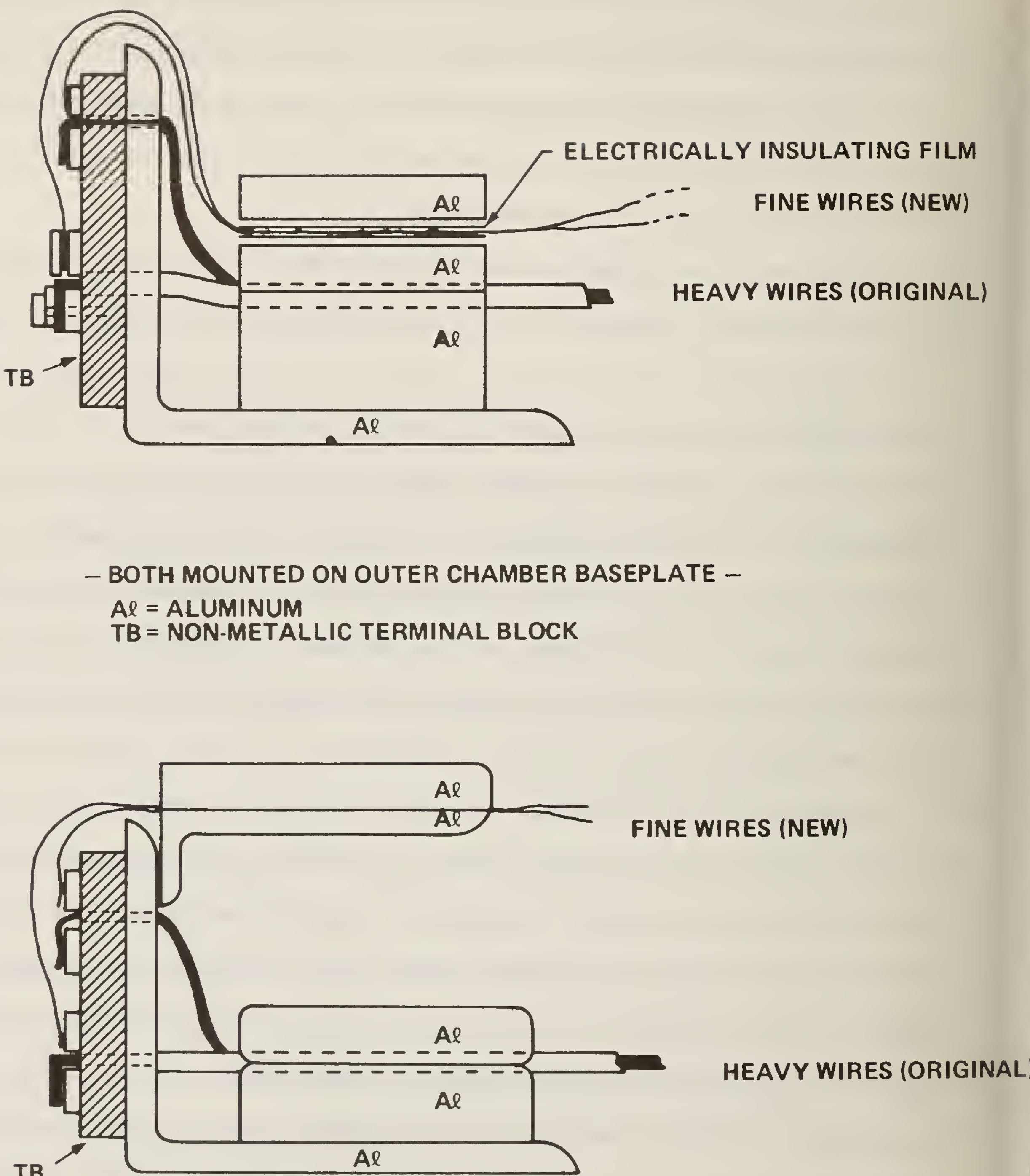


Figure 12. Thermocouple Tempering Blocks.

inside a heavy metal enclosure called the Recorder Terminal Box. The TC wires join the corresponding calibrated wires, which continue to an ice reference bath, where they are joined to thermocouple-grade copper wires. The copper wires connect to the TC emf selector switches. Both Type K thermoelements as well as the copper wire are 0.13 mm in diameter exclusive of electrical insulation. The wires within the ice bath are wrapped around copper tubes over a length of 20 cm for thermal tempering. The wires and copper tubes are divided into 3 groups and placed in glass tubes filled with pure mineral oil. Rubber stoppers at the tops of the tubes hold the wires from being pulled out and prevent ice from entering the tubes during renewal of the ice bath.

Two additional thermocouples are used to gain information on conditions outside the stack. One is mounted on the fluid line between the TCP and the shroud to indicate the coolant temperature at this point. The other is mounted to obtain the cabinet temperature as a drift in this temperature may affect the performance of the enclosed electronic components. Figure 13 shows the location of the measuring thermocouple junctions in the stack.

2.6.3 Selector Switches

The original 20-position selector switch was utilized for a time to read thermocouple emf's and MH voltage/current. After about one year of use, it became electrically noisy and was replaced by two double-pole rotary switches specifically designed for microvolt level measurements. Experience has shown that these switches, when mounted in an isothermal environment, are noise free to well below 1 μV.

The switch positions and corresponding data are indicated in table 1. Refer to figure 13 for thermocouple locations. Switch "A" allows the reading of data germane to the operation of and effectiveness of guarding within the apparatus

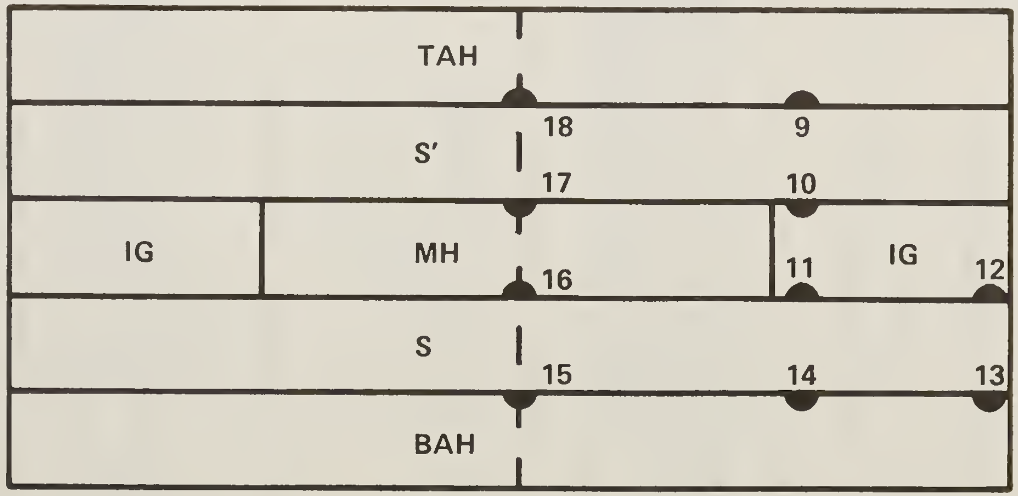


Figure 13. Locations of Measuring Thermocouple Junctions in Stack.

Table 1. Information Obtained At Selector Switch Positions

Switch A (Switch B = 0) Position	Switch A Data	Switch B (Switch A = 0) Position	Switch B Data
0	Open	0	Open
1	TC1 (Environmental Chamber Terminal Block)	1	Shorted (Allows DVM Zeroing)
2	TC2 (Shroud Inlet Coolant Line)	2	TC15-TC16 (Bottom Specimen Gradient)
3	TC4 (Cabinet Terminal Block)	3	TC18-TC17 (Top Specimen Gradient)
4	TC6 (KN-KN') For the calibration of old vs. new TC wires	4	Shorted (Allows DVM Zeroing)
5	TC7 (KP-KP') For the calibration of old vs. new TC wires	5	TC15 (Primary stack temperatures)
6	TC9-TC18 (Differential readings)	6	TC16 (Primary stack temperatures)
7	TC10-TC17 (Differential readings)	7	TC17 (Primary stack temperatures)
8	TC11-TC16 (Differential readings)	8	TC18 (Primary stack temperatures)
9	TC12-TC16 (Differential readings)	9	MH Voltage
10	TC13-TC15 (Differential readings)	10	MH Current
11	TC14-TC15 (Differential readings)	11	TC3 (Temperature Controller Cabinet)

while Switch "B" yields the primary data used in computing thermal conductivity. The control sensors are not shown in figure 13. All of the thermocouples used for temperature measurement (not control) are referenced to an ice bath and the differential readings are obtained by appropriate wiring at the selector switches.

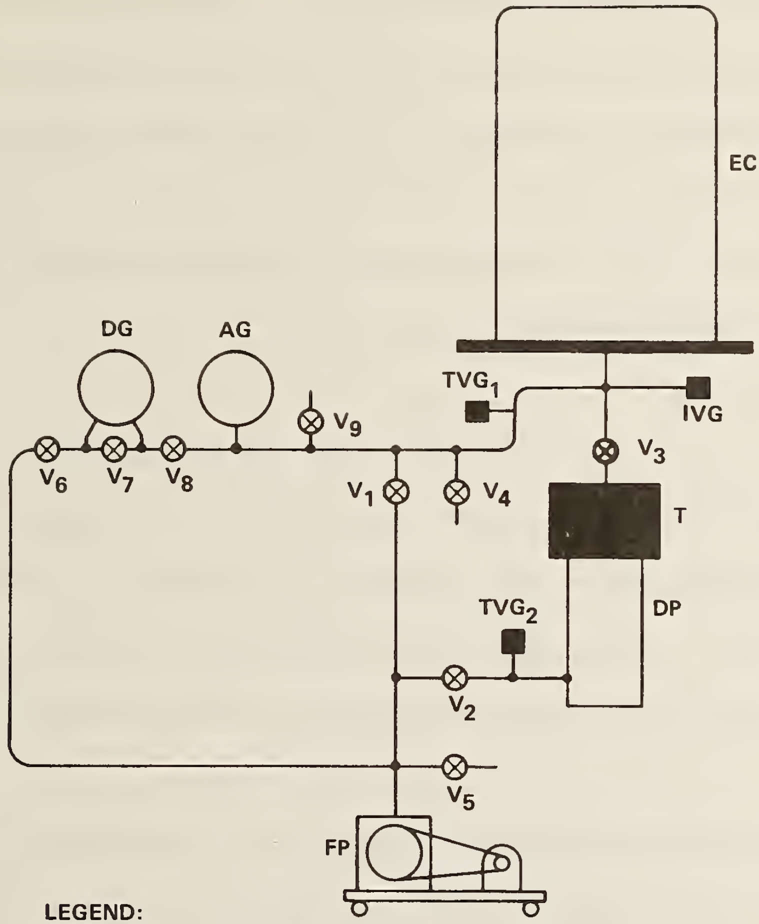
2.7 Vacuum/Gas Back-Fill System

The principal change made to the vacuum system was the addition of two dial gauges (DG and AG in fig. 14) to permit monitoring of gas pressures between 133 Pa (1 torr) and 1.33 x 10^5 Pa (1000 torr) within the environmental chamber. Two thermocouple gauges (TVG1 and TVG2) and an ion gauge (IVG), already present, allow the measurement of pressures below 133 Pa (1 torr).

Absolute gauge AG is a precision Bourdon-tube (BT) type whose dial is calibrated to read absolute pressures up to 1.33 x 10^5 Pa (1000 torr) with a resolution of 665 Pa (5 torr) over a dial scale displacement of 75 cm.

Differential gauge DG is also a BT type but whose dial is calibrated to read up to 6650 Pa (50 torr) with a resolution of 27 Pa (0.2 torr) over a scale displacement of 75 cm. The gauge reads the differential pressure between the inside of the BT and the space exterior to the BT but inside the sealed gauge body. By means of valves V6, V7, and V8 one can bring the gauge body to any desired pressure and then monitor departures from that pressure. Since the dial is not of the center-zero type, however, it reads only positive changes from its initialized pressure. For this reason the gauge body is usually fully evacuated and pressures up to 6650 Pa (50 torr) are read on it with greater precision than can be done using AG.

The atmospheric entrance to valve V4 was somewhat inaccessible so valve V9 was added to facilitate back-filling the Environmental Chamber with any desired gas.



LEGEND:

AG = ABSOLUTE PRESSURE GAUGE
DG = DIFFERENTIAL PRESSURE GAUGE
DP = DIFFUSION PUMP
EC = ENVIRONMENTAL CHAMBER
FP = FOREPUMP
IVG = ION GAUGE
T = LIGUID-NITROGEN-COOLED TRAP
TVG1 = } THERMOCOUPLE GAUGES
TVG2 =
V_1 = ROUGHING VALVE
V_2 = } VALVES TO ISOLATE DP
V_3 =
V_4 = AIR-BLEED VALVE
V_5 = AIR-BLEED VALVE
V_6 = DG CASE EVACUATION
V_7 = DG PRESSURE EQUALIZER
V_8 = INLET TO DG
V_9 = GAS BACK-FILL INLET

Figure 14. Vacuum/Gas Back-Fill System.

The above changes permit measurement of thermal conductivity as a function of fill-gas species and pressure in gas-permeable specimens at any gas pressure at and below atmospheric pressure. It is noted that the gas pressure must be adjusted low enough to prevent condensation onto the cold plates. Condensation causes serious errors in the measurement of effective conductivity because the fluid migrates between the hot and cold plates.

3. DATA ACQUISITION AND CALCULATIONS

The purpose of this apparatus is to establish and measure equilibrium conditions of one-dimensional heat transmission through the metered areas of the two specimens. Under certain conditions, specified by ASTM test method C-177, these data can be utilized to define the effective thermal conductivity of the specimens.

The effective thermal conductivity, k, of the specimens in the guarded-hot-plate apparatus can be determined from

$$k = \frac{Q}{2A} \frac{\Delta X}{\Delta T} = \frac{VI}{2A} \frac{\Delta X}{\Delta T} \quad (1)$$

where Q is the rate of heat flow through the specimens found from the product of heater voltage, V, and current, I; 2A is the combined area of the two specimens each of area A; ΔX is the average thickness of the two specimens; and ΔT is the average temperature difference between the hot and cold surfaces of both specimens. This value of k is usually associated with the mean temperature of the hot and cold surfaces, $\overline{T} = (T_1+T_2)/2$.

The data required for these calculations are, thus, heat generated by the MH, Q = VI, the average thickness of the specimen, ΔX, the metered area, A, and the temperatures of the specimen surfaces. After equilibrium has been established, as evidenced by the drift rate of the specimen surface temperatures, various data are recorded as indicated on the sample data sheet illustrated in figures 15 and 16.

These figures are self explanatory in conjunction with table 1 and figure 14. The data are normally taken in triplicate approximately 30 minutes between each set. This procedure is specified by ASTM C-177 to assure equilibrium conditions. The data from figures 15 and 16 are entered into a computer and processed as indicated by the steps listed in table 2. The output of this computer program contains the variables involved in eq (1) for each equilibrium point as well as the various conditions under which that run was conducted.

The conditions which can be varied and should be specified are: a) specimen characterization, b) pressure and composition of the fill gas, c) plate emissivity, and d) hot and cold surface temperatures. Sets of output from this program are further analyzed to yield dependencies of heat transmission on various parameters such as temperature, temperature difference, pressure, and density. These final analyses are considered beyond the scope of this report and are not discussed further here.

4. ERROR ANALYSIS

The apparatus is designed for the determination of heat flow through the specimens under a prescribed set of boundary conditions and the subsequent calculation of effective thermal conductivity, k, when applicable. The error (uncertainty) analysis will be directed toward determining the systematic uncertainty and imprecision of k. Errors due to the inapplicability of the definition of k and material variations (inhomogeneity) will be ignored, i.e., experimental measurement and control uncertainties will be the sole consideration.

CARD NO.

Start in Col. 1 with run number:Suffixes;R1, R2, . .=repeat runs; OGO=outer guard off; SU,SL=Single sided run (upper,lower); X=invalid for conductivity, NMS=No Mini-Shield; P=preliminary data (before Nov., 1979); Q=data questionable based on analysis.

(run number=maximum of 7 characters - use no blanks, start in col. 1)	CARD 1

Blank	specimen/material		date (yr,mo,da)	time	run number	
THERMAL CONDUCTIVITY DATA FOR						CARD 2

CODES:

Mini-shield:
0=not present
1=present

Plate Type:
0,1,2=low temperature
(0 2 after 7/20/81)
11,12=high temperature

Spacer Matl:
0=quartz
1=S. Steel

Res. in Power Circuit:
0=Div. Netw. (prec.)
1=No Div. Netw. (prec.)
2=Div. Netw. (not Prec.)

Apparatus Parameters

Plate Spacing-cm	Eff. M.H. Dia.-cm	Plate emiss.		Mini-shld	Plate type	Spcr. Matl.	Prec. Res.			
										CARD 3
1	11	21	31	41	46	51	56	61	66	

Gas Code: 0=Nitrogen; 1=air; 2=He; 3=Argon
Lot No.: Fiberboard=58,59,61,70,78
Fiberblanket=79

Mtl. Code:
0=fiberboard
1=fiberblanket

Specimen Parameters

Pressure (mm Hg)	Area Dens (g/cm^2)		Gas Code	Lot No.	Mtl. Code		
							CARD 4
1	11	21	31	36	41	46	

Thermocouple and Power Emf's (mV)/(Switch positions - S1-A must be in "0")

DTC1615 S1-B (2)	DTC1718 S1-B (3)	TC15 S1-B (5)	TC16 S1-B (6)	TC17 S1-B (7)	TC18 S1-B (8)	Htr.volt. S1-B (9)	Htr.curr. S1-B (10)	
								CARD 5
								CARD 6
								CARD 7
1	11	21	31	41	51	61	71	

Figure 15. Thermal Conductivity Data Form (Side A)

OPERATOR ____________

REFERENCE: Laboratory Book No. _____ Pages: ________

ENVIRONMENT: Stack-to-shroud insulation: Vermiculite __ , Other (_______________)
Aux. Htr. Offset insulation: Fiberglass __ , Other (_______________)
Coolant: Water __ ; LN __ ; Other (_______________)

SPECIMENS: Conditioning: _______ hrs at _____°C

SPECIMEN CODES: bottom ________; top ________
SPECIMEN GUARD CODES: bottom ________; top ________

Inner Grd.: Range: _______μV Noise BW _______(μV)
TAH: _______ _______
BAH: _______ _______

Wheatstone Bridge TAH _____________
BAH _____________
Outer Grd: Dial Setting: _________

ICE REF. FIXED AT _____________________

IG/MH TP _________μV Top (+ to mid)
_________μV Bottom (- to mid)

TIME:				REMARKS
(For all these TC readings S1-B must be in "0" position)				
S1-A 1-(TB-BJ)				
Pos. 2-(Cool't)				
MV 3-(TB-CAB)				
μV 4-(KN-KN')				
5-(KP-KP')				
6- (9-18)				
7-(10-17)				
8-(11-16)				
9-(12-16)				
10-(13-15)				
11-(14-15)				
FLUKE CURR (MA)				

Figure 16. Thermal Conductivity Data Form (Side B)

Table 2. Outline of computer program for analysis of thermal insulation data

1. Read experimental data
2. Compute main heater power
3. Compute temperatures of all thermocouples from calibration equation
4. Compute temperature differences
5. Compute mean temperatures
6. Correct specimen area for thermal expansion
7. Correct specimen thickness for thermal expansion
8. Compute average effective thermal conductivity from equation 1
9. Print and store results for further analysis

The operational definition for k, eq (1), shows that the uncertainties to be considered are those in the control and measurement of V, I, ΔX, A, and ΔT. Since k is usually associated with the mean temperature, $\overline{T}$, its determination also contributes to the total uncertainty. Probably the most subtle and difficult uncertainty to estimate is that of the heat flow through the specimen, $Q = VI$. This difficulty arises not through the inaccurate measurement of V and I but rather mainly through the correct establishment of (a) stable and (b) unidirectional heat flow. The second most difficult aspect of this measurement is the correct determination of the temperature difference across the specimen. Again this is not caused primarily by calibration or read-out shortcomings, but rather, due to the uncertainty in the effective plane of the thermocouples, the discontinuous nature of the specimen-plate interface, and deviations from planar isotherms. Estimates of random (imprecision) and systematic variations in the measured and calculated quantities for a typical run are listed in table 3. The bottom line of the estimates in table 3 indicates an estimated imprecision, 2σ, of about ±0.4% and a systematic uncertainty of near ±0.6%. This imprecision is approximately equal to the scatter in the data as reported in publications on glass fiberboard and glass fiberblanket [1,2].[1] The overall uncertainty of ±1% cannot be confirmed or refuted on the basis of comparison with certified data for the glass fiberboard (1.5% disagreement), since the certified data are uncertain by ±2%.

Table 3 contains uncertainty estimates for room temperature measurements. To make similar estimates for liquid nitrogen temperatures, one notes that both the measurement and control thermocouples are less sensitive by approximately a factor of two. This decrease in sensitivity, combined with a reduction in heat

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[1] Numbers in brackets indicate literature references at the end of this paper.

Table 3. Estimates of random variations and systematic uncertainties in the measured quantities at room temperature.

Variable	Absolute Variations		Percent Variations	
	random	systematic	random	systematic
V_{Htr}	200 μV	200 μV	0.01	0.01
I_{Htr}	20 μA	20 μA	0.01	0.01
$Q = (VI)_{Htr}$	60 μW	60 μW	0.01	0.01
Q(drift)[a]	1 mW	1 mW	0.2	0.2
Q(radial)[b]	1 mW	1 mW	0.2	0.2
Q(total)	2 mW	2 mW	0.4	0.4
V_{TC}(calib.)	0	1 μV	0	0.1
V_{TC}(meas.)	0.5 μV	2 μV	0.05	0.2
V_{TC}(radial)	0	2 μV	0	0.2
V_{TC}(total)	0.5 μV	5 μV	0.05	0.5
$\overline{T}$	0.02 K	0.2 K	0.007	0.07
ΔT	0.01 K	0.1 K	0.04	0.4
ΔX	0	0.05 mm	0	0.2
A	0	30 mm^2	0	0.2
k	0.2 mW/m·K	0.3 mW/m·K	0.4	0.6

[a]Effect of drift of the main heater plate as calculated from heat capacity and mass (corroborated by experiment) = 0.4% error in Q at a drift rate of 1 μV/h. which equals a 0.016% error in Q for a drift rate of 1 mK/h.
[b]Effect of MH/IG gap offset as measured experimentally (corroborated by calculation) is a 0.04% error in Q per μV of thermopile offset which also equals a 0.04% error in Q per millikelvin of gap temperature offset.

flow by a factor of four (for typical glass fiber insulations), results in an imprecision of about 2% and a systematic uncertainty of about 3%. For intermediate temperatures the imprecision and systematic uncertainty are estimated by linear interpolation of these extremes. These uncertainties in k have been calculated by first summing the absolute values of the errors in Q and V and then by calculating the square root of the sum of the squares of the uncertainties in the variables of eq (1). The latter calculation is justified since the errors in these variables are experimentally uncorrelated, i.e., they are measured by distinctly different instruments, except for $Q = (VI)_{Htr}$, but that component of uncertainty is small.

Corroborating experiments to support some of the estimates in table 3 have been performed. For example, the resistance of the main heater (metered section) has been calculated from V/I and plotted versus temperature. The scatter in the resistances cover a range of ±0.03% as compared to the estimated value for the scatter in VI of ±.02%. Offset runs of the inner and outer guards have been conducted to determine the sensitivity of the results to potential offsets within detectability limits. The effect of intentionally introduced drifts in the main heater plate has also been studied. Errors due to drift and offset are shown at the bottom of table 3. These experiments tend to confirm the validity of the estimates in table 3 as upper limits to our total error.

A potential systematic error due to deviations from uni-axial heat flow in the metered region is not estimated. However, it is noted that the ratio of specimen thickness to the metered diameter is 1:4 which is usually accepted as adequate to reduce this error to below 1%. In addition, the use of the aluminized mylar mini-shield surrounding the MH/IG assembly should further reduce this systematic bias. For further information on this subject the reader is referred to the work of Woodside [3].

5. SUMMARY

This report describes the modifications and performance of a guarded-hot-plate apparatus designed and built to meet the specifications of the ASTM C-177 test method. The purpose of these tests and modifications was to produce an apparatus which could be used for state-of-the-art standard reference material measurements. The major modifications performed are listed below in the order of importance to precision and accuracy:

I. Improved the stability of the temperature controllers on the TAH and BAH.

   a) Replaced unstable low signal level amplifiers with amplifiers having sub-microvolt stability. These null detector amplifiers also provided the operator with a clear indication of the system stability.

   b) Replaced temperature control thermocouples with absolute resistive sensors. This eliminated the effect of drift from an ice reference bath or an electronic ice reference as well as reduced the effect of spurious emf's on the stability of the system.

   c) Installed an adjustable power delivery network to better match the power required by the TAH and BAH for more stable operation.

II. Improved the sensitivity and stability of the MH/IG gap controller.

   a) Same as Ia above.

   b) Replaced the original 8 pair Type K thermopile with a 20 pair Type E thermopile. This resulted in a three fold improvement in control sensitivity of the gap offset.

   c) Same as Ic above.

III. Replaced power and measurementation instrumentation with highly stable and accurate devices.

   a) Replaced the main heater power supply with a unit having at least ten-fold improved stability and accuracy.

b) Replaced DVM and power measuring network with units having at least ten-fold improved stability and accuracy.

c) Replaced all original thermocouples with accurately calibrated thermocouples, replaced electrically noisy selector switches with high quality sub-microvolt selector switches, and improved the thermal anchoring of thermocouple wires to reduce spurious emf's.

IV. Provided improved isolation of the stack and coolant paths for increased efficiency of operation.

a) Increased the spacing and insulation between the cold plates and shroud and the warmer parts of the system.

b) Installed vacuum-insulated coolant lines.

c) Replaced the bell-jar environmental chamber with a vacuum insulated dewar.

The significance of the improvements described in this report can be appreciated by noting that the initial apparatus according to preliminary tests, produced results which varied by ±15%, and greater under some circumstances. The improvements not only decreased the scatter and uncertainty to about one-fourth their original values, but also provided the operator with an increased capability to detect when the system is not operating properly.

6. REFERENCES

[1] D. R. Smith and J. G. Hust, Measurement of Effective Thermal Conductivity of a Glass Fiberboard Standard Reference Material, to be published in Cryogenics.

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[3] W. Woodside, "Analysis of Errors Due to Edge Loss in Guarded Hot Plates", ASTM STP 217, American Society for Testing and Materials, 1957, pp. 49-64.

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D. R. Smith, J. G. Hust, and L. J. Van Poolen

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10. SUPPLEMENTARY NOTES

[ ] Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

This report describes a guarded-hot-plate apparatus used to determine the effective thermal conductivity of glass fiber insulations. Measurements can be performed at temperatures from 80 K to 360 K, from atmospheric pressure to a vacuum of 10^{-4} Pa (1×10^{-6} torr). Various fill gases such as air, nitrogen, argon, and helium can be utilized. Overall uncertainties of thermal conductivities at atmospheric pressure are 1% at the higher temperatures and 5% at the lower cryogenic temperatures. The modifications of the commercial apparatus described in this report resulted in approximately a four-fold improvement in uncertainty.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

Guarded-hot-plate apparatus; insulation; low-temperature; thermal conductivity.

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