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EVALUATION OF THE THERMAL PROPERTIES OF MATERIALS

Final Report
Volume I
Technical Report

Prepared by

AVCO CORPORATION
AVCO SPACE SYSTEMS DIVISION
Lowell, Massachusetts 01851

AVSSD-0197-66-CR
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29 June 1965
28 June 1966

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Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MANNED SPACECRAFT CENTER
2101 Webster-Seabrook Road
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FOREWORD

This report was prepared by Avco Corporation, Space Systems Division, Lowell, Massachusetts under NASA, MSC Contract Number NAS 9-4874, dated June 29, 1965. The time period of performance of this contract was June 29, 1965 to June 28, 1966. This report is of work performed by Avco SSD during the course of the contract and managed by Michael E. Ihnat of the SSD Materials Development Department. The technical monitor for the National Aeronautics and Space Administration, Manned Spacecraft Center located in Houston, Texas was John W. Orsag of the Structures and Mechanics Division, Thermal Technology Branch.

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ABSTRACT

This report presents the results of measurements of the thermophysical properties of materials used for NASA manned spacecraft. Unlike a specific-application component study, the program discussed in this report was diversified. The purpose of the experimental program was to provide the thermal properties of structural materials, ablators, insulations, seals, and adhesives for manned spacecraft. The environments considered were atmospheric pressure and vacuums to 10^{-5} torr, and some materials were evaluated both in the virgin and charred state. The temperature range of measurement was from -250 to 2000°F.

The parameters evaluated were apparent thermal conductance, apparent thermal conductivity, enthalpy and specific heat, density, weight loss, reaction energies, heat of combustion, viscous and inertial resistance coefficient, optical reflectance, material transient response, anistropy of various parameters, char temperature, and prechar procedures.

For convenience, the report is divided into two volumes:

Volume I, Technical Report
Volume II, Data Handbook

The first volume discusses the measurement techniques, program context, material variations and peculiarities, calibration procedures and results, ASTM procedures, and the program conclusions. The second volume presents all of the raw data, analysis, correlations, and material compositions, and can thus be used as a reference handbook.

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1.0 INTRODUCTION

This report is of a diversified study of the thermophysical properties used for NASA manned spacecraft. The purpose of the reported study was to record thermal properties of structural materials, ablators, insulations, seals, and adhesives. The environments considered were atmospheric and vacuum (to 10^{-5} torr) at temperatures of -250° to 2000° F. Selected materials were evaluated both in their virgin and charred state. Volume I of this report presents a discussion of techniques and procedures, program context, test results, and conclusions. For in-depth tabulations and illustrations of raw data, analysis, correlations, and material composition, refer to Volume II, Data Handbook, of this report.

The material application catagories and the materials studied under each were as follows:

a) Structural Materials

1) Aluminum honeycomb panels
2) Stainless steel honeycomb panels
3) Epoxy laminated fiberglass
4) Phenolic laminated fiberglass
5) HT-424 laminated fiberglass
6) Marinite

b) Heat Shield Materials

1) Avcoat 5026-39
2) DC-325
3) NASA Langley-Purple Blend
4) Armstrong Cork A 2755
5) Armstrong Cork A 2755, fabricated as ascent heat shield composite
6) Teflon
7) Polyethelene

c) Insulations

1) Thompsaglass 15, 000 (TG 15000)
2) SI-62
3) NRC-2

d) Seal and Adhesive Materials

1) Epon 931
2) RTV 560
3) HT-424
4) Sylgard 182-2

The materials are listed above by their standard designations. In Volume II of the report, the materials are identified by supplier and composition. Several of the materials were not defined except by name and supplier because the composition or fabrication process was proprietary.

The primary parameters evaluated during the program were thermal conductance, thermal conductivity, enthalpy-specific heat, and density. Thermal conductance was restricted to honeycomb panel measurements, and, with the exception of bulk density, none of the other parameters applied. The other primary parameters applied to all the remaining materials.

The environmental conditions, described above, did not apply to all materials, since the conditions are defined or limited by their use in the application. In most cases, the parameters were evaluated above their recommended maximum use temperature. The over-temperature criterion was used to determine the effect of such a condition if it did occur during application. Enthalpy and specific heat of any material was not determined under vacuum conditions since evaluations of this type would not produce useful data.

Such secondary parameters as weight loss, reaction energy, heat of combustion, viscous and inertial resistance coefficient, char temperature, and prechar properties were required to support the primary parameter measurements of heat shield materials. Some were also performed as support measurements of the other material types. Optical reflectance, transient response, and anistropy were support measurements applying to aluminum honeycomb panels.

Thermal conductivity and conductance tests were performed using guard hot plate techniques, adhering as closely as possible to ASTM C-177-63[1] procedures. Additional instrumentation was incorporated into the apparatus to present data verifying one dimensional heat flow in very thick metal honeycomb samples. Where there was concern that face-plate thickness on honeycomb sandwich panels would affect measurements, two piece specimens were used. A two piece specimen introduces a 1/8-inch gap between the test section and the guard ring. The specimen in this form is then in the configuration of the main and guard heaters. Tests were performed on metal honeycomb materials, using 0.008-and 0.015-inch face plates in a single and two-piece specimen configuration; no deviations in test data were detected, indicating that the face plate acts as a distributor not affecting the parameter measurement.

Specific heat, as derived from change in enthalpy data, was measured using drop calorimetry essentially as described in ASTM procedure C351-61.[2] Modifications were made to the Avco apparatus to accept automation and improve the reliability of the data obtained. The modifications are a deviation from the basic operations of the procedure; these, however, do not cause non-adherance to the procedure. A second specific heat technique was evaluated for use in this contract. The technique was a transient one, using differential scan calorimetry. It was possible with differential scan calorimetry, once firm calibrations were

established, to obtain data during transitions in diathermous materials and to provide specific heat data in place of drop calorimeter methods. The transient method provides more information, since specific heat data points are provided at every 10 degree interval, thus, reaction energies can be quantitatively measured.

Vacuum thermal conductivity and conductance tests proved to be the usual problem. Because a-priori information on the stability of the test materials in a vacuum environment at temperature was not known, several phenomena were encountered. Careful attention was placed on these phenomena when they appeared. These phenomena are discussed in detail where applicable in this report.

All data analysis for this program was performed statistically. Thermal conductivity and conductance tests were fitted by least-squares techniques; property values are cited at specific temperatures. When sufficient tests were performed, the property values were further analyzed to determine the arithmetic mean and standard deviation.

Specific heat, as measured by drop calorimetry and represented by the slope of the ΔH versus temperature curve, can be variously interpreted, depending upon the method used to fit the curve to the data points. In this program, specific heat was determined by a Gerber Derivimeter. Specific heat data were analyzed similarly to thermal conductance and conductivity data as discussed in the previous paragraph.

At intervals during the program, all test apparatus were checked with available NBS secondary standards. These verifications included NBS certified thermal conductivity samples, synthetic sapphire boules as specified by the Bureau for enthalpy determination, and standard melting point metals for evaluation of temperature calibrations and energy of reaction. All calibration and verification test data obtained during any period of this contract are reported. When a sufficiently established standard was not available, as was the case with thermal conductivity values between 0.5 Btu/hr-ft-°F and those of Armco Iron (39 to 17 Btu/hr-ft-°F), the laboratory used such stable materials as zirconia, (1.2 to 1.3 Btu/hr-ft-°F) and Pyroceram 9606 (3.2 to 1.6 Btu/hr-ft-°F). These materials were measured; the results were compared with many reference published values. The materials were used as a means of measuring the repeatability of tests on which a curve and deviation was established. Sufficient numbers of data obtained from repeated long term verification tests were used to establish the curve and its deviation.

Although, synthetic sapphire boules were used for enthalpy measurements, this is not a direct reference material. In boule form, the material is pure Al_2O_3 single crystals. The crystals which were used were ones which had been broken, during their manufacture, thus became unacceptable for commercial use. This form of the material was used by Ginnings and Furakawa of NBS[3] to establish a reference enthalpy versus temperature curve. The material as related to the

contract test procedures was used to establish and check variations of calorimeter equivalents in the method-of-mixtures technique of specific heat measurements. It was also used for deflection calibration for Differential Scanning Calorimeter techniques.

A preliminary study of charring ablators was performed to provide the support information required for an interpretation of the primary parameter behavior when the material is tested beyond temperatures where changes in the virgin composition and structure occur. The tests performed were weight loss, by thermogravimetric analysis; heat of combustion by Parr Bomb calorimetry; and mass-flow coefficients determination with an Avco designed apparatus. These tests included prechar procedures to predetermined temperatures. The study was also required to provide a more useful parameter for an advanced charring ablator model being developed by NASA MSC during the course of this contract.

Optical property measurements were used for comparative evaluation of aluminum honeycomb adhesives. These measurements were performed with a Beckman Model DK-2 spectrophotometer having an integrating sphere attachment.

Transient response measurements of aluminum honeycomb panels were made, but because these were requested late in the program, there was insufficient time to perform analyses and draw conclusions from these measurements. The primary purpose of these measurements was to obtain time-temperature data when experimental arrangements, sources, and instrumentation techniques were varied. The resulting curves are intended for qualitative analysis only.

ASTM recommended practices were used throughout. Although all practices are not specifically designated at any point in this report, those used are listed and abstracted in the appendix.

2.0 TECHNICAL DISCUSSION: MEASUREMENT TECHNIQUES

2.1 GENERALIZED THEORY

A brief presentation of the relationships which apply to the parameters discussed in the following section is necessary so that the limitations, when these parameters are used in calculating data, are realized. Thermal conduction principles are discussed using Fourier's Law; elaborations of its applicability are made when radiation transport exists, (a departure from Fourier's boundary condition of an opaque solid.) Discussions of the theory of specific heat exist in the literature. These discussions deal with such idealized conditions that, for this report, the theory has been deleted. The theory of specific heat is still at the point where calculations cannot be made for most solid substances. A definition of specific heat was accepted for this report[2]: The mean specific heat is an essential property of a material when this material is used under conditions of unsteady or transient heat flow. It is part of the parameter thermal diffusivity, which governs the rate of temperature diffusion through a material. It is a basic thermodynamic property of all substances; its value depends upon chemical composition and temperature.

2.1.1 Fourier's Law -- True Conduction

Fourier (1768-1830) was the first to present a mathematical theory of heat transfer in opaque solids by conduction. Conduction, in this sense, is a transfer of energy by a molecular process. In differential form Fourier's Law for one dimensional heat transfer is:

$$\frac{\partial Q}{\partial t} = - k A_n \frac{\partial T}{\partial n} \qquad (1)$$

where

$\partial Q/\partial t$ = instantaneous time rate of heat flow

k = material conductivity at that instant of time

A_n = cross-sectional area normal to axis of heat flow

$\partial T/\partial n$ = instantaneous temperature gradient along the axis of heat flow.

The negative sign is necessary because heat flow is a vector quantity and thus has direction. If we define the heat flow as $\partial Q/\partial t = q$, and the normal to the axis as the x plane we can rewrite equation (1) as:

$$q = - kA \frac{\partial T}{\partial x} \qquad (2)$$

If steady-state conditions exist

$$q = -kA \frac{dT}{dx} \tag{3}$$

or

$$qdx = -kA\,dT \tag{4}$$

For the usual case, we can integrate this equation directly using the assumptions:

a) Steady-state or q = constant

b) k = constant

c) A = constant

$$\int_0^l qdx = \int_{T_1}^{T_2} -kA\,dT \tag{5}$$

$$(ql) = -kA(T_2 - T_1)$$

or

$$q = -kA \frac{T_1 - T_2}{l} \tag{6}$$

It is reasonable to say that (conductivity being constant) a small temperature interval is required to make the relationship valid for nonlinear conductivity curves. When significant variations of conductivity exist and/or if the temperature extremes are excessive, it is necessary to consider the conductivity-with-temperature variation; this is usually overlooked. An approximation which is used when variations are encountered is the arithmetic mean value of conductivity between the temperature extremes. The mean can be expressed as:

$$k_m = \frac{1}{T_2 - T_1} \int_{T_1}^{T_2} k(T)\,dT \tag{7}$$

where $k(T)$ is usually of the form:

$$k(T) = k(1 + a_1 T^2 + a_2 T^2 + a_3 T^3 + \ldots\ldots a_n T^n) \tag{8}$$

In this case, one can substitute the exact value of $k(T)$ from equation (8) for k from equation (4) and solve Fourier's Law rigorously. It is obvious that the maintenance of small temperature differences over the range of evaluation will provide more accurate results for all cases.

Recognition of these conditions prompted the procedure that the tests whose results are reported in this document were measured in apparatus containing auxiliary heaters. The inclusion of auxiliary heaters was a deviation from ASTM C 177-45;[2] this has, however, been included in the most recent ASTM C 177-63 specification. The equipment was operated with gradients greater than the 40°F/in. minimum and less than a maximum of 120°F/in. to obtain a thermal conductivity versus temperature relationship.

2. 1. 2 Apparent Thermal Conductivity

When they are experimentally measured, non-opaque materials, including many of the heat shield materials (plastics and plastic laminates) and good insulators (mat, fiber, and powdered materials) can be reported only on the condition that the measured value is "apparent". The designation of "apparent" arises from the fact that with true conduction, radiation and convective transport may exist.

Genzel[4] has derived an expression for apparent thermal conduction which is:

$$k_A = k_L + \frac{16\,\sigma\,n^2}{3a}\,T^3 \tag{9}$$

where

k_L = true thermal conduction

σ = Stefan-Boltzman constant

n = index of refraction

a = absorption coefficient (assumed to be gray)

T = temperature

This relationship is especially useful for estimating the effect of radiation on total thermal conduction. Figure 1 illustrates an approximate solution for within certain bounds and without true thermal conduction and absorption as a parameter. The figure represents the relationship:

$$T^4(z) = \frac{T_o^4\,(2 + 3\,an)\,2\,T_h^4}{4 + 3\,ah} - \frac{3(T_o^4 - T_h^4)}{4 + 3\,ah}\,az \tag{10}$$

Genzel has also defined a finite true thermal conductivity expression. Thus, with fixed thermal conductivity coefficients, k_L, and with different absorption values, a, Figure 2 was obtained. The general character of the curves provides that--

a) The temperature distribution at the inner part of the curve varies as $4\sqrt{z}$.

b) At the faces, the curves satisfy the boundary values T_o and T_h.

c) For every k_L chosen, there is a critical, a, value at which the deviation of the curve from the straight line ($a = 0$ and ∞) becomes a maximum.

d) The smaller the k_L value the larger is the maximum deviation.

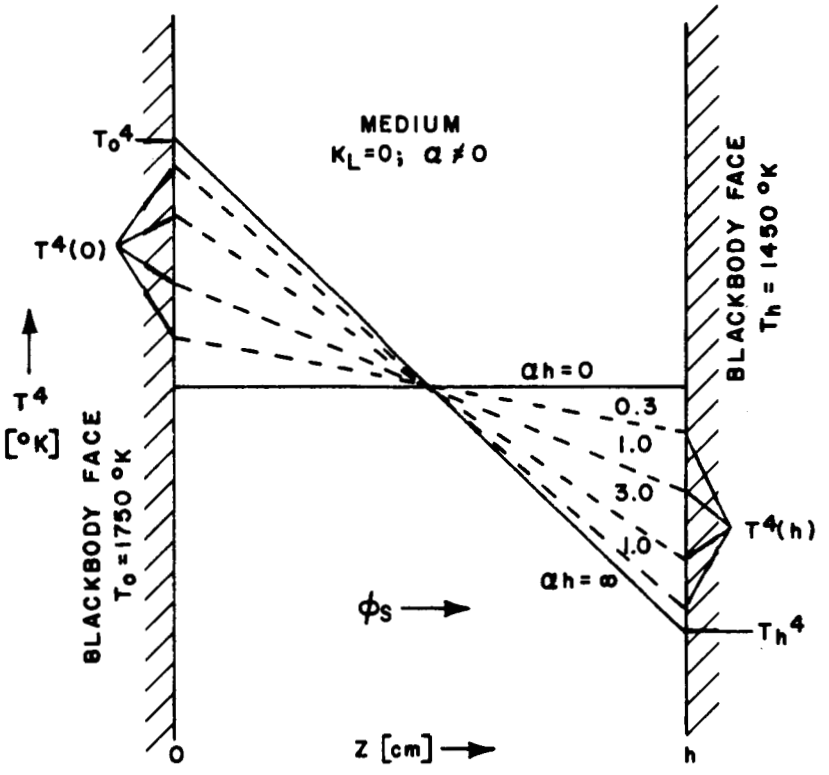


Figure 1 EQUATION 10 SOLUTIONS, ASSUMING ABSORPTION A AS PARAMETER

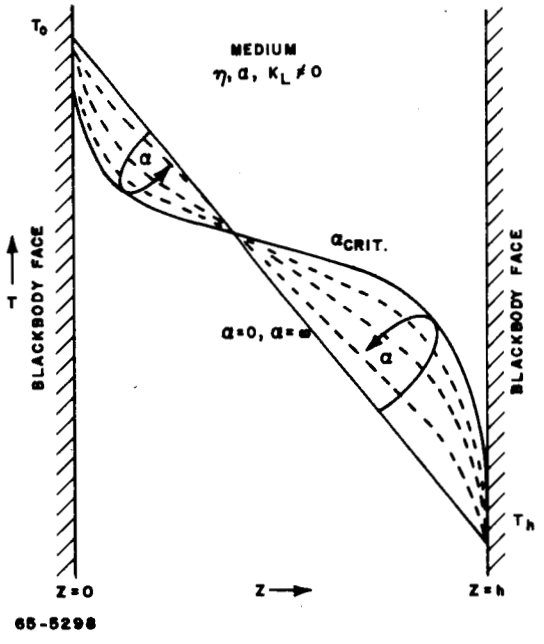


Figure 2 TEMPERATURE DISTRIBUTION IN A THERMALLY CONDUCTING MATERIAL AT VARIOUS LEVELS OF ABSORPTION COEFFICIENT

2.2 TEST APPARATUS: PRIMARY PARAMETERS

The discussions of the various apparatus used for the reported measurements are preceded by an abstract of the related ASTM specification so that adherence or deviations from the specifications may be noted. The apparatus specifications, as they exist at the time of this report, are abstracted for brevity and clarity. A direct statement quoted from the procedures: "If the results are to be reported as having been obtained by this method, then all pertinent requirements prescribed in this method shall be met."[2].

In the specification abstract, a description of the apparatus used for the measurements has been given with particular emphasis on adherence or deviation from the specification during this contract. These descriptions have been included because of their importance when data comparisons are made.

2.2.1 Thermal Conductivity of Materials by Means of the Guarded Hot Plate (ASTM C-177-63)[1].

The procedure covers two types of guarded hot-plate apparatus. The first is a metal-surface guarded hot plate, generally recommended for measurements at mean temperatures from -100 to 500°F. The second apparatus is a refractory guarded hot plate recommended for mean temperatures of 200 to 1300°F. Through certain modifications, for this program the overall range of the guarded hot plate was extended to -260 to 2100°F. The temperature range is a deviation from ASTM C177-63; therefore the tests were performed on a modified apparatus that is nonconforming to the specification. The modification was an increase of insulation surrounding the apparatus (specified to be 12 inches greater in diameter than the assembly diameter) to ensure that the radial thermal resistance was adequate to guarantee unidirectional heat flow.

The primary difference between the two units discussed above is that the refractory guarded hot plate contains more functional items in the test stack than the metal surface hot plate. The additional items listed in sequence from the specimen cold-face side are: a second surface plate (distributor), an auxiliary heater, and insulation. (See Figures 3, which shows a schematic arrangement, Figure 4, an external view, and Figure 5, an internal view of the apparatus). The auxiliary heaters allow an adjustment of the temperature differential across the specimen, whereas, in the metal surface type, the differentials are regulated by the temperature of the cooling fluid in the end plates.

Table I presents a comparison of the mechanical specifications of the ASTM procedure with those used in Avco apparatus. It will be noted that the test apparatus used conforms in every case. In addition to the thermocouple units specified, there were three pairs of differential thermocouples, which monitor the radial temperature gradient and aid in ensuring unidirectional flow. Figure 6 shows schematically the function and number of thermocouples used in one apparatus.

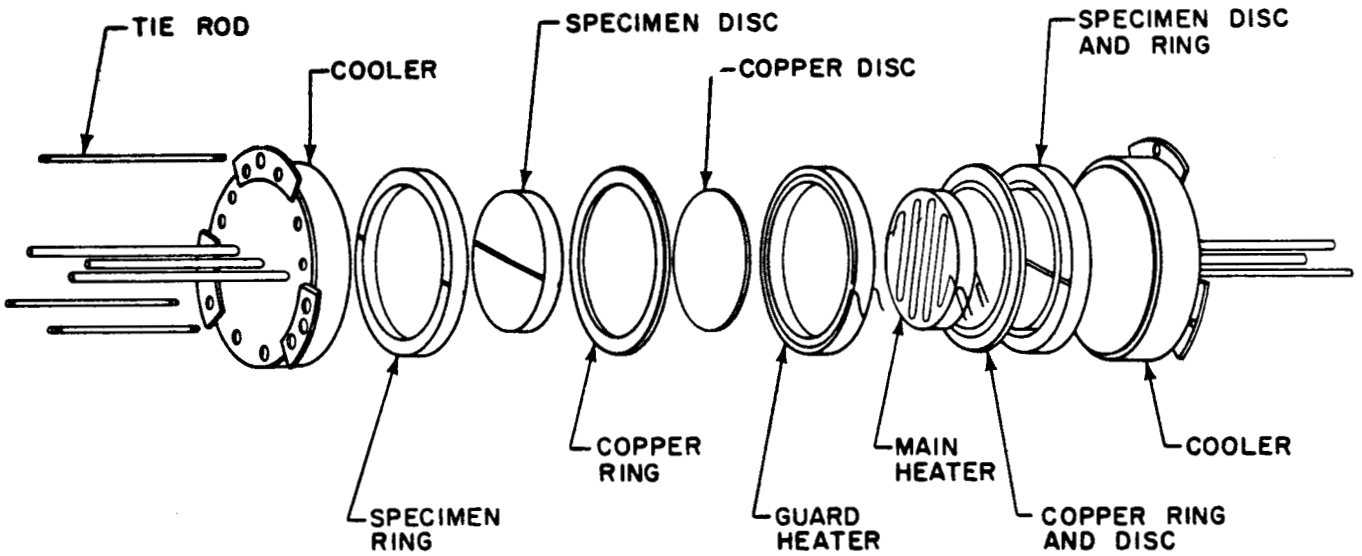


Figure 3 SCHEMATIC OF THERMAL CONDUCTIVITY APPARATUS

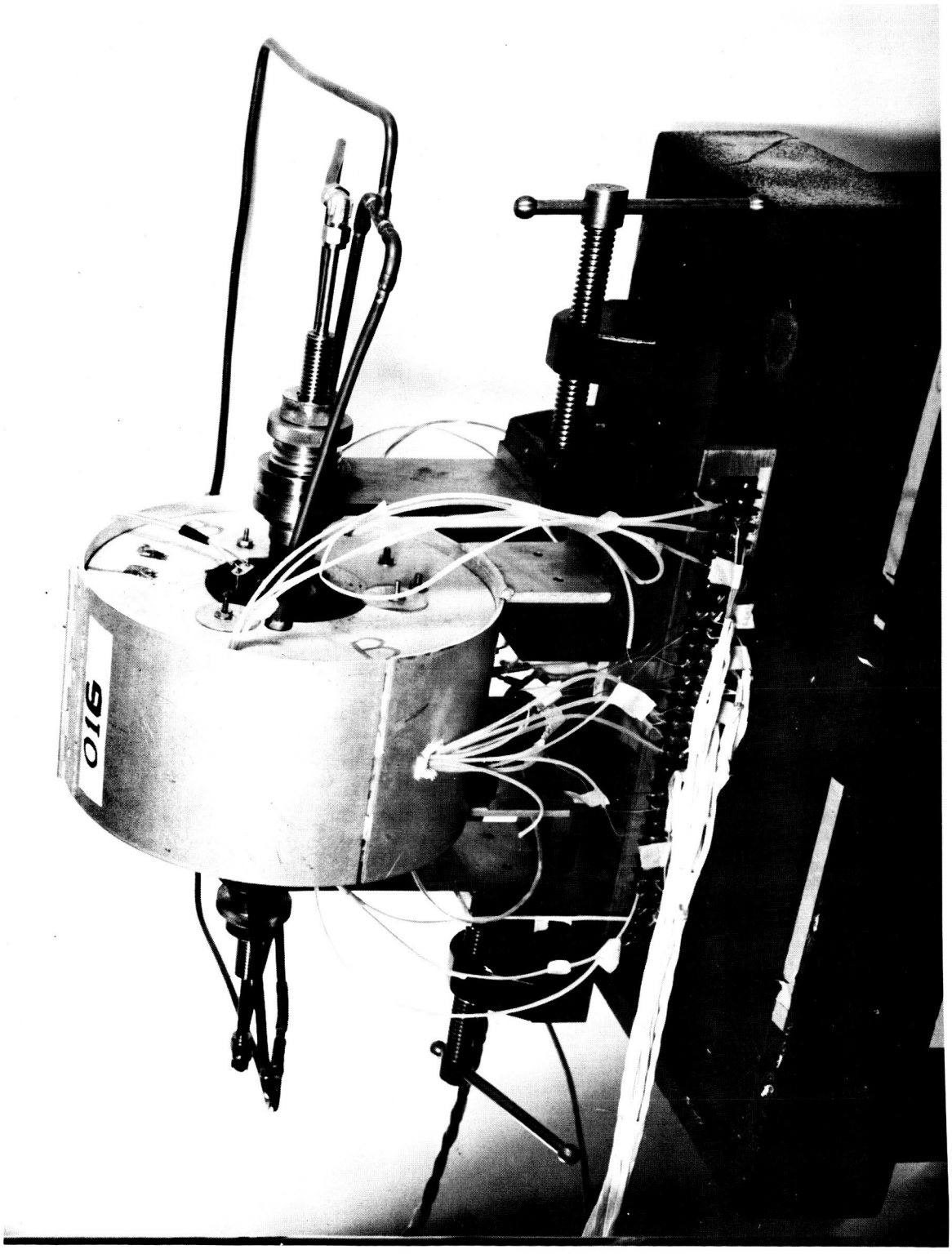


Figure 4 EXTERNAL VIEW OF GUARDED HOT-PLATE APPARATUS

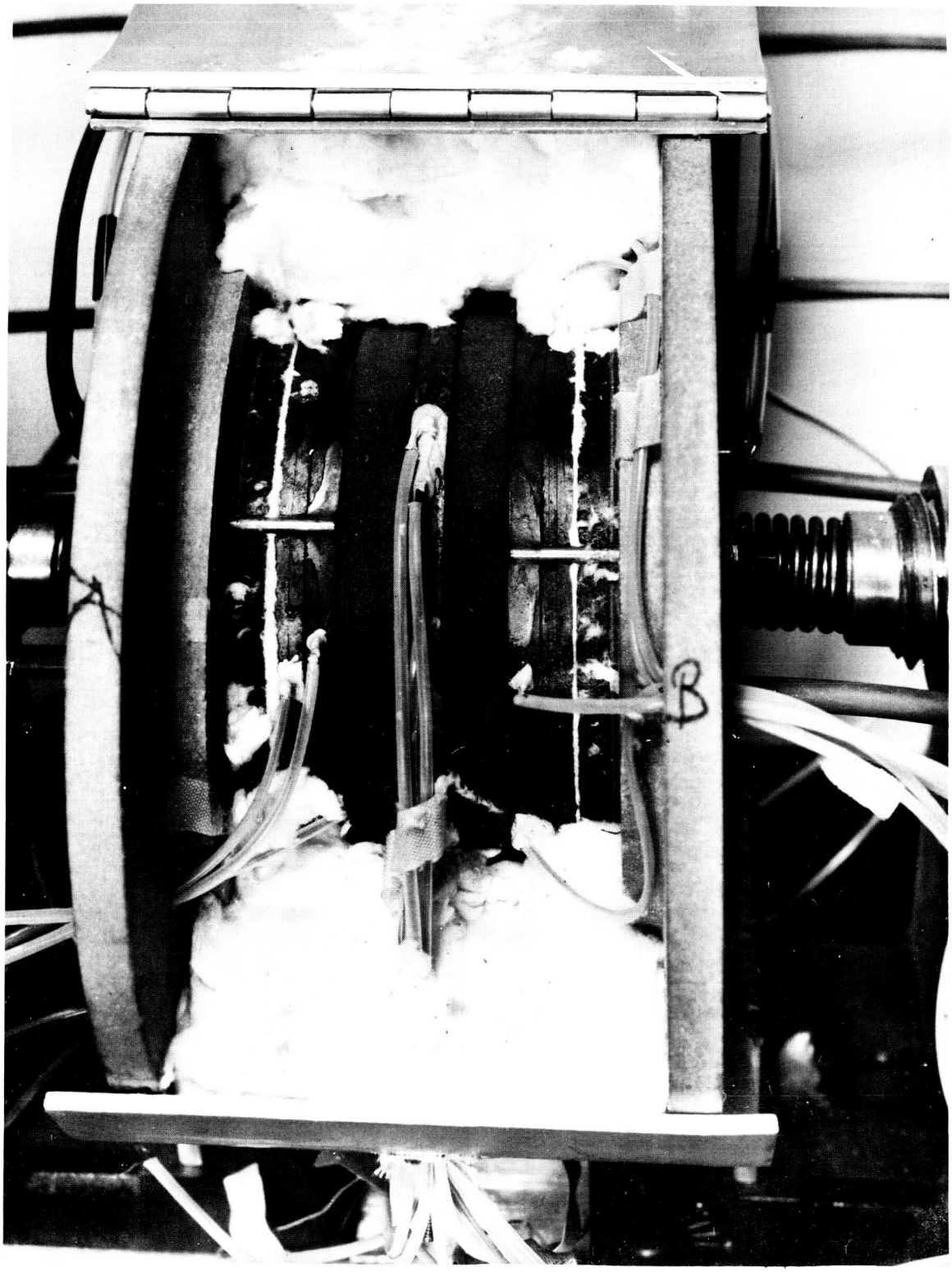


Figure 5 INTERNAL VIEW OF GUARDED HOT-PLATE APPARATUS

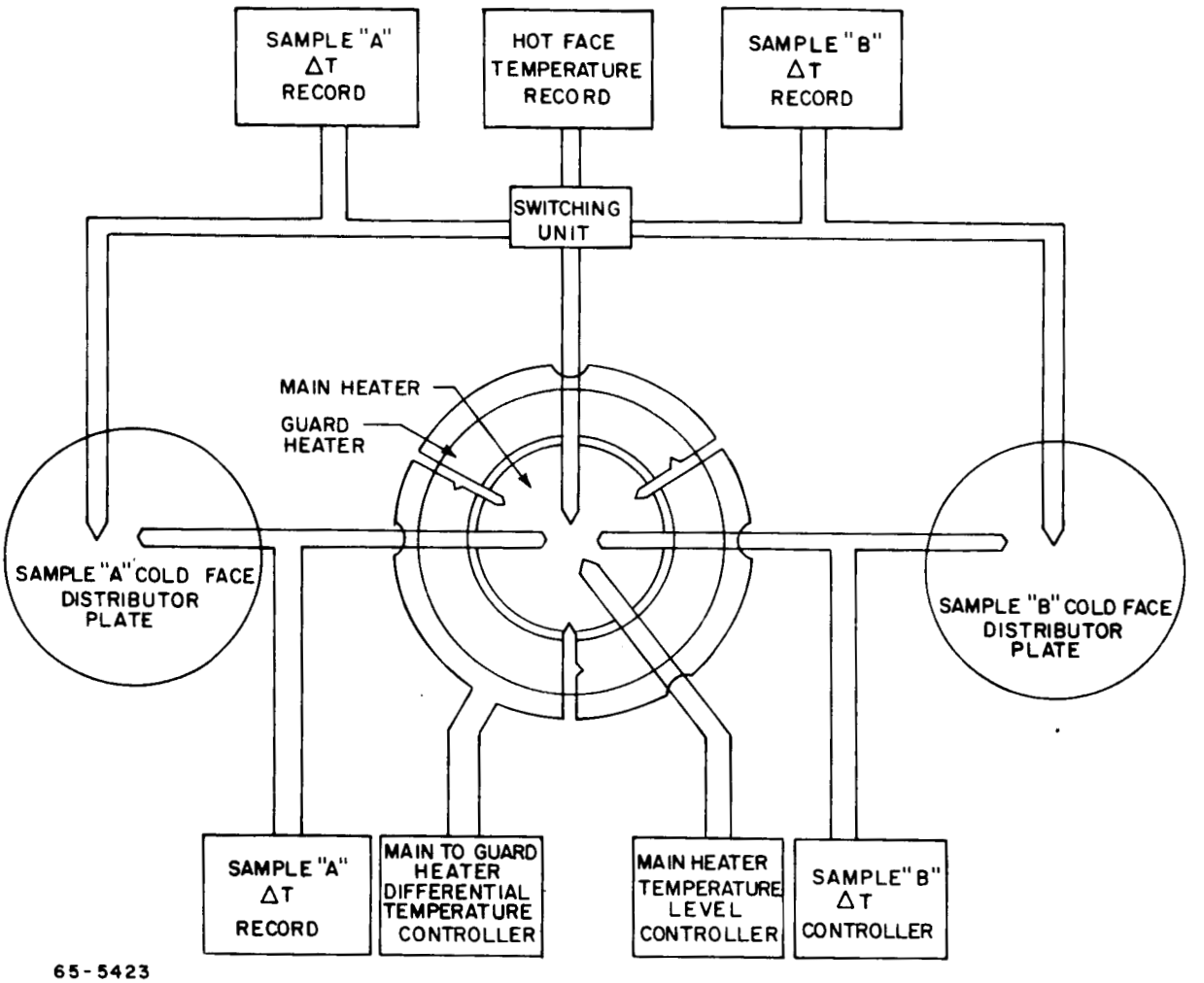


Figure 6 AUTOMATIC GUARDED HOT-PLATE THERMOCOUPLE SCHEMATIC

TABLE I

MECHANICAL SPECIFICATIONS OF ASTM C-177-63 WITH STATEMENT OF CONFORMANCE BY AVCO SSD

No.	Specification	ASTM	Avco
1	Maximum departure of surfaces from a plane	± 0.003 in. /ft	± 0.0005 in.
2	Maximum gap between central surface plate and guard plate	0.125 in.	0.125 in.
3	Maximum separation between heating windings of central section and guard section (heavy copper)	0.750 in.	N/A
4	Maximum separation between heating windings of central section and guard section (other materials)	0.125 in.	0.125 in.
5	Cooling and heating surface areas	Equal in size	Conforms. See Figure 3
6	Maximum diameter of thermocouple wire for surface plates (metal-face system)	0.0226 in.	0.005 in.
7	Maximum diameter of thermocouple wire for surface plates (refractory system)	0.0253 in.	0.005 in.
8	Maximum specimen surface thermocouple wire (metal-face system)	0.0113 in.	0.005 in.
9	Maximum specimen surface thermocouple wire (refractory system)	0.0179 in.	0.005 in.
10	Permanently installed thermocouples	In grooved surface plates	Conforms. See Figure 3
11	The number of thermocouples in the surface plate shall not be less than 1/8 $A_{sp_2} = N_{sp}$	0.515 unit	1 unit
12	The same number will be installed at corresponding position in facing cold plate		Conforms. See Figure 6
13	For rigid specimens, it <u>may</u> be important to place thermocouples in face of specimen. The number of thermocouples shall not be less than 1/4 A_{m_2} Area of Avco metering location = 3.973 in.^{2}	0.50 unit	1 unit
14	The refractory guarded hot plate shall be surrounded with coaxial insulation at least 12 inches greater than the assembly diameter		Conforms. See Figure 5

Table II is a compilation of the parametric specifications. Table III presents the specimen specifications. It will be noted that item 1 of Table III specifies homogeneous and opaque materials; thus, any reference "apparent" thermal conductivity would be inconsistent with the specification, and the term "modified" must be used in reference to non-opaque materials.

Table IV presents the ASTM-specified equations and symbols for calculations of the various parameters. Equation (11) shows the relationship for pure conduction and indicates that by maintaining a lower temperature differential a better definition of the thermal conductivity versus temperature curve will result.

2.2.2 Automatic Guarded Hot-Plate Apparatus

The apparatus used for the tests described in this report are completely automatic, the detailed design of which is considered proprietary. The automatic control console shown in Figure 7 operates four guarded hot-plate devices such as the ones described earlier and the one shown atop the console. The main feature of this console is that it controls automatically and sequentially four guarded hot-plate apparatus preprogrammed for 5 to 10 temperature levels. The operation proceeds unattended, completing, for a 12-hour test interval per apparatus, four tests, consisting of five data points each (20 data points total), in a 48-hour period. This feature allows the apparatus to function over a weekend, providing a 7-day a week, 24-hour operation.

Figure 8 shows the apparatus that was operated at high temperatures in an inert environment. This apparatus was required for testing charred materials. A test apparatus was also located in a Avco-designed container that permitted measurements in a vacuum environment. (See Figure 9.) The environmental adaptations work directly with the automatic control console, also shown in Figure 9. These test variations did not hinder the features of automatic control; stabilization times, however, had to be extended because of thermal lags inherent in vacuum operations.

Another feature of automatic control is the elimination of the human control factor and data randomness. Automatic control also allows more frequent apparatus tests to be made using NBS-certified standards; as a result, more precise data is achieved.

The large amount of data generated by the apparatus was controlled by incorporating a Benson-Lehner decimal converter, coupled to an L&N recorder by a retransmitting potentiometer attached to the recorder slidewire. The decimal converter digitized the data and, through proper scaling, presented the data in tabular form to both an electrotype and IBM 026 card punch unit. The data were then processed by a computer program.

The digitizing unit is interchangeable with the automatic specific heat apparatus, discussed later, and is not shown here. Where heavy but short work-load periods arose, a second digitizing unit, used in conjunction with optical measurements, was

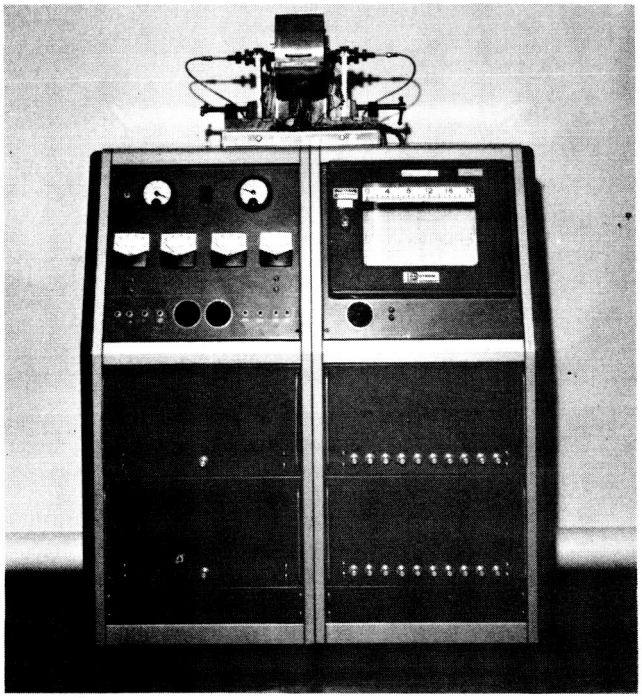


Figure 7 AUTOMATIC GUARDED HOT-PLATE CONTROL CONSOLE

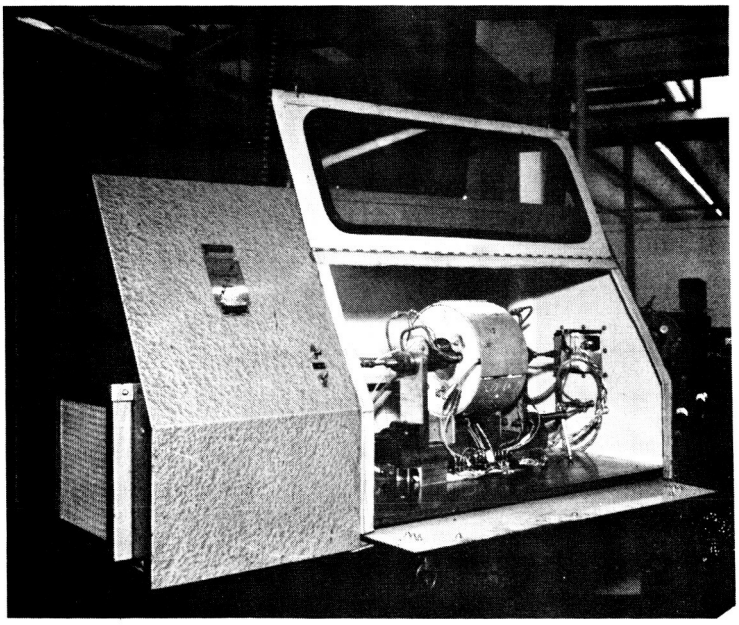


Figure 8 GUARDED HOT-PLATE APPARATUS FOR CONTROLLED ENVIRONMENT TESTING

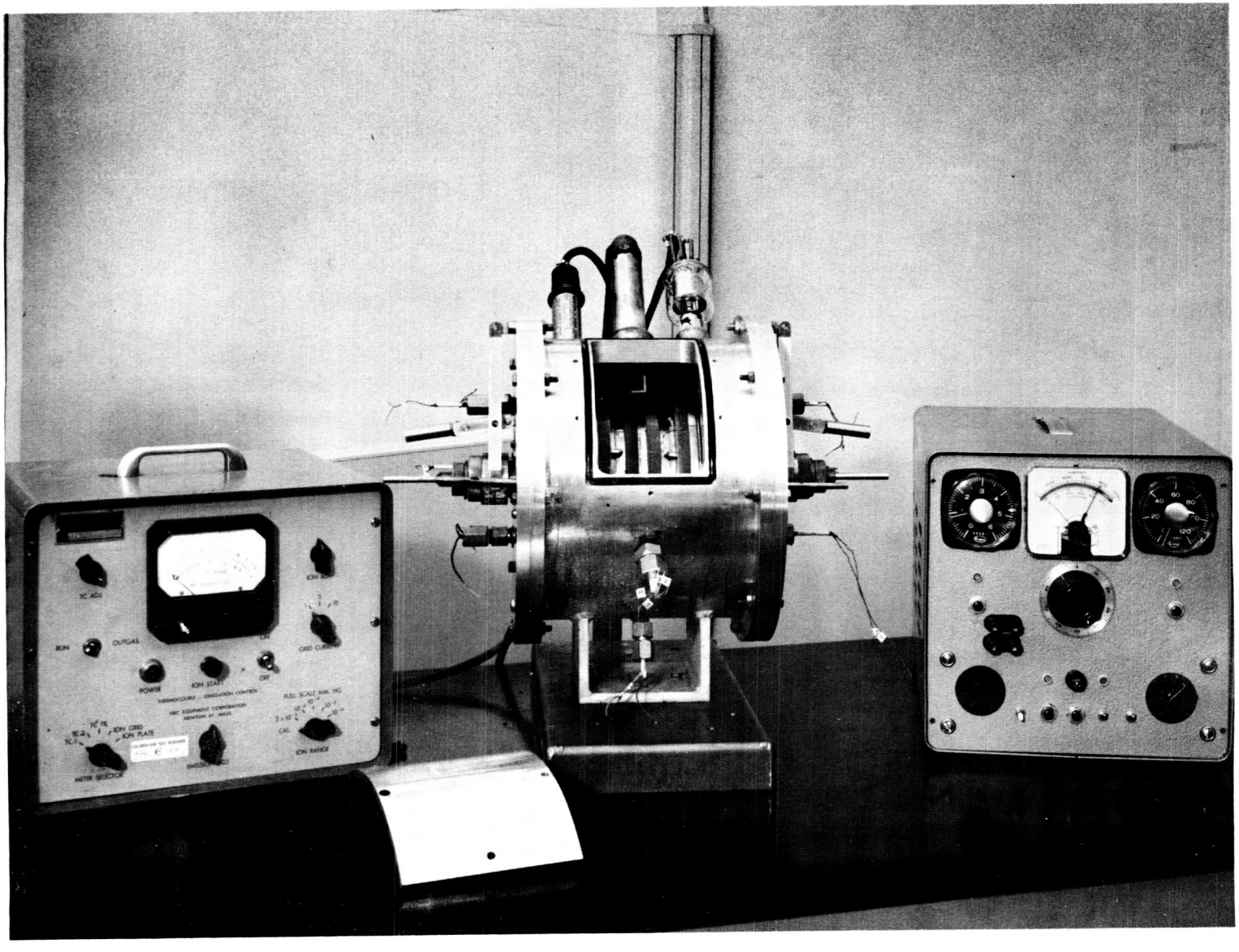


Figure 9 VACUUM THERMAL CONDUCTIVITY TEST APPARATUS

TABLE II

PARAMETRIC SPECIFICATIONS OF ASTM C-177-63

No.	Specification	ASTM
1	The recommended use mean-temperature range of the metal-surface guarded hot plate apparatus	-100 to 500°F
2	The recommended use mean-temperature range of the refractory guarded hot plate apparatus	200 to 1300°F
3	Upper thermal conductance limit	10 Btu/hr-ft^2-°F
4	The minimum temperature difference between the hot and cold plate for any test	10°F
5	The minimum temperature difference between the hot and cold plate for good insulators	40°F/in.
6	It is unlikely that the required maximum pressure on the specimen will exceed	50 lb/ft^2
7	All surface plates shall have a total emittance at operating temperature greater than	0.8
8	The maximum gap temperature imbalance	0.5%
9	Central heater supply regulated constancy of temperature difference between hot and cold plate	0.5%
10	Potentiometer-galvanometer sensitivity	1 μv or better
11	Heat losses in radial direction shall not exceed 1/5 the heat flow through the two specimens	
12	Maximum variation of conductivity made at four intervals of 30 minutes	1%

TABLE III

SPECIMEN SPECIFICATION OF ASTM C-177-63

No.	Specification
1	The specimen materials must be homogenous and opaque.
2	All specimens, homogeneous solids, blanket type, or loose filled materials shall be dried, weighed before and after test, and an "as-tested" density shall be calculated.
3	Two specimens as nearly identical as possible shall be used.
4	Rigid material faces shall be made flat to within 0.003-inch per foot (Avco specification 0.0005-inch for 0.465-inch diameter specimen).

TABLE IV

SYMBOL AND EQUATION SPECIFICATION ASTM C-177-63 FOR FLAT SLABS

No.	Specification
1	The thermal conductivity is calculated as follows: $$k = \frac{qL}{A(t_1 - t_2)} \quad (11)$$
2	The thermal conductance is calculated as follows: $$C = \frac{q}{A(t_1 - t_2)} = \frac{k}{L} \quad (12)$$
3	The thermal resistance is calculated as follows: $$R = \frac{1}{C} = \frac{L}{K} \quad (13)$$
4	Symbols k = thermal conductivity in Btu/in.-hr-ft^2-°F C = thermal conductance in Btu/hr-ft^2-°F R = thermal resistance in °F-hr-ft^2/Btu q = time rate of heat flow in Btu/hr A = area measured on a selected isothermal surface in square feet L = thickness of specimen measured normal to isothermal surface in inches t_1 = temperature of hot face in °F t_2 = temperature of cold face in °F

incorporated into the automatic conductivity apparatus, both experiments thus providing simultaneous output data in punch card and tabular form.

ASTM specifications for guarded hot-plate procedures formerly required a 5-hour stabilization period before measurements could be made. The new requirements are presented in Table II: conductivity test values should not vary by more than 1 percent for evaluations made at 4 intervals, 30 minutes apart. Such experiments as the one shown in Figure 10 were performed: the apparent thermal conductivity was measured 0.20 Btu/hr-ft-°F. The figure shows that a stable condition was reached in 30 minutes on the electronically controlled apparatus. Figure 11 illustrates the stabilization of a thermal conductivity measurement of polystyrene. This material exhibited stability within 1 1/2 hours. As a result of these tests and 25 additional verifications, it was determined that a stabilization period of 2 hours, which is equivalent to the new specification, was adequate for materials whose thermal conductivity was below the limit specified for this procedure.

Previous reference was made to a 12-hour-total test period. This period was based on five preprogrammed test temperature levels of 2 hours each; five 15-minute data-collecting periods; and a combined 45-minute loading and apparatus cooling period. The 12-hour test periods allowed testing two sets of specimens per day and four sets per 2-day weekend.

2.2.3 Semi-Automatic Guarded Hot-Plate Apparatus for Low Temperatures

The guarded hot-plate technique was extended for low-temperature measurements by a minor modification of the test apparatus described above. The change included a replacement of the auxiliary heater-cooler arrangement with a constant-level, low-temperature liquid reservoir. The apparatus used for low-temperature measurements is shown both schematically (Figure 12) and pictorially (Figure 13). The change modifies a refractory guarded hot plate configuration to a metal-face guarded hot plate having end-plate cooler assemblies. For low-temperature tests, the specimen faces were instrumented. Thin insulating mats were interposed between the heater plates and the specimens to electrically insulate the thermocouples and to assist in obtaining the desired temperature differential across the specimen. Combined with a variety of cooling fluids contained in the end-plate reservoirs, the electrical power to the main heater provided incremental thermal conductivity measurements from -250°F to room temperature.

2.2.4 Radial Thermal Conductivity Apparatus

It was found that measurements of thermal conductivity for SI 62 multilayer in-insulation were unsatisfactory when tested in the guarded hot plate vacuum apparatus. Several factors contributed to this condition: the level of conductivity was so low that the power required in the main heater approached the minimum detectable for the system; the relative edge losses for the sample configuration were excessive; handling of the specimens was too awkward.

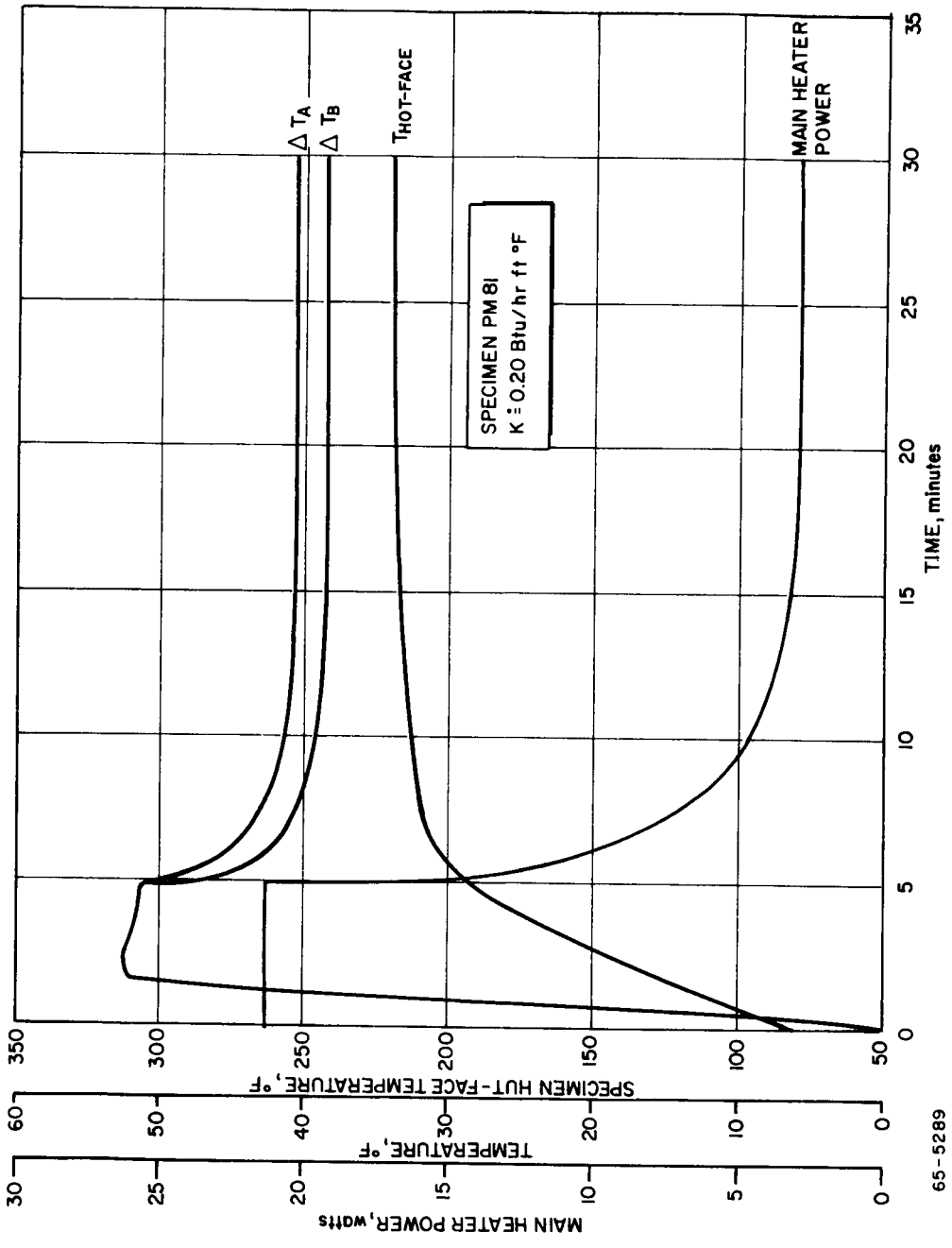


Figure 10 AUTOMATIC THERMAL CONDUCTIVITY APPARATUS RESPONSE CURVE

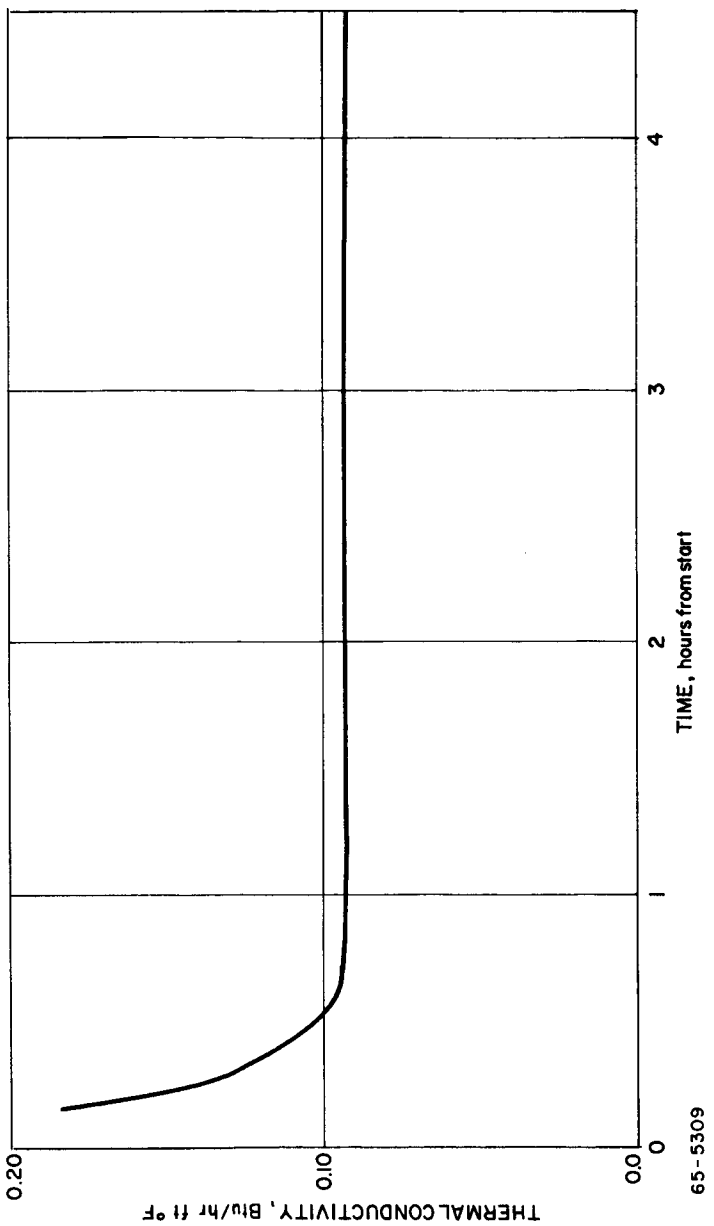


Figure 11 AUTOMATIC THERMAL CONDUCTIVITY APPARATUS, CONDUCTIVITY VERSUS TIME FOR POLYSTYRENE

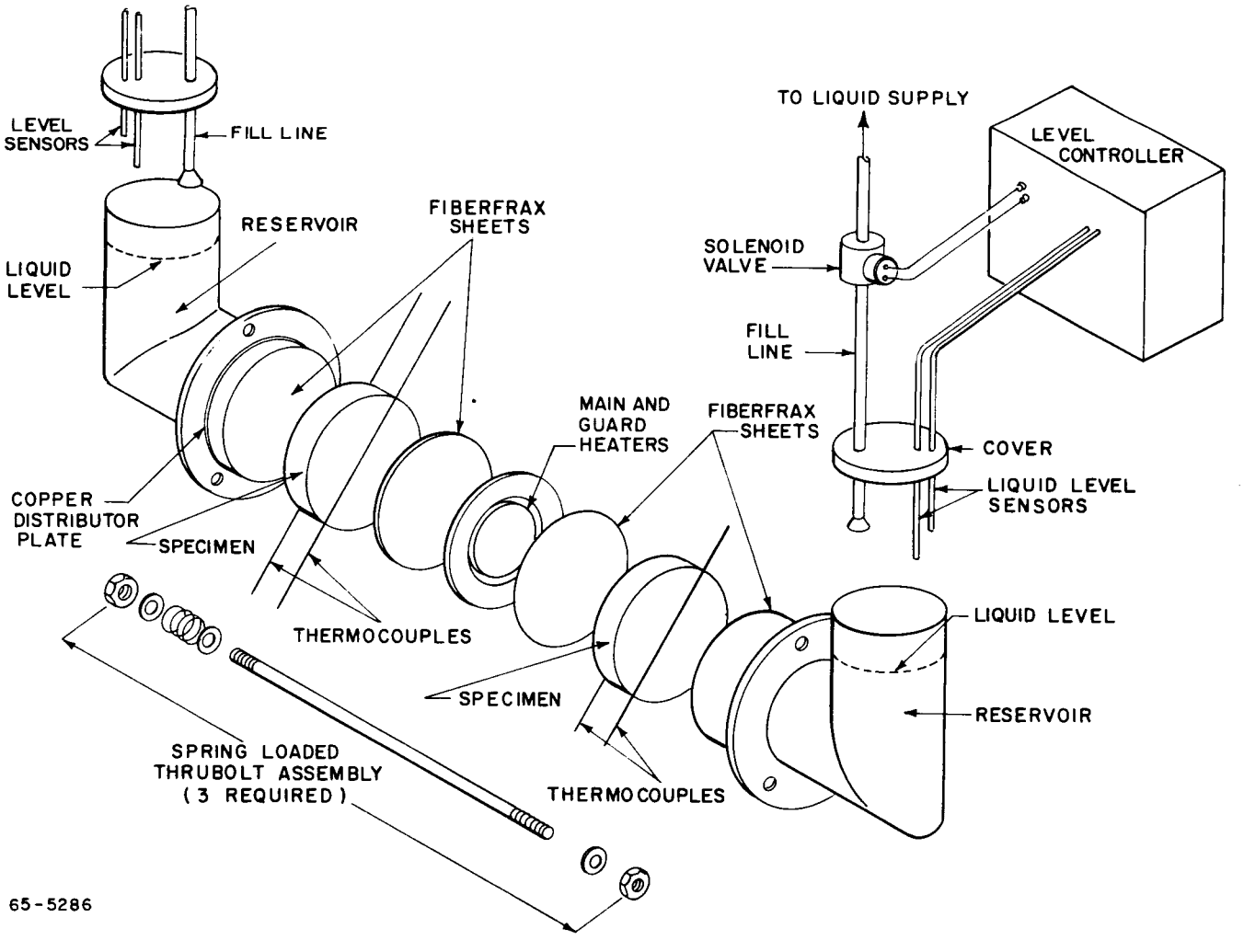


Figure 12 SCHEMATIC OF ARRANGEMENT OF AVCO SEMI-AUTOMATIC, LOW TEMPERATURE, GUARDED HOT-PLATE APPARATUS

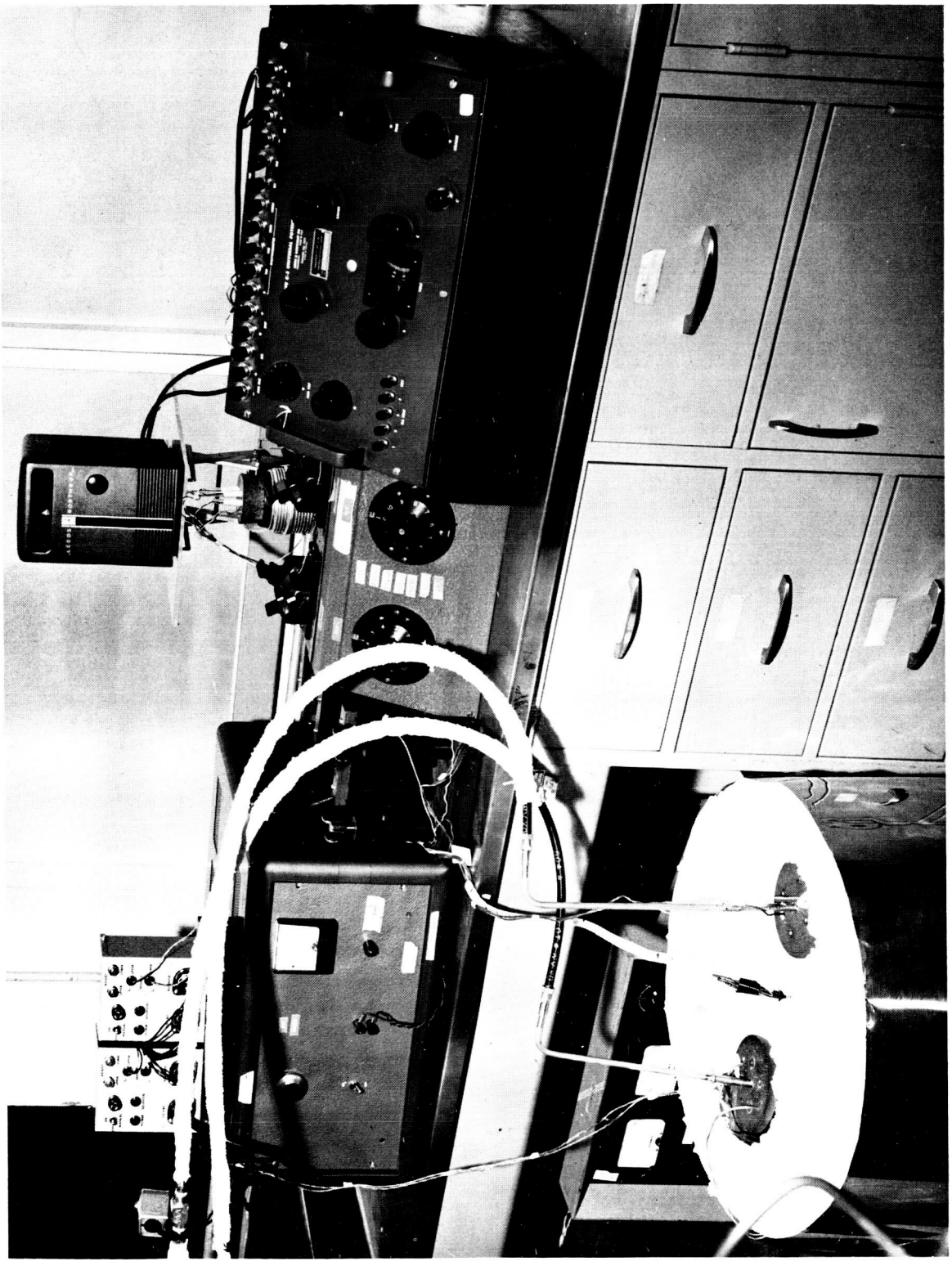


Figure 13 LOW TEMPERATURE THERMAL CONDUCTIVITY APPARATUS

A radial conductivity apparatus was found to be more suitable. It offered higher power levels, less edge loss effects, and simpler sample preparation. A schematic diagram of the basic apparatus is shown in Figure 14. The heater core was constructed into three separate heaters: the center, or main, heater, whose power level was obtained from the potential taps and used in the conductivity determination; the other two, located on each side of the main heater, served as guard heaters to control the temperature profile along the inner surface of the test sample and to minimize axial heat losses. Nine thermocouples, arranged in groups of three, were installed along the heater tube. Each group was equally spaced around the heater, positioned in the same axial location and in the center of each of the three test zones. These were used in conjunction with nine thermocouples installed in the same relative position along the "cold" face of the test specimen. Differential electrical connections were made between several hot-face thermocouples in the main and guard section. These differential thermocouples were connected to automatic controllers that provided continuous control of the guard heater power levels, thereby maintaining a flat and uniform temperature profile along the test zone.

The cooling region was constructed of a sleeve of copper with cooling tubes brazed to its outer surface.

Temperature levels and temperature gradients were obtained through combinations of the heater power, cooling fluid selection, and flow rate. Fluids included liquid nitrogen, super cooled alcohol, nitrogen, dry air, and water.

The assembly was located in a stainless steel vacuum chamber in which tests were conducted in air, inert atmosphere, and vacuum environments.

Figure 15 illustrates the test apparatus and a typical test specimen after the completion of a test. Calculations of thermal conductivity were based on the standard one dimensional heat conduction equation for radial flow, rearranged for the conductivity parameter:

$$K = \frac{Q \ln\left(\frac{r_2}{r_1}\right)}{2\pi l (t_2 - t_1)} \quad , \tag{14}$$

where

Q = total heat flow through the test zone (Btu/hr)

r_2 = outer specimen radius (ft)

r_1 = inner specimen radius (ft)

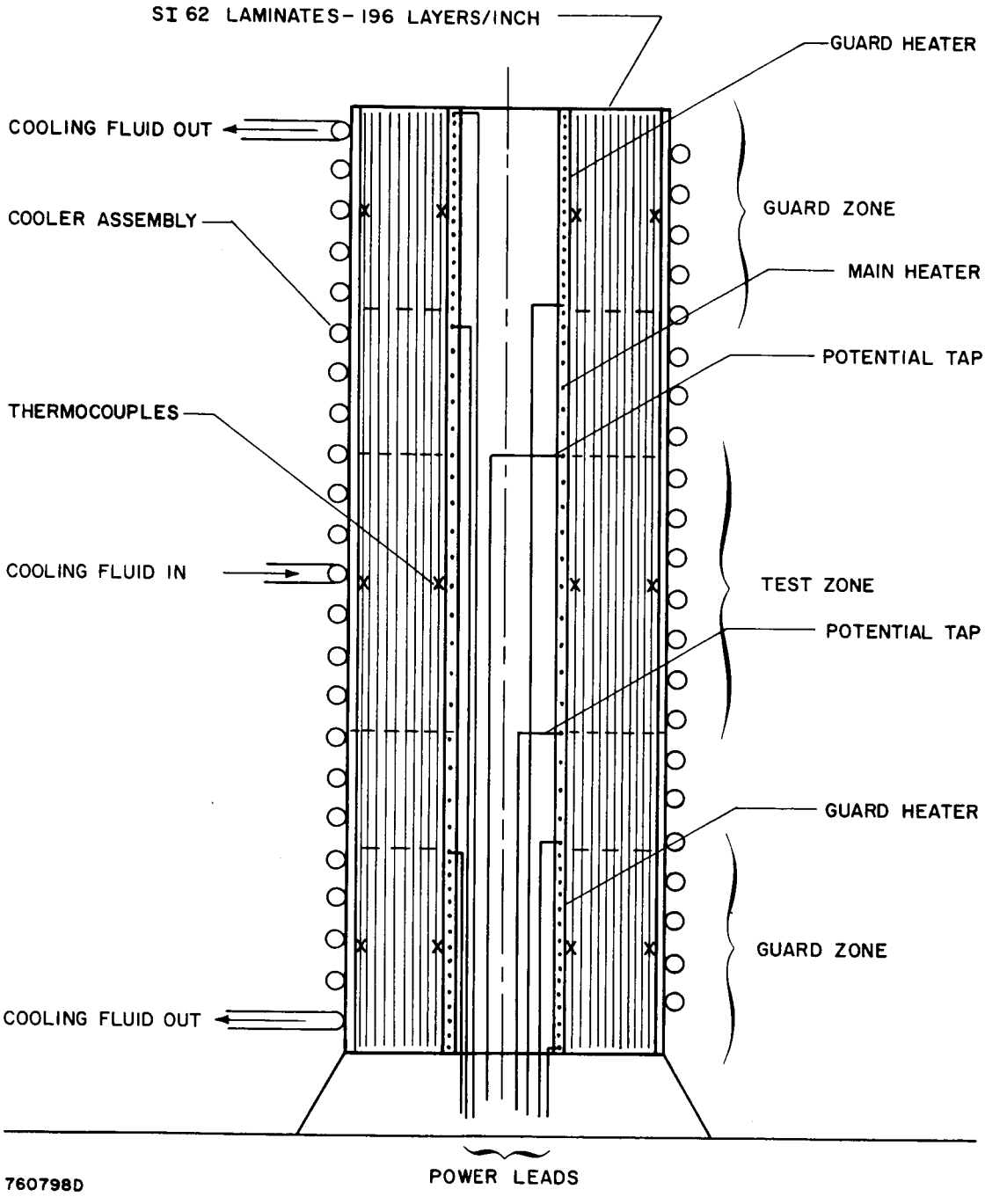


Figure 14 RADIAL CONDUCTIVITY SCHEMATIC

Figure 15 RADIAL THERMAL CONDUCTIVITY APPARATUS AND SPECIMEN

L = length of the test zone (ft)

$(t_2 - t_1)$ = temperature difference between specimen hot and cold faces (° F).

2.2.5 Mean Specific Heat of Thermal Insulations (ASTM C351-61)

The specification that deals with specific heat measurements is commonly referred to as the method of mixtures. The procedure consists of adding a known mass of test material at one temperature to a known mass of water (or other medium) at a different temperature and determining the resulting equilibrium temperature. The heat absorbed or liberated both by the water (or other medium) and the containing vessel can be calculated, and this value equated to the expression for the heat transferred by the test material. From this equation, the unknown specific heat can be calculated:

$$C_s = \frac{\dfrac{(M_w + E)\, C_w\, (T_m - T_c)}{(T_h - T_M)}}{M_s} - M_c\, C_c \quad , \qquad (15)$$

where

E = water equivalent of the calorimeter and its accessories

M_w = mass of calorimeter water

C_w = mean specific heat of calorimeter water

M_s = mass of the specimen

$M_c\, C_c$ = thermal capacity of the capsule

C_s = mean specific heat of the test specimen over the temperature range T_h to T_m

T_h = temperature of the capsule and specimen, capsule, or standard after heating

T_m = temperature of the mixture extrapolated back to time 10 minutes

T_c = temperature extrapolated ahead of time 10 minutes, of the calorimeter water before capsule or standard is dropped.

A comparison of ASTM and Avco specifications for specific heat is presented in Tables V, VI, and VII. There was a notable difference between these specifications; Avco, therefore, could not stipulate adherence to ASTM Procedures. Although the basic procedures were similar, Avco made the apparatus more versatile, eliminated the problems associated with water corrections, and avoided dropping the specimen directly into water.

2.2.6 Automatic Specific Heat Apparatus

With the exception of the sample loading requirement, the test facility used during this program was completely automatic. The facility consisted of five complete assemblies, automatic control, electrotyper, and a decimal converter, all shown in Figure 16. The entire calorimeters assembly was enclosed in a plexiglass chamber, which prevented minor ambient variations from influencing a test. A schematic illustration of a single assembly is shown in Figure 17. Each assembly was individually designed to provide a calorimeter block mass, matched to a corresponding furnace temperature. The matching process provided a uniform calorimeter temperature change over a large heater temperature range and permitted more accurate automatic recording of the test temperature changes.

The overall schematic arrangement of the specific-heat apparatus used is shown in Figure 18. The programmer shown schematically in Figure 19 is the principal component in the automatic process. The unit contains two multiple cam timers and two multiple-throw–multiple-pole rotary switches. The long period timer selects the time interval over which a particular series of events occur and positions the master selection switch for information presentation (e. g. , furnace emf, calorimeter emf, or reference-block emf). The short period timer positions a second rotary switch for sequential reading of the five furnace-calorimeter sets. Included in the programmer was a signal cam that delays digital readout until the analog resistance converter is balanced. The analog resistance converter consists of a high-gain amplifier operating at null balance, a direct-current reference supply, and a servo motor that drives two ganged precision potentiometers. The device provides a high impedance signal for the decimal converter. The data obtained from this system was both in the form of typed reference copy and of IBM punch cards. The volume of data that was obtained using this apparatus was the basis for incorporating a small digital program (1663), written to provide an emf-to-temperature conversion, data calculation, and plots of enthalpy as a function of temperature.

The conversion of an enthalpy curve to specific heat must be performed with care, unless, of course, it is a straight-line curve. In general, the simplest curve or series of curves should be used to describe (fit) the enthalpy data. Over-fitting

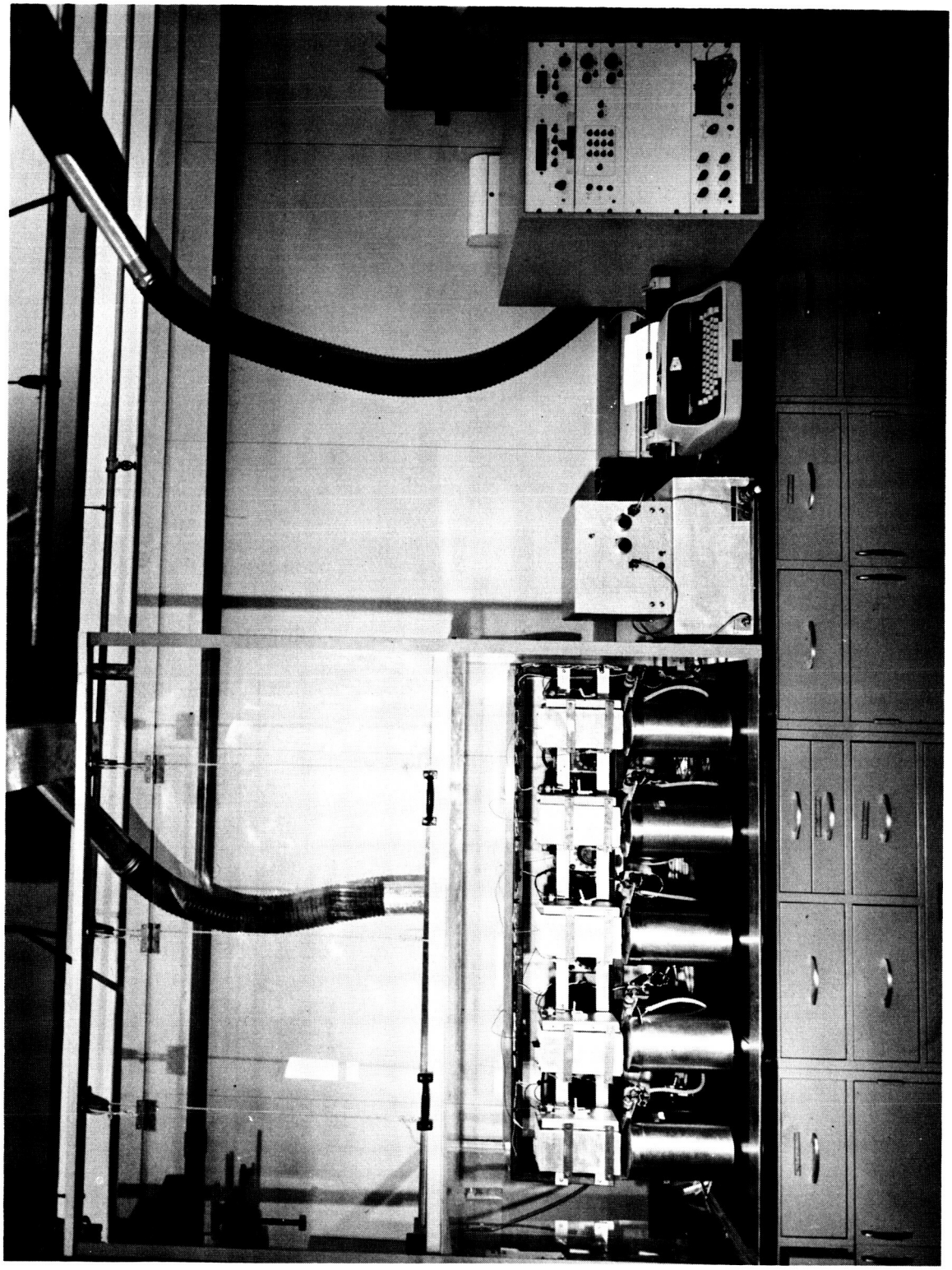


Figure 16 AUTOMATIC SPECIFIC HEAT APPARATUS

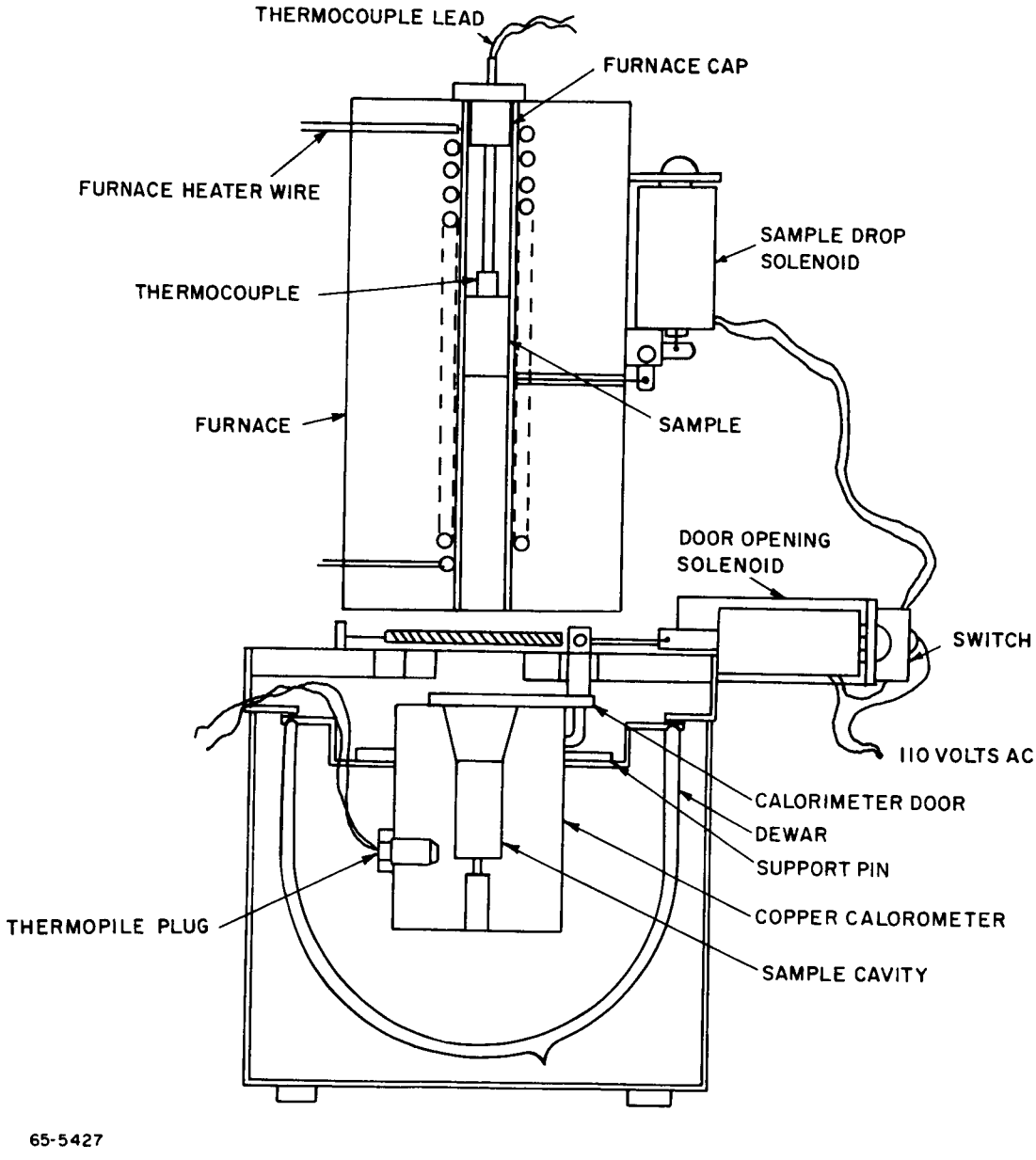
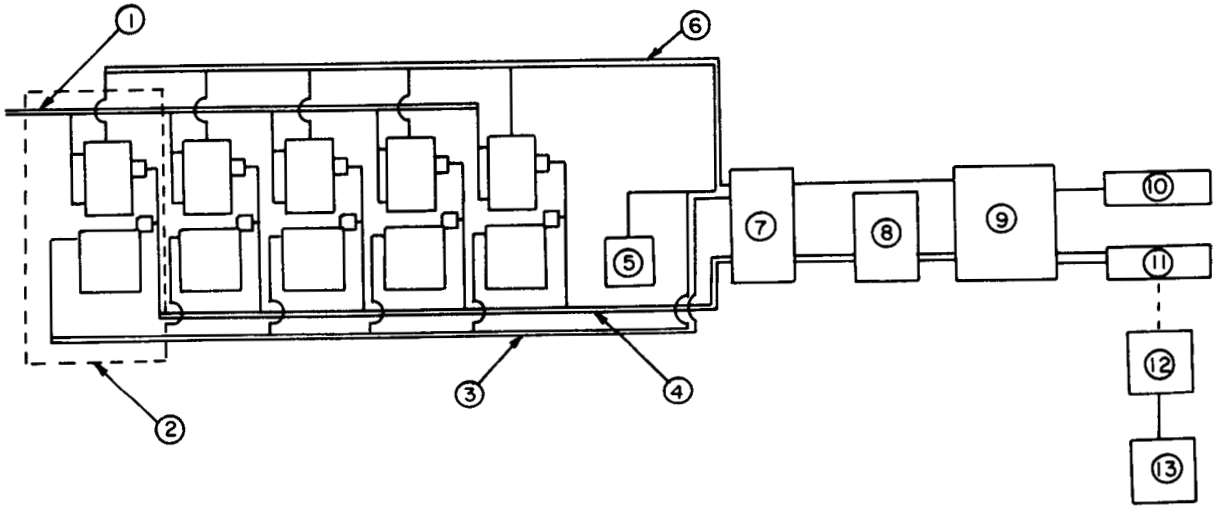


Figure 17 SCHEMATIC OF AUTOMATIC FURNACE-CALORIMETER SPECIFIC HEAT SET



1. FURNACE POWER
2. FURNACE CALORIMETER SET
3. CALORIMETER THERMOPILE LEADS
4. DOOR OPEN & SAMPLE DROP LEADS
5. REFERENCE POWER
6. FURNACE THERMOCOUPLE LEADS
7. PROGRAMMER
8. EMF TO ANALOG RESISTANCE CONVERTER
9. ANALOG TO DIGITAL CONVERTER
10. TYPER
11. CARD PUNCH
12. DIGITAL COMPUTER
13. PLOTTER

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Figure 18 SCHEMATIC ARRANGEMENT OF AUTOMATIC SPECIFIC HEAT COMPONENTS

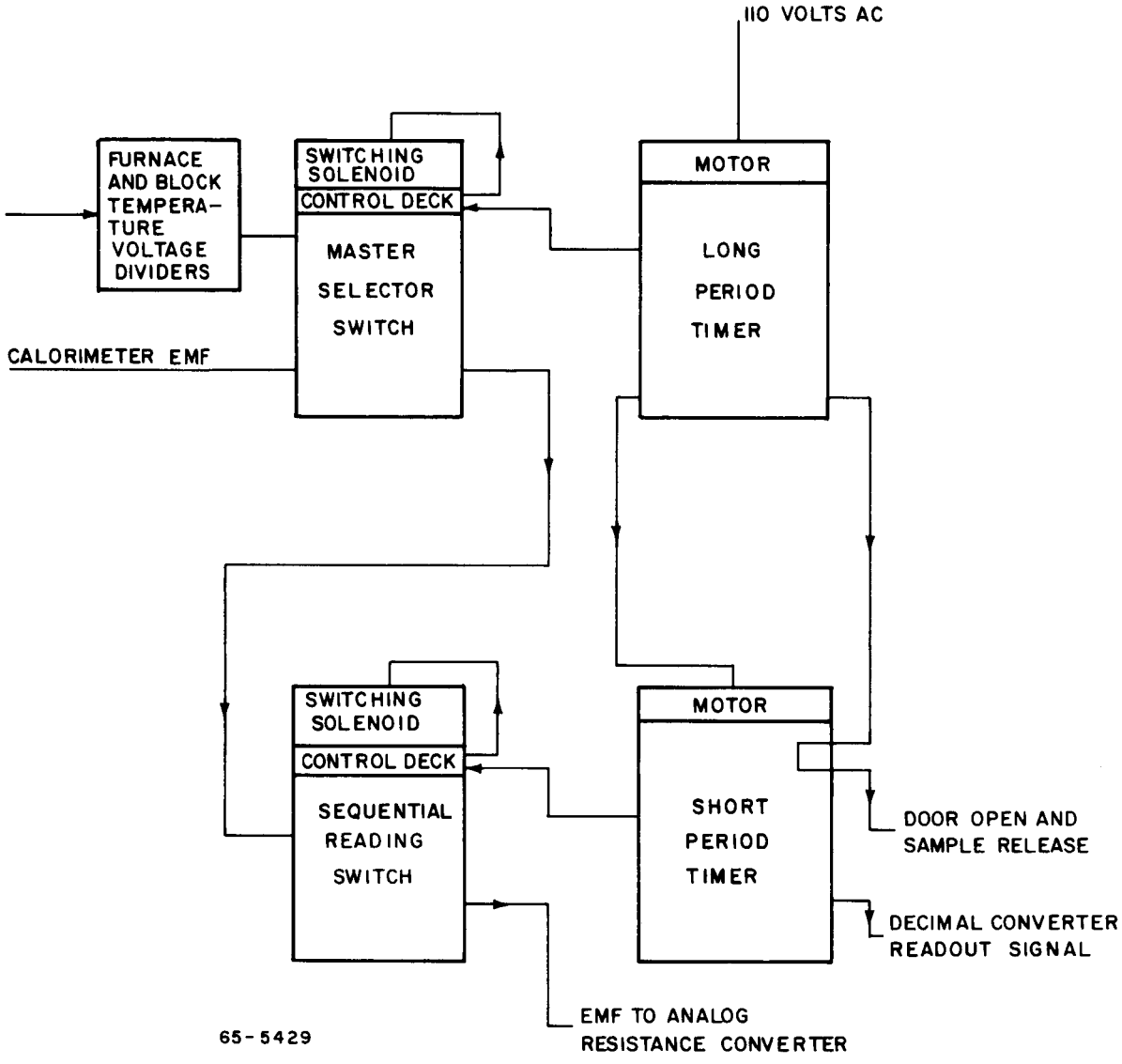


Figure 19 SCHEMATIC ARRANGEMENT OF AUTOMATIC SPECIFIC HEAT PROGRAMER

TABLE V

MECHANICAL SPECIFICATIONS OF ASTM C351-61

No.	Specification	Astm	Avco
1.	Range of capacity of unlagged Dewar flask (ml)	500 to 750	750
2.	Calorimetric medium	Water	OHFC Copper
3.	Capsule assembly diameter (in.)	1.0	0.37 I.D.
4.	Calibration standard dimension (in.)	2 x 1/4 x 1 in.	0.32 dia x 1 length
5.	Heater core dimensions (in.)	$1\frac{1}{2}$ dia x 10-in. length	1/2 dia x 6 length
6.	Insulation thickness-heater unit (in.)	1 dia	4 x 4
7.	Capsule assembly length (in.)	2	1
8.	Thermocouple wire diameter (in.)	B & S 30 (0.010)	B & S 36 (0.005)

TABLE VI

PARAMETRIC SPECIFICATIONS OF ASTM C351-61

No.	Specification	ASTM	Avco
1	Mean temperature range	68 to 212°F	-250 to 2000°F
2	Thermometry temperature range	68 to 212°F	-250 to 2000°F
3	Readable thermometry increments	0.02°F	0.02°F
4	Maximum variation over heater length	±2°F	±0.5°F to ±3/4%
5	Voltage source stability	±1%	●1%
6	Maximum capsule capacity	6×10^{-3} Btu/°F	Variable or none
7	Measurement apparatus precision	0.2°F	0.2°F
8	Room temperature constant for 20-minute period		Conform
9	Specific heat standard value	0.093 Btu/lb°F	Variable based on NBS Data

TABLE VII

SPECIMEN SPECIFICATIONS OF ASTM C351-61

No.	Specification	ASTM	Avco
1	Homogeneous material in solid state	OK	OK
2	Calibration standard material	Electrolytic copper	Synthetic sapphire
3	Capsule assembly material	Brass	Aluminum foil
4	Test Sample dimensions (in.)	2 x 1/4 x 1	0.32 dia x 1 or longer length
5	Number of test specimens per determination	3	12

could result in erroneous inflections or "tailing off" at the data extremes. The simple fit minimizes errors in converting enthalpy to specific heat. It was found that is most cases enthalpy data are used directly; for this report, however, the specific heat data were obtained using a derivimeter.

Instead of determining the water equivalent of copper, as described in the ASTM procedure summarized above, the apparatus used for this study was periodically calibrated using synthetic sapphire and data recommended by the NBS.[3] Several calibration series provided sufficient information to obtain an estimate of the system precision. This method obviates the need for an error analysis of the various system components.

The automation of this apparatus, as in the automating of thermal conductivity experiments, eliminated the human operator variable from the results.

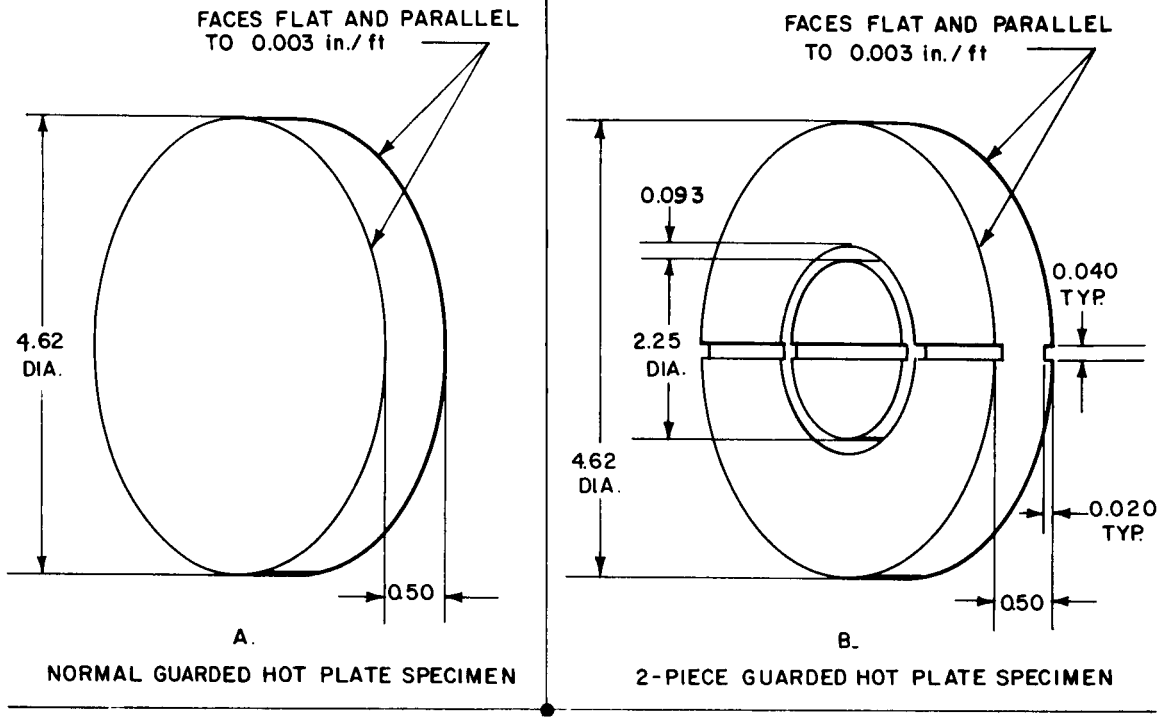
2.2.7 Sample Sizes

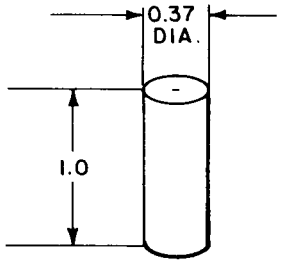
Figure 20 illustrates the various types of solid specimens that were used for testing. The first specimen is typical of a virgin or charred sample used for guarded hot-plate tests. A charred specimen was machined to the dimensions illustrated from an oversized virgin specimen following the charring process. The two-piece guarded hot-plate specimen was used for materials having higher than usual thermal conductivity (i.e., metal honeycombs). The air gap in the specimen assisted in ensuring unidirectional heat flow, and the groove provided for thermocouple installation and avoided the small interface gap that can cause serious errors in measurements. This type of specimen has been used with guarded hot-plate techniques to measure apparent thermal conductivities as high as 5 Btu/hr-ft-°F, and cross checks have been made using cut-bar techniques. The agreement between methods was excellent; thus Avco had sufficient evidence to indicate that the upper limit of apparent thermal conductivity specified in ASTM C-177-63 could be raised to 5 Btu/hr-ft-°F if the specimen configuration shown is utilized. The remaining specimen drawing was used for specific heat sample preparation.

2.2.8 Charred Materials Formation

Material charring to 2000°F was performed automatically in a controlled atmosphere nickel enclosure that ensured a uniform temperature distribution. In preparation, the degradation container was filled with the test material, sealed except for the gas inlet and exit, and placed in a furnace. A regulated amount of inert or other gas was allowed to pass through the container to produce the desired atmosphere. The degradation cycle was programmed, and the furnace and container were allowed to come to preset elevated temperature equilibriums.

Resinous materials that generally have a volatile phase were held at the resin decomposition temperature until the reaction was completed and then were exposed to a higher temperature. The delay at the decomposition temperature helped to





C.

SPECIFIC HEAT SPECIMENS

65-5284 A

Figure 20 SPECIFICATIONS OF THERMAL PROPERTIES TEST SPECIMENS

reduce the possibility of the material splitting or cracking that accompanies severe gradients existing within the specimens.

A minimum of 2 to 3 hours of soaking time was found adequate to completely char a diathermous material. Equally important was the control of the cooling rate of the degradation furnace. Failure to program the cooling process produced specimens that were warped, split, or otherwise not usable.

A typical charring program is illustrated in Figure 21. A 2000 and 5000°F char sequence is shown; also indicated are the several temperature plateaus programmed for various phenolic diathermous materials.

2.3 TEST APPARATUS: SECONDARY PARAMETERS

The various test apparatus discussed in this section were used to investigate phenomena that directly influenced variations in the primary parameters. The data obtained from a support apparatus were used as evidence of validity of a test in a temperature range or as additional information related to a parametric response under different conditions.

2.3.1 Transient Response Evaluations

Transient response tests were performed to supplement the primary parameter evaluations of aluminum honeycomb panels only.

Tests of the type performed in this category could have taken several forms, depending upon the input heating requirements. The NASA MSC input requirement for these tests is shown in Figure 22. The heating rate is considered very low, and tests could be performed using many standard laboratory-type apparatus. The input requirement was defined as a surface temperature history only.

2.3.1.1 Thermal Response Tests: Aluminum Honeycomb Panels

Several transient thermal response measurements were performed, as requested by NASA MSC, on various aluminum honeycomb panel samples. The regular two-piece thermal conductivity specimens were shown to be suitable for use throughout the program. The specimen size was compatable with several test apparatus, and the availability of components necessary for modification of existing equipment added to their usefulness. By utilizing the two-piece specimen, one-dimensional heat flow in the samples was reasonably ensured.

The tests as conducted were classed into three major categories: (a) radiant source-free convection, (b) back-to-back, and (c) hot plate methods.

2.3.1.2 Radiant Source: Free Convection Method

Tests by this method were performed with a twofold purpose: (a) to characterize the instrumentation specification applying to temperature monitoring of the

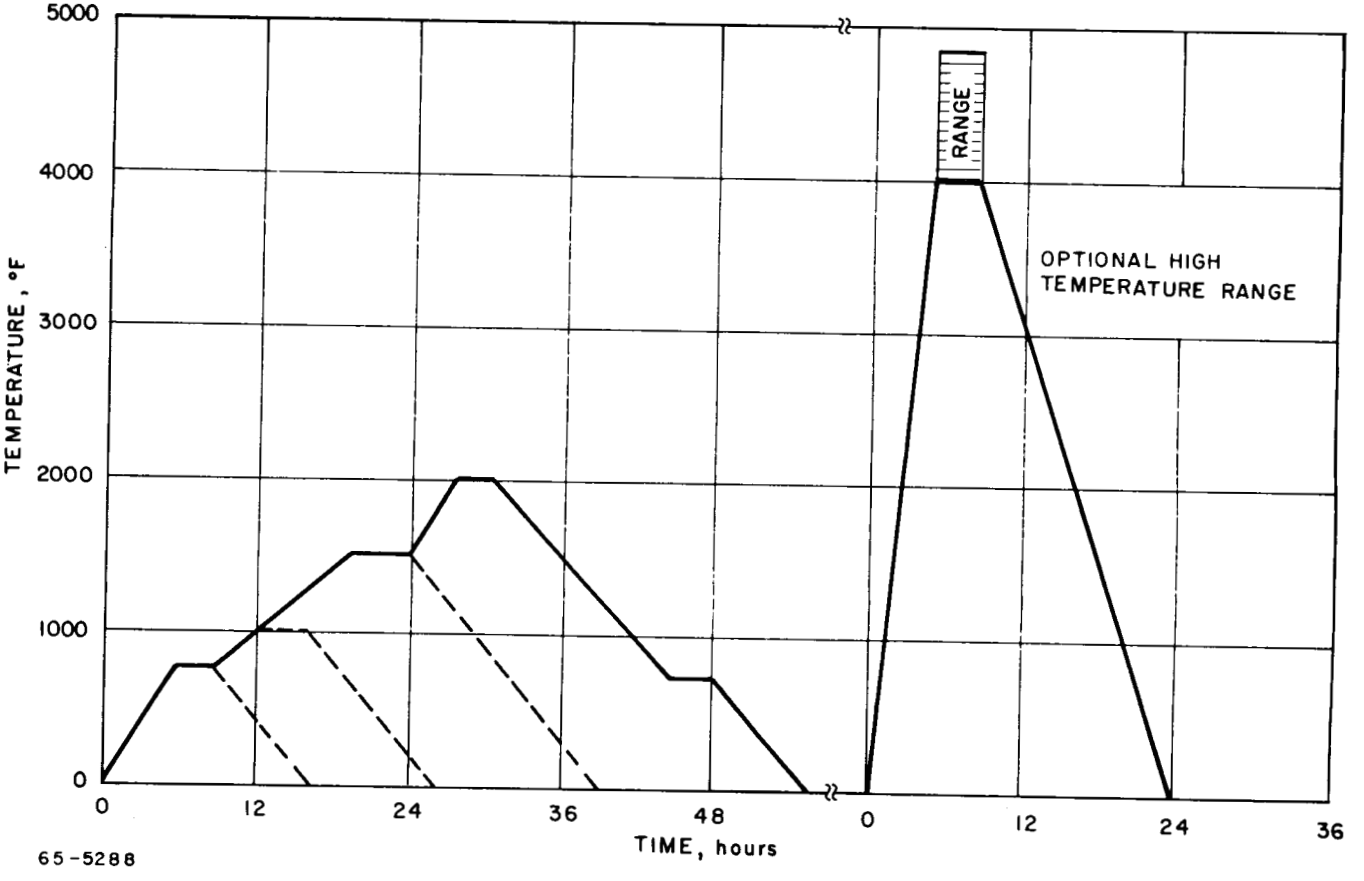


Figure 21 TYPICAL CHARRING SCHEDULE FOR PHENOLIC RESIN SYSTEMS

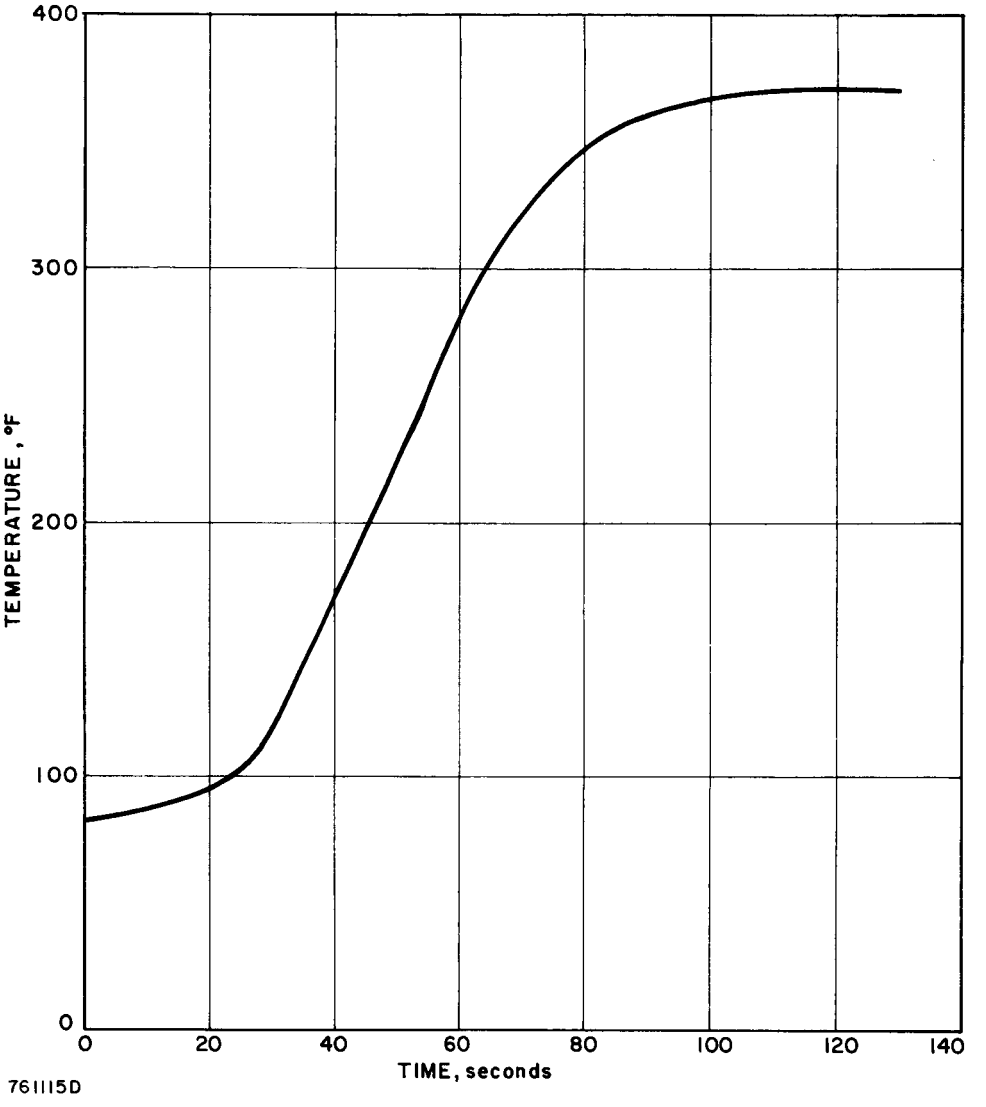


Figure 22 TIME-TEMPERATURE REQUIREMENT FOR THERMAL RESPONSE TESTS OF ALUMINUM HONEYCOMB PANELS

aluminum backup structure and (b) to evaluate the thermal response of the aluminum honeycomb paneling when it is subjected to the input temperature history illustrated in Figure 22.

The Avco 20-kw quartz-lamp radiant facility (Figure 23) was adapted for these tests, and provisions were made to control the input temperature history through the use of variable power controls and specimen-to-lamp distance adjustments. Insulation material was installed around the specimen edges to minimize side heating of the panel and yet allow free convection at the specimen back face with the panel in the horizontal position (Figure 23-1).

One instrumentation variation included spot welding a thermocouple to a 0.010-inch thick x 3/16 triangular stainless steel tab and, using Epon 934 bonding agent, fastening the tab assembly to the surface of the aluminum panel. Thickness of the bond layer was specified as 0.010 inch. The first part of this evaluation was a comparison of the temperature response of the tab instrumentation with that of a thermocouple in contact with the surface of the panel when the panel is subjected to the specified temperature history. Tab assemblies were bonded to each face of the panel, and two thermocouples were fixed directly to the panel surfaces. A six-channel rapid response recorder (Offner Dynagraph Type RM) was used to monitor the thermocouple outputs during the test.

The second part of the study was an evaluation of the thermal response of the panel, with the addition of a calorimeter at the backface of the specimen to measure the heat flow out of the material. The calorimeter was an instrumented thin copper disk of equal diameter as the specimen. The calorimeter was guarded by a thin copper ring whose dimensions were also equal to the specimen guard-ring dimensions. The time-temperature traces of the calorimeters were included in the six-channel record during test, and this information was converted to heat flow rate during the transient tests.

2.3.1.3 Back-to-Back Method

This technique required "sandwiching" the previously mentioned guarded disk calorimeter between two panel specimens (TS 513 configuration and instrumented directly on each surface). These were then placed between two identical heater plates (Figure 24) whose power levels were controlled to ensure symmetrical heat flow through the specimens. The back-to-back test configuration has the advantage of an adiabatic back face. The six-channel Offner record was used to monitor the four sample face temperatures, and the calorimeter output.

2.3.1.4 Hot-Plate Method

This method was operationally simple in that it utilized a preheated commercial hot plate, and the specimen under test was placed on the heater. The resulting temperature rises were recorded. This test configuration included the guarded disk calorimeter, which was placed on the specimen back face. The principal feature in this experiment was the very rapid and uncontrolled initial hot face

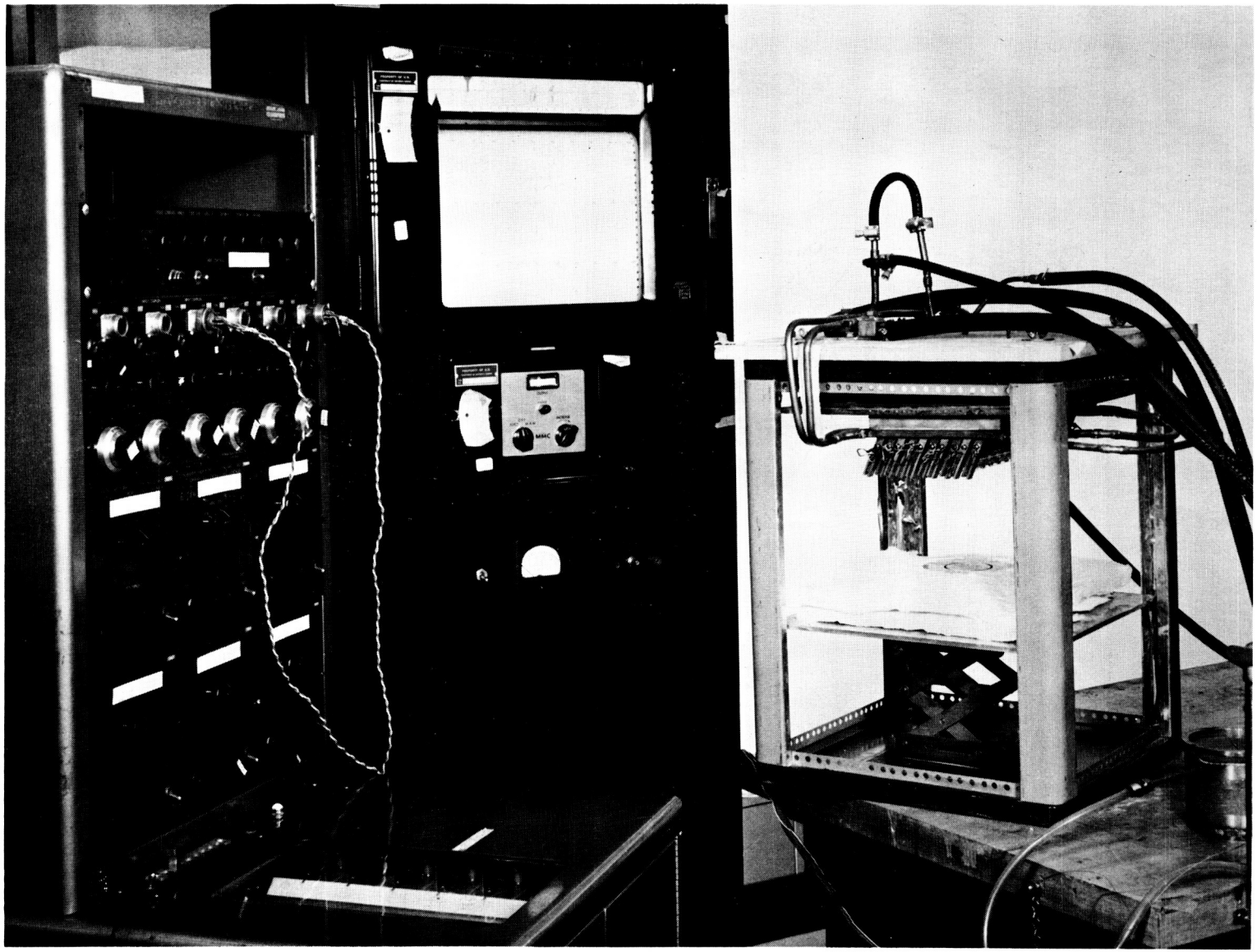


Figure 23 RADIANT HEAT FACILITY

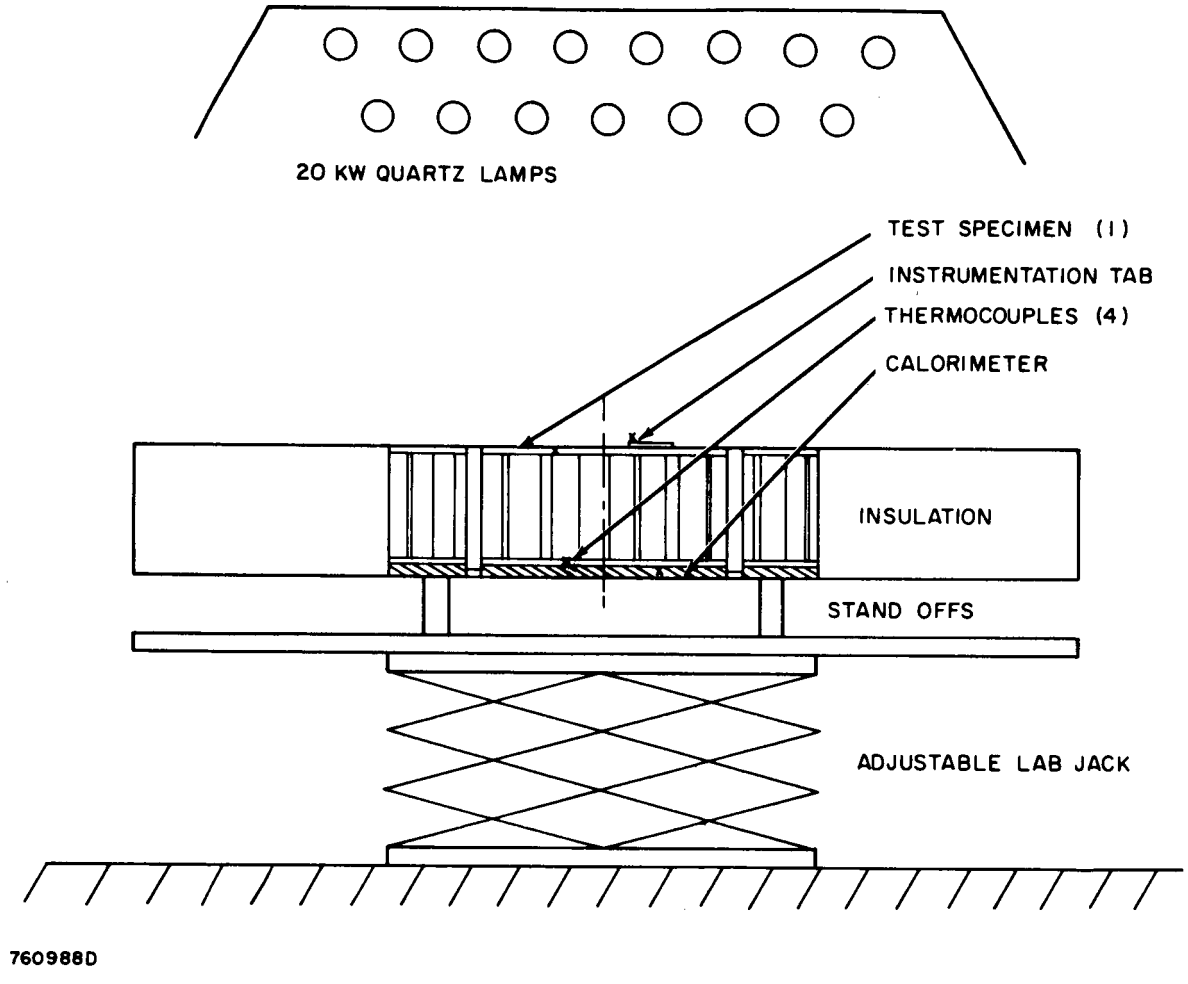


Figure 23-1 RADIANT SOURCE: FREE CONVECTION TRANSIENT SCHEMATIC

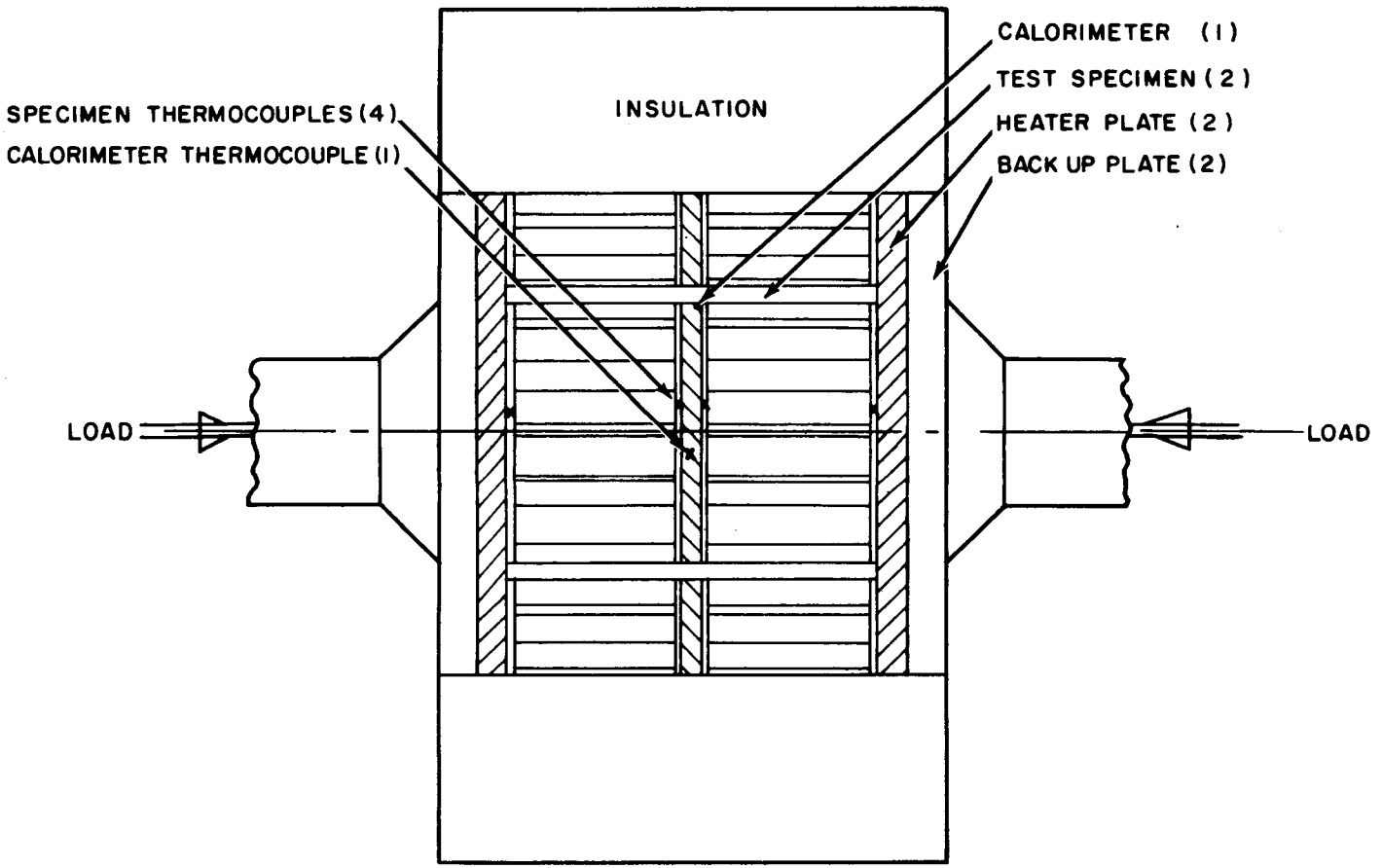


Figure 24 BACK-TO-BACK TRANSIENT RESPONSE TEST SCHEMATIC

temperature rise. The free convection cooling at the sample or calorimeter back face requires a correction of the calorimeter data, particularly in the latter periods of the measurements. During the test, a cyclic temperature history was observed at the sample hot face. This phenomenon was attributed to the thermostatic control on the source. The history was retained with the rest of the data, for possible use in analyzing the effects of this type heat input.

Thermocouple instrumentation for the test was similar to the back-to-back arrangement; the thermocouples were placed in direct contact with the face plates of the honeycomb structure.

The time-temperature data obtained by these schemes and the comparison of two thermocouple attachment methods were the extent of the effort in this area. When it was possible to measure the heat flux out of the sample, the measurements were included into the test and reported.

2.3.2 Differential Scan Calorimetry

The differential scanning calorimeter, referred to in this report as DSC, is a relatively new type apparatus based on the same scheme as differential thermal analysis (DTA). The basic operational concepts are the same, the exception being that the DSC measures the required energy to heat an unknown material; DTA measures only a temperature difference. As an energy measuring device, the DSC offers the advantage of quantitative analysis. Both instruments are operated at several heating rates. The DSC scheme measures power while it maintains an equal temperature between a reference and an unknown material. DTA measures temperature excursions while it maintains equal power to both the reference and unknown materials.

The apparatus used during this contract was a Perkin-Elmer DSC-1 (Figure 25), modified to overcome certain operational limitations and to allow IBM punch card data acquisition. Semi-automation significantly reduced the time required for data reduction.

The recorded results of a test with the DSC-1 superficially resemble those obtained from a DTA, i.e., the abscissa represents temperature. The operator obtains the key temperature from the digital dial display on the instrument control panel and writes the temperature on the chart as the base reference figure from which all other temperatures are derived. A second pen draws a data line that represents enthalpy when no transitions are in progress. "Peaks" in the traces indicate transitions in the sample material. The information derived from the tracing is as follows:

a) The specific heat of a material.

b) The temperature at which a transition occurs, indicated by the onset of a deflection from the baseline.

Figure 25 DIFFERENTIAL SCAN CALORIMETER

c) The endothermic or exothermic nature of the transition, indicated by the excursion direction of the pen from the baseline. In these respects, the DSC interpretation is identical to that of traditional DTA.

d) The area under a peak is directly proportional to the energy absorbed or liberated by the material in a transition and is unaffected by sample geometry, sample heat capacity, or such instrument operating parameters as scanning rate. The apparent area will change for example, if, recording chart speed is changed or if the calorimeter output signal is electrically attenuated prior to recording.

The instrument temperature scale (x axis) is calibrated using NBS certified metals, and the enthalpy (y axis) is calibrated using synthetic sapphire. The specific heat is obtained directly from the relationship

$$Cp_s = Cp_r \times \frac{d_s}{d_r} \times \frac{m_r}{m_s} , \qquad (16)$$

where

Cp = specific heat, Btu/lb°F

d = recorder deflection, in.

m = weight of material, lb,

and subscripts

s = unknown sample

r = reference sample

2.3.3 Pressure Drop

During the course of the program, the NASA MSC advanced heat shield programs required the experimental determination of viscous and inertial resistance flow coefficients. The coefficients were needed for both virgin and various prechar-red states of ablators. Anisotropy of these parameters was also necessary to verify whether multi-directional considerations were necessary in an analytical model.

The experiment for obtaining these parameters consisted of a pressure drop measurement made at various mass flow rates. It was desired to measure gases of different molecular sizes; time, however, permitted measurements using only dry air.

The experimental apparatus and a typical specimen configuration are shown schematically in Figure 26. A photograph of the experimental setup is shown in Figure 27. At the inlet side, the arrangement includes air from the laboratory supply and a heatless fractionator air drying unit. The mass flow was controlled by an inlet valve. Diffusion through a screen and flow straightening using aluminum hexagonal honeycomb were the means of obtaining uniform flow characteristics over the tube area. The sample fabrication incorporated a fiberglass outer ring that contained an O-ring seal to prevent flow around the outer edges of the sample. Connections for a U-tube type water-or-mercury manometer were located at either side of the sample to measure the pressure drops. Water was used for its greater sensitivity at low-pressure drops, and mercury was used at the higher levels. The mass flow was measured using a series of Fisher-Porter Model 10A1017A-LK flowmeters having a useful range from 1.4×10^{-4} cfm to 28 cfm. Some difficulty was encountered at the very low pressure drops because of flowmeter vapors that caused the float to stick to the tube walls. The vapors were assumed to have come either from the samples, the measurement tubes, or the system when repeatability measurements were performed.

The data were analyzed using the procedures described in Reference 5. The viscous resistance coefficient (α) and the inertial resistance coefficient (β) are characteristics of the porous specimen and depend on the state of the material; i.e., they will vary as the state of the material varies. The reference cited suggests combining

$$\frac{dP}{dy} = \alpha \mu v + \beta v^2 \tag{17}$$

and

$$p = \rho RT, \tag{18}$$

calling

$$pv = \dot{m}$$

to provide

$$\frac{1}{RT} \frac{dP^2}{dy} = 2\alpha\mu\dot{m} + 2\beta\dot{m}^2 \tag{19}$$

where

p = pressure

v = velocity

μ Gas viscosity

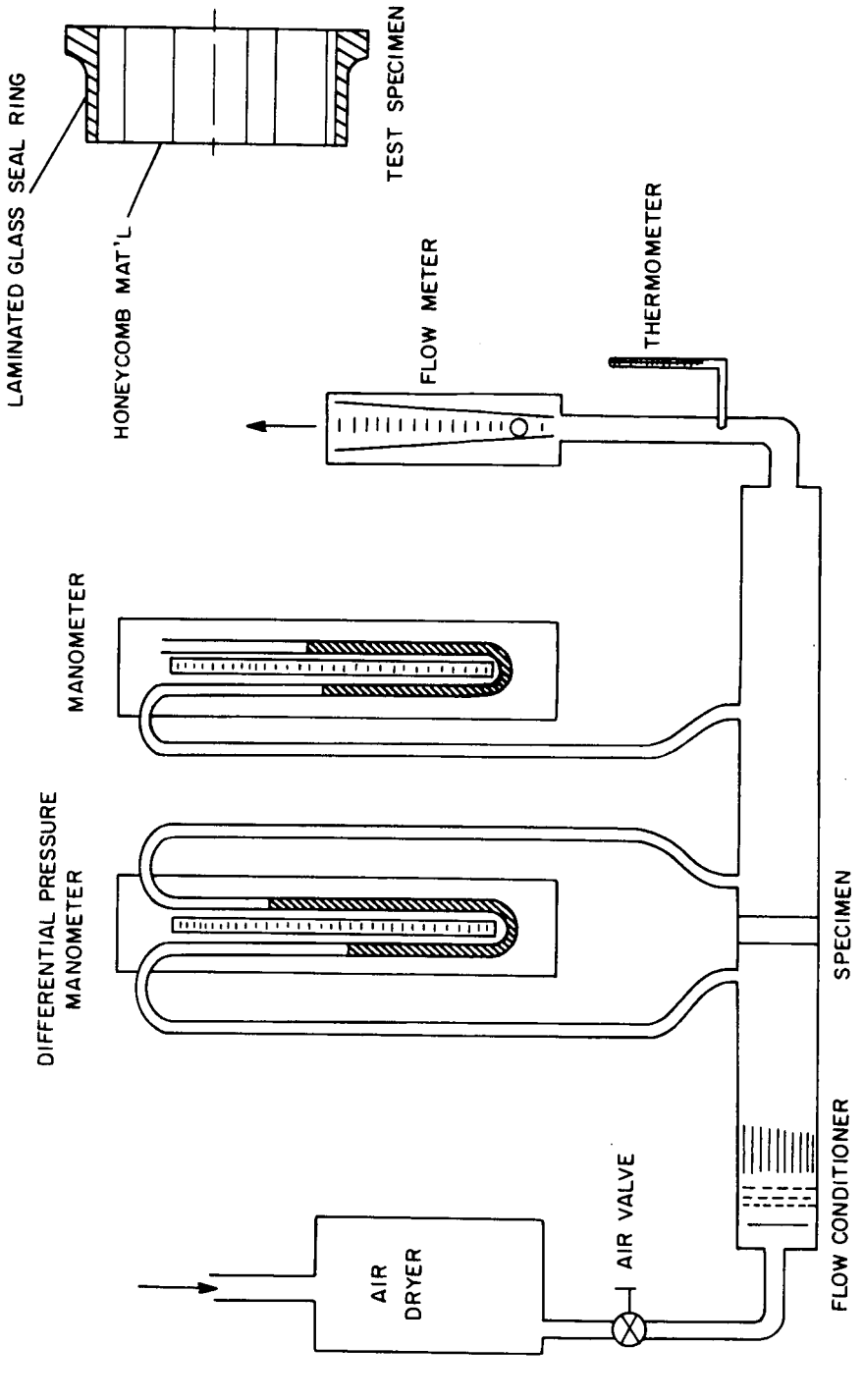


Figure 26 PRESSURE DROP SCHEMATIC

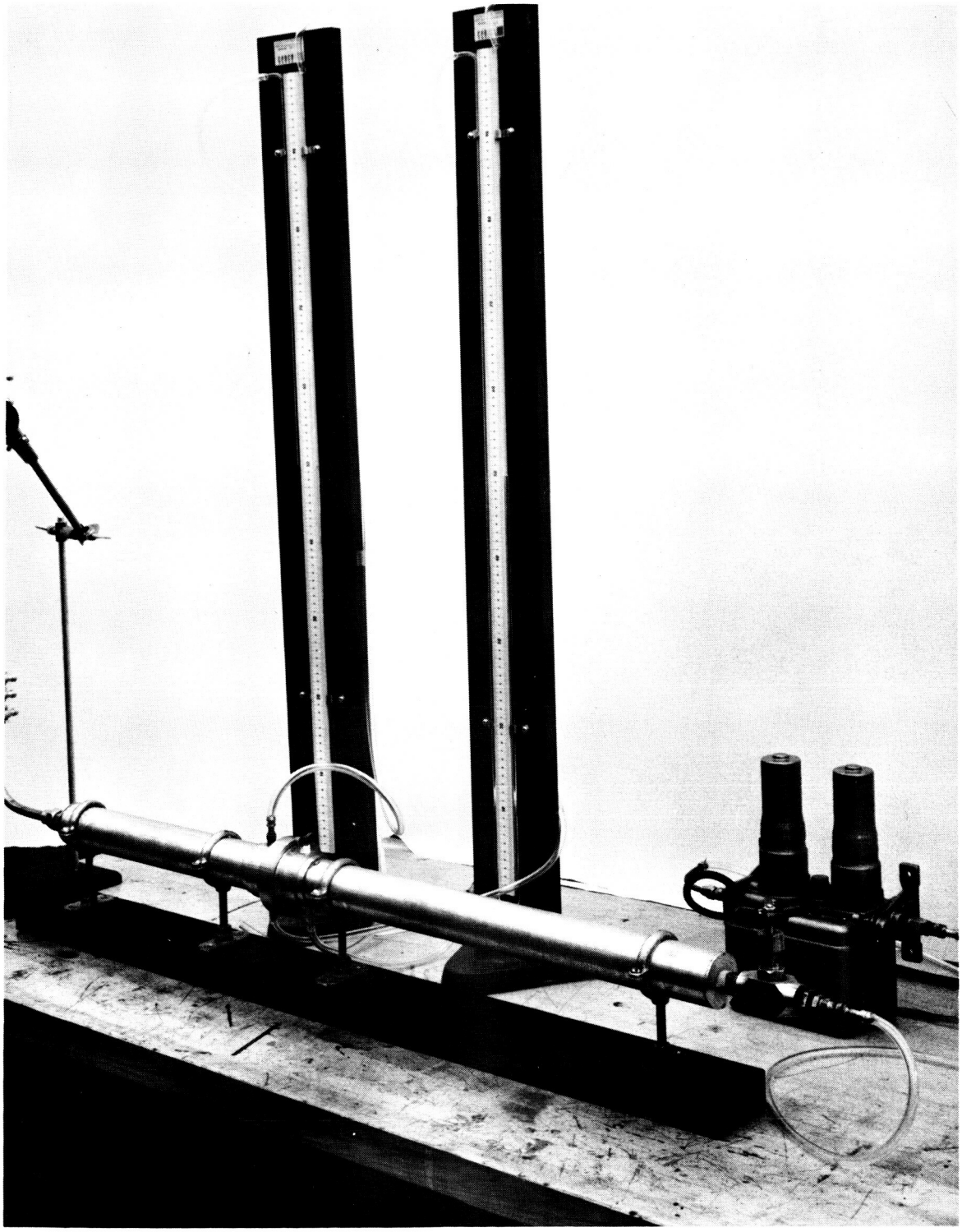


Figure 27 PRESSURE DROP APPARATUS

ρ = Gas density

R = Gas constant

The resistance coefficients were obtained from mass flow experiments by using small specimen thicknesses and by controlling pressures so that the resulting mass flow was essentially constant across the specimen diameter and the temperature varied insignificantly. The form of equations for experimental data reduction was as follows:

$$\frac{\Delta p^2}{RT\, L\dot{m}^2} = 2\alpha \frac{\mu}{\dot{m}} + 2\beta \,, \tag{20}$$

where

$$\Delta p^2 = P_i^2 - P_d^2 \,, \tag{21}$$

i = signifying inlet

d = downstream conditions

L = the specimen thickness

The left hand quantity of Equation (20) is a friction factor per unit length, and $\dot{m}/\mu$ is a Reynolds number per unit length.

The inertial and viscous resistance coefficients are usually computed by the least-squares method. If a sufficient amount of data is obtained and plotted as resistance factor versus Reynolds number, adherence or deviation from Darcy's law[5] can be established.

2.3.4 Thermogravimetric Analysis

Thermogravimetric analyses were performed during the program for several purposes. Most of the measurements were made for better interpretation of the results of steady-state measurements. A definition of the onset of degradation of a heat shield material, an attempt to show that a weight loss occurs when thermal conducitivity values undergo abrupt changes, and a need to interpret data obtained from differential scan calorimetry are several problem areas that are defined through the use of DTA information.

Two pieces of apparatus were used during the course of this contract. The first is a TGA and DTA apparatus manufactured by Harrop Precision Furnace Company (Figure 28). This apparatus has an auxiliary furnace wound with platinum-iridium wire to permit operation to 3600°F. The unit operates in oxidizing,

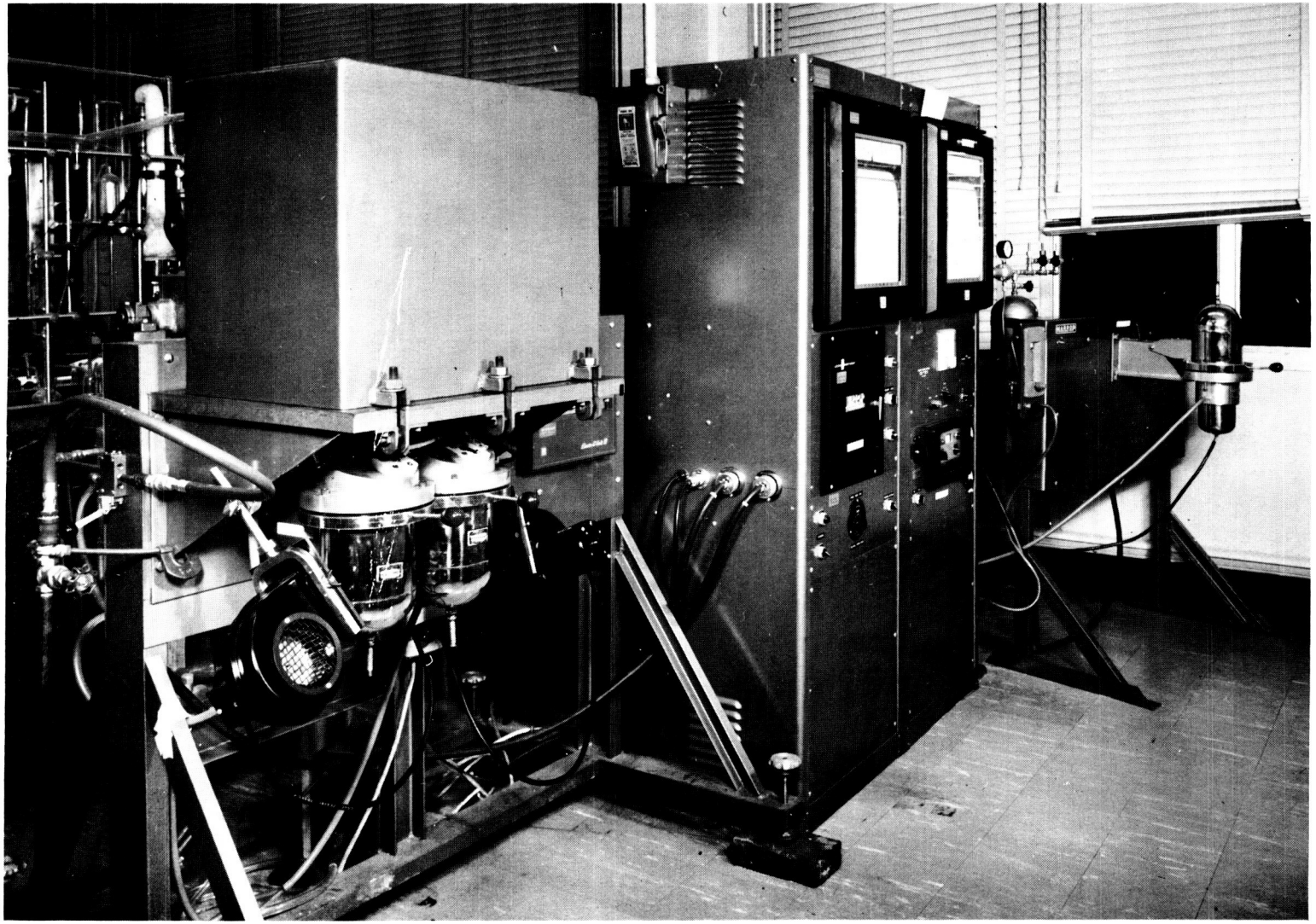


Figure 28 TGA-DTA THERMOANALYSIS EQUIPMENT, HARROP PRECISION FURNACE COMPANY

inert, and vacuum environments. The second apparatus is used for more precise measurements (sensitivity of 0.2 micrograms). This unit is shown schematically in Figure 29 and pictorially in Figure 30. The second apparatus can be used to 2700°F and can be operated in the variety of environments similar to those discussed above.

A detailed discussion of the apparatus operation will not be included in this report, since the apparatus is a standard laboratory device. TGA is the measurement of the weight-loss-at-temperature of a material when heated. It is subjected to various heating rates.

The data obtained from these devices have other important uses to analytical prediction models; these, however, were not required during this program; consequently, no evaluations were performed. Their principal use was to determine the ablator material upper temperature limits and to aid DSC analysis.

2.3.5 Heat of Combustion

Heat of combustion experiments were performed on a charring ablator to provide some knowledge of decomposed ablator material characteristics. Correlation feasability studies that would be useful for design purposes were attempted; time, however, and insufficient analysis of combustion residues limited the extent of these studies during this contract. The tests were made; no attempt was made, however, to correlate the data with other results.

Heat of combustion experiments were performed using a Parr Oxygen Bomb Calorimeter. The tests consisted of a series of measurements of materials in the virgin state materials precharred to 5000°F.

The results obtained during this phase of the contract can be labeled only as heat-of-decomposition, because a residual analysis was not completed. To reduce these data to heats-of-combustion, a correction would have to be included to account for the heat-of-formation of the residual components. Several of the charred materials decomposed completely; these cases can be designated heats-of-combustion.

The basic operation of the procedure used in these measurements consists of burning an accurately weighed sample in oxygen under high pressure. This is performed in a strong, thick-walled metal vessel (an "oxygen combustion bomb") securely protected against leakage or contamination. Provisions are made for supporting the sample within the bomb, filling the bomb with compressed oxygen, igniting the sample, and releasing the residual gas when combustion is complete. Ignition is accomplished by passing an electric current through a short length of resistance wire that is in contact with the sample. The combustion takes place within a few seconds, with almost explosive violence, although there is no external evidence of the reaction. Extremely high shock and static pressures are retained in the bomb.

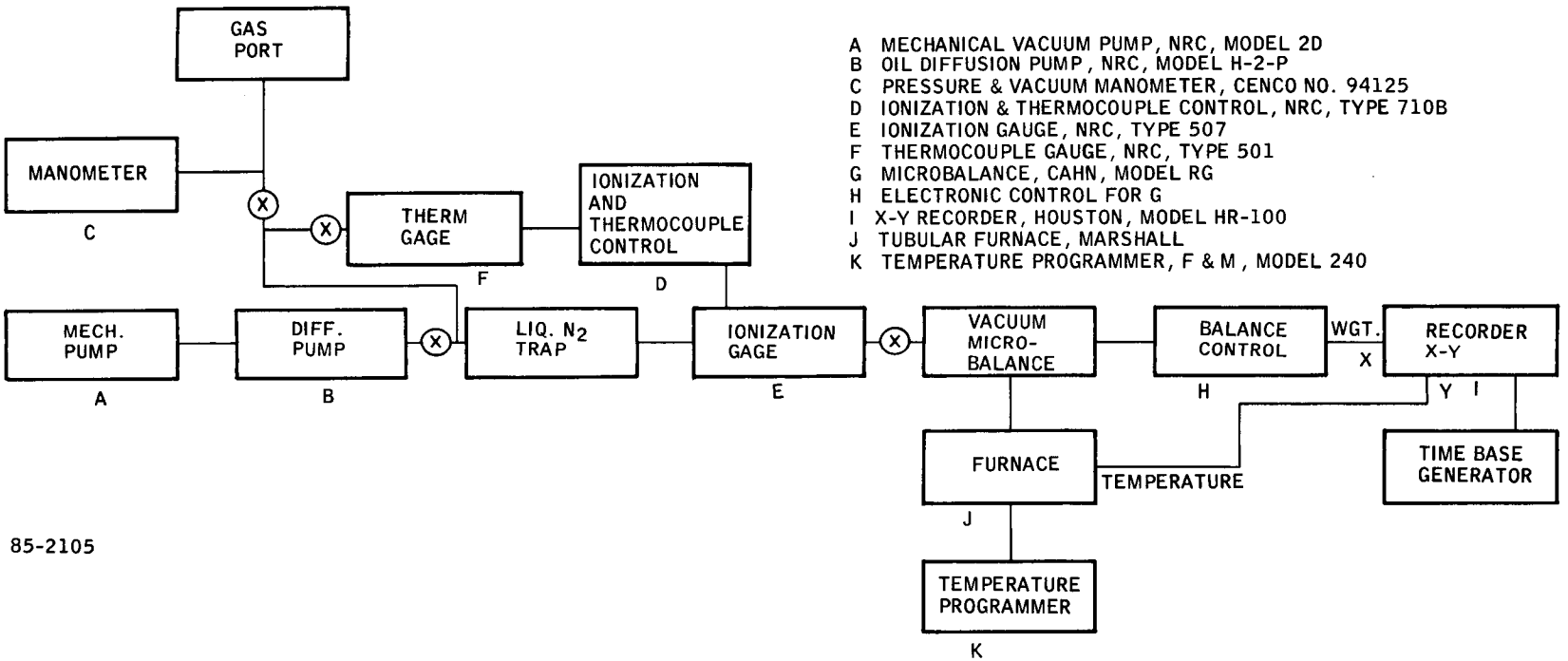


Figure 29 DIAGRAM OF APPARATUS FOR HIGH SENSITIVITY THERMOGRAVIMETRY

Figure 30 APPARATUS FOR HIGH SENSITIVITY TGA

Distilled water placed in the bottom of the bomb absorbs the soluble oxides and acids produced by the reaction. Valves are provided for releasing the residual gases and collecting them if desired.

Calorific tests are made in a thermally insulated jacket that surrounds the bucket containing the bomb, which is submerged in a measured quantity of water. Precise temperature measurements taken before, during, and after combustion are used to determine the heat-of-combustion of the sample.

The unit is adiabatic; thus the heat transfer between the calorimeter and its surroundings is a negligible quantity, and corrections for radiation losses are eliminated. The adiabatic design embodies an oval shaped container that holds the bomb and a measured quantity of water within a chamber that is completely enclosed by a circulating water jacket. During the temperature rise after ignition, the operator maintains the temperature of the jacket equal to that of the calorimeter bucket by manually operating valves supplying hot or cold water from an external source.

The gross heat of combustion (H_g) is calculated using the following relationship:

$$H_g = \frac{\Delta t W - e_1 - e_3}{m} , \qquad (20)$$

where

Δt = $t_f - t_a$

t_f = final maximum temperature corrected for thermometer scale error

t_a = temperature at time of firing, corrected for thermometer scale error

W = energy equivalent of the calorimeter

e_1 = correction for heat-of-formation of nitric acid (HNO_3)

e_2 = correction for heat-of-formation of sulphuric acid (H_2SO_4)

e_3 = correction for heat-of-combustion of fuse wire

m = mass of sample in grams.

The gross heat-of-combustion is usually reported in preference to the net value, because of the difficulty of accurately determining the hydrogen content of the sample.

The apparatus was standardized using NBS-certified benzoic acid. The standardization provides the energy-or-water equivalent. This factor represents the combined heat capacity of the water bucket, of the water itself, of the bomb and its contents, and of the parts of the thermometer, stirrer, and bucket supports.

2.3.6 Optical Properties Measurements

The limited amount of optical measurements made during this contract was performed on a Beckman extended-range-ratio recording spectrophotometer. To understand the experiments performed, a short discussion of the measurement is summarized below.

2.3.6.1 Monochromator

The light path in a Monochromator can be described as follows: (Refer to Figure 31 for a schematic arrangement of the apparatus.) An image of light source (A) is focused by the condensing mirror (B) and the 45-degree mirror (C) on the entrance slits (D) and (E), the lower of two slits placed vertically over each other. Light falling on the collimating mirror (F) is rendered parallel and reflected toward the quartz prism (G). The back surface of the prism is aluminized so that light refracted at the first surface is reflected back through the prism undergoing further refraction. The collimating mirror then focuses the spectrum in the plane of the slits. Light of the wavelength for which the prism is driven passes out of the monochromator through the exit slit, through the reference cell (J), then through the absorption cell (M). The two paths are then directed to the appropriate pickup tube (P or Q) by the semi-aluminized rotating mirror (N). The output of the phototube is amplified, and the ratio of the intensity of the two paths is directly recorded as percentage of light transmitted.

The unit chops the source beam at 480 cps to secure the optimum signal-to-noise ratio with the lead sulphide detector and automatically switches the beam 15 times a second from the reference to the sample.

2.3.6.2 Reflectance

Figure 32 illustrates the positions of the component units and the light paths for monochromatic illumination. Total reflectance measurements were performed during the Contract. The type of measurement made with the integrating sphere reflectance unit is described as follows.

To measure the diffuse reflectance of a sample, the sample and reference are placed at the exit ports of the integrating sphere. The sample and reference exit ports are equipped with shift plates. When the shift plates are used for diffuse reflectance, they position the sample-and-reference normal to the radiation beam so that the specular component is rejected and only the diffuse reflectance reaches the detector. When the plates are used for total reflectance, they position the sample-and-reference at a 5-degree angle to the incident beam; the specular component as well as the diffuse component reaches the detector.

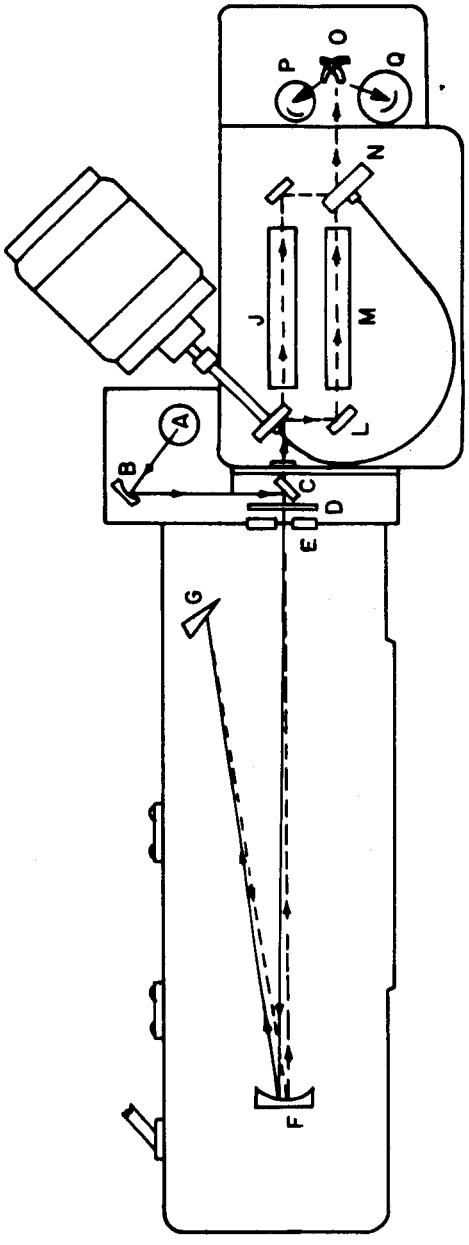


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Figure 31 MODEL DK, OPTICAL DIAGRAM, TRANSMISSIVITY

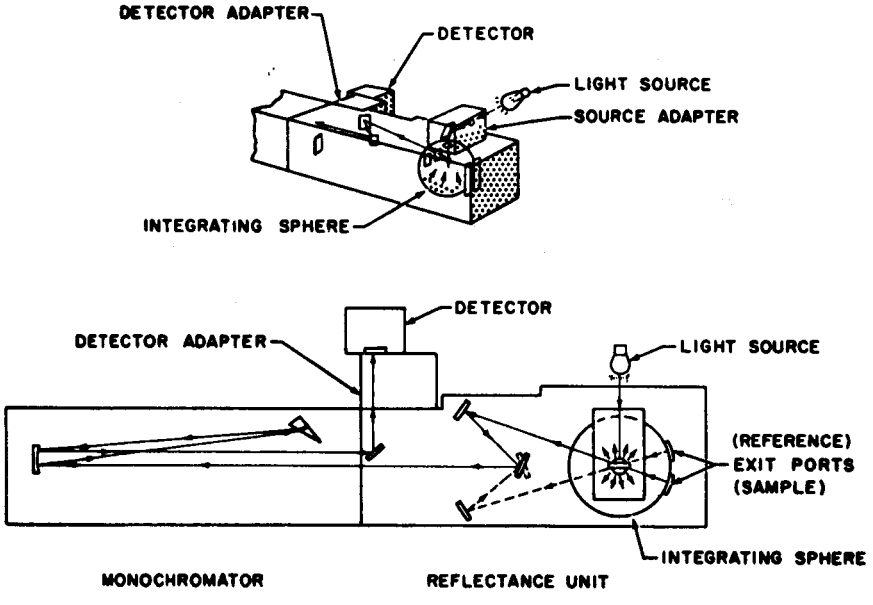

Figure 32 MONOCHROMATIC DETECTION LIGHT PATH

2.4 THERMAL CONDUCTIVITY CALIBRATION RESULTS

Many calibrations were performed during the period of this contract. Calibrations are necessary to establish accuracy; in most cases, however, they are used to determine differences between several pieces of apparatus and to check the apparatus when questionable data points are obtained.

Test performed during this program are illustrated in Figures 33 and 34 and are tabulated in Table VIII. Figure-33 tests were obtained using a fibrous glass sample certified by the NBS, and provide test certification in the range of 0.02 to 0.03 Btu/hr-ft-°F. Figure 34 illustrates the same type of tests; in this case, however, the thermal conductivity is approximately one magnitude higher at 0.15 Btu/hr-ft-°F. The test ranges are limited to those specified by the National Bureau of Standards so that material degradation do not introduce errors.

In a significant number of comparative calibrations of all automatic apparatus, the thermal conductivities differed from NBS values by approximately 3 percent, and the variation between different test apparatus was ±3 percent. Precision in all tests are assured within ±6 percent. (See Figure 34.)

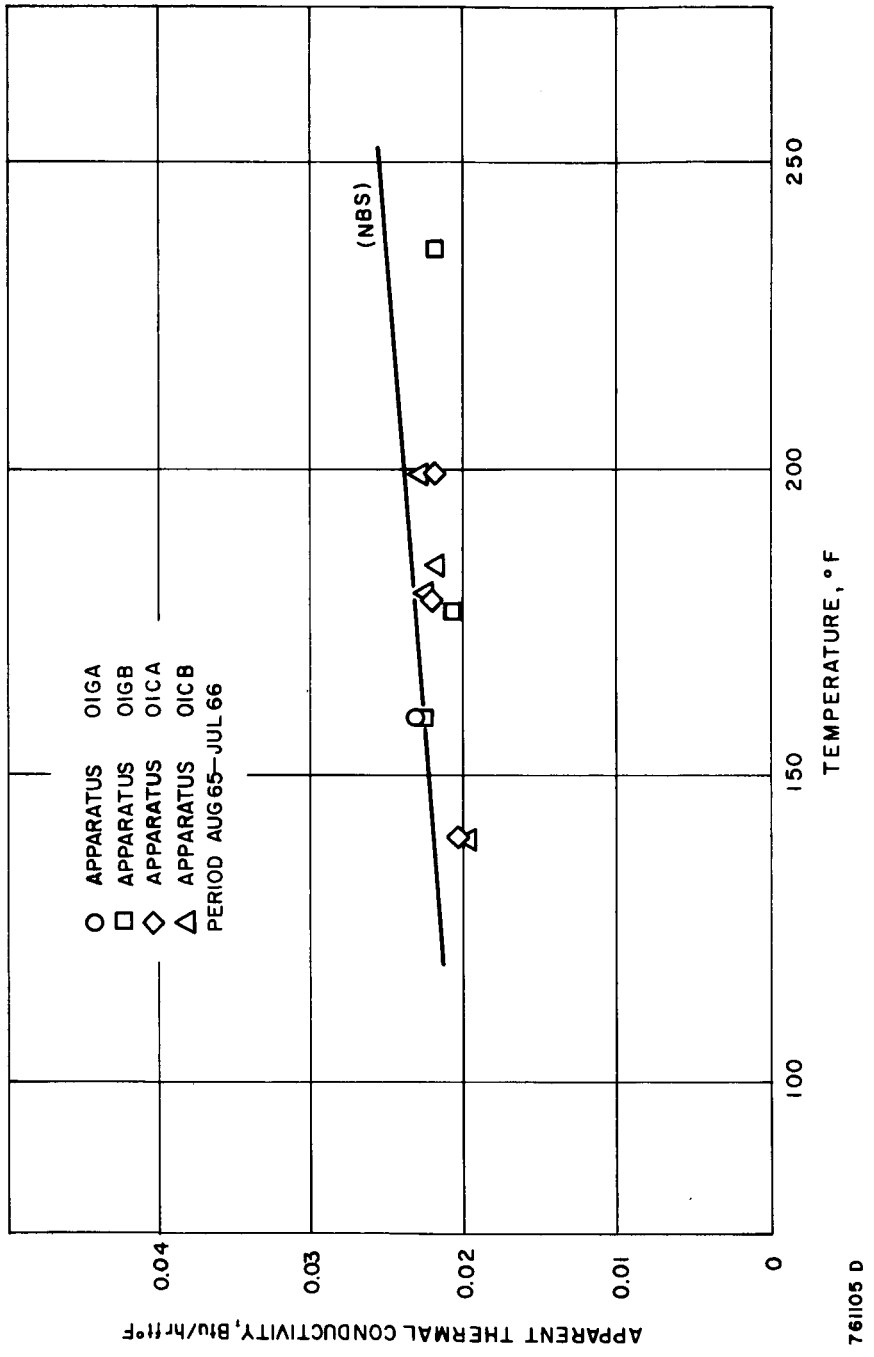

Figure 33 APPARENT THERMAL CONDUCTIVITY CALIBRATION TESTS USING NBS CERTIFIED FIBROUS BOARD

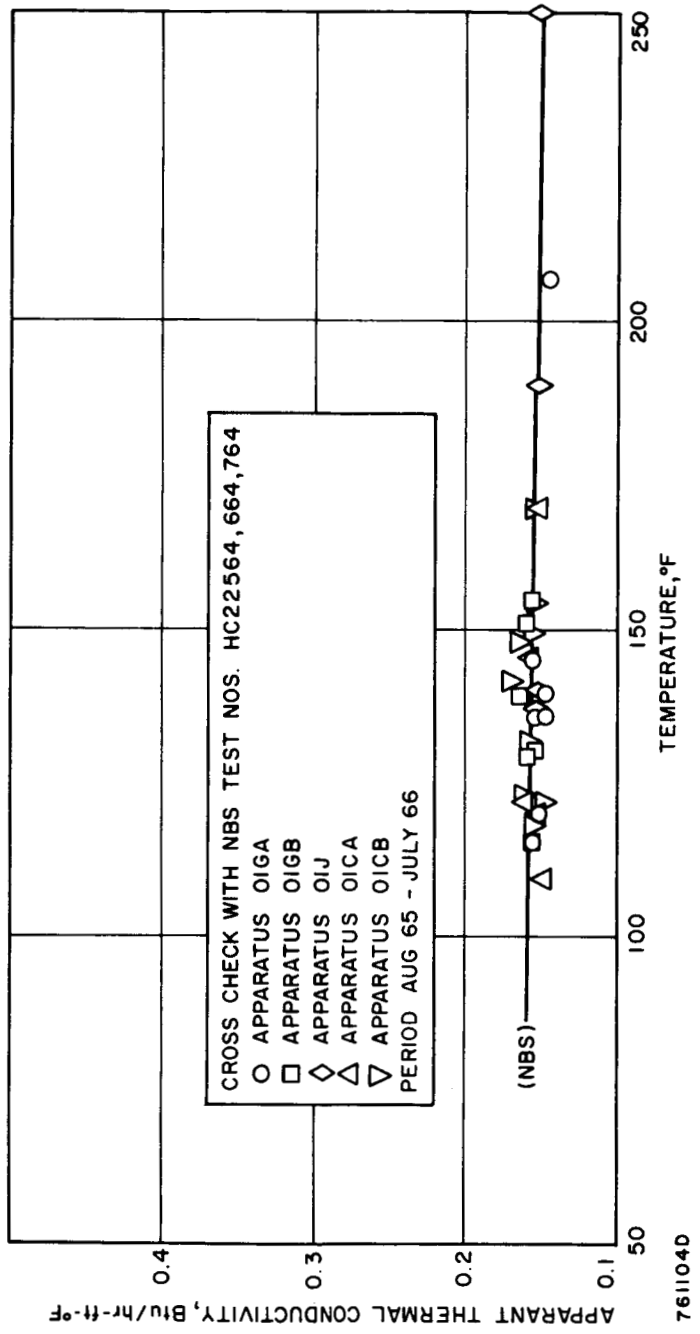

Figure 34 APPARENT THERMAL CONDUCTIVITY CALIBRATION TESTS USING NBS CERTIFIED SILICONE RUBBER

TABLE VIII

THERMAL CONDUCTIVITY CROSSCHECKS WITH NATIONAL BUREAU OF STANDARDS MEASUREMENTS

Date	Temp (° F)	K (Measured)	K(NBS)	Δ Percent
01GA				
14 Jan 66	116	0.156	0.157	-0.6
2 Aug 65	139	0.148	0.156	-5.1
2 Aug 65	207	0.145	0.153	-5.2
25 Oct 65	120	0.151	0.157	-3.8
4 Oct 65	159	0.0232	0.0228	+1.7
4 Oct 65	145	0.156	0.156	0
2 Jul 66	137	0.148	0.156	-5.1
7 Jun 66	136	0.154	0.156	-1.3
01GB				
14 Jan 66	116	0.155	0.157	-1.3
2 Aug 65	177	0.0207	0.0234	-10.3
2 Aug 65	236	0.0220	0.0251	-12.4
25 Oct 65	130	0.156	0.157	-0.6
4 Oct 65	160	0.0229	0.0228	+0.4
4 Oct 65	155	0.158	0.155	+1.9
24 Jan 66	128	0.160	0.155	+3.2
7 May 66	151	0.161	0.156	+3.2
7 Jun 66	138	0.164	0.157	+4.5
01J				
19 Jul 65	248	0.152	0.150	+1.3
1 Dec 65	189	0.152	0.153	-0.7
11 Feb 66	154	0.156	0.156	0
1 Jun 66	137	0.153	0.156	-1.9
2 Jul 66	140	0.152	0.156	-2.6
17 Jan 66	120	0.153	0.157	-2.6
4 Jan 66	146	0.157	0.156	+0.6
10 Sep 65	199	0.0220	0.0240	-8.3
25 Oct 65	108	0.149	0.158	-5.7
4 Oct 65	169	0.151	0.155	-2.6
4 Oct 65	178	0.0221	0.0235	-6.0
13 Sep 65	140	0.0204	0.0221	-7.7
26 Apr 66	123	0.160	0.157	+1.9
01CB				
14 Jan 66	118	0.156	0.157	-0.6
3 Jan 66	148	0.167	0.156	+7.1
4 Jan 66	142	0.171	0.156	+9.6
5 Jan 66	149	0.158	0.156	+1.3
10 Sep 65	199	0.0230	0.0242	-5.0
25 Oct 65	122	0.150	0.157	-4.5
4 Oct 65	169	0.156	0.155	+0.7
4 Oct 65	178	0.0225	0.0235	-4.3
13 Sep 65	140	0.0198	0.0221	-10.4
24 Aug 65	184	0.0219	0.0237	-7.6
23 Jun 65	132	0.160	0.157	+1.9
26 Apr 66	123	0.162	0.157	+3.2

3.0 TECHNICAL DISCUSSION: TEST MATERIALS

The purpose of this section is to discuss the factors that are pertinent to the results of the experimental data presented in Volume II. Each material classification is discussed separately to note the factors that are important to the interpretation of the test results. In some cases, the reason support measurements were undertaken is discussed, and their importance to design is pointed out. Some of the more important comments are repeated in Volume II; this section summarizes all the various factors considered during the program.

3.1 ANALYTICAL PREDICTIONS OF METAL HONEYCOMB PROPERTIES

Reference 6, 7 and 8 present analyses and some confirmation of the prediction of the effective thermal conductivity of honeycomb panels. Reference 6 presents an analysis that would account for the various modes of heat transfer in a honeycomb panel. The analysis provides the following relationships:

$$k_o = \frac{(Qa + Qm + Qr)\,1}{\Delta T} \quad , \tag{21}$$

where

k_o = effective thermal conductivity, Btu/hr-ft-°F

1 = core depth, ft

ΔT = temperature difference from the hot to cold side, °F,

and

$$Qa = \frac{qa}{A} = \frac{k_a}{1} \left(1 - \frac{\Delta A}{A}\right) \Delta T \tag{22}$$

$$Qm = \frac{qm}{A} = \frac{km}{1} \left(\frac{\Delta A}{A}\right) \Delta T \tag{23}$$

$$Qr = \frac{qr}{A} = 0.664\,(\lambda + 0.3)^{-0.69}\ \epsilon^{1.63(\lambda+1)^{-0.89}}\ \sigma([T_H]^4 - [T_C]^4) \tag{24}$$

where

Q = heat flux per unit area, (Btu/hr-ft^2)

k = thermal conductivity (Btu/hr-ft-°F)

l = core depth, ft

ΔA = cross-sectional area of conduction path through core material, ft^2

A = area, ft^2

λ = ratio of core height to cell diameter, l/d

ϵ = emissivity

σ = Stefan Boltzman constant, 0.476×10^{-12} $Btu/ft^2\text{-sec-}°R$,

and subscripts

a = air

m = metal

r = radiation

The foil edge to honeycomb area is expressed by

$$\frac{\Delta A}{A} = \frac{\sqrt{a^2 - x^2} + a}{2at} \tag{25}$$

where

t = foil thickness.

The other parameters are obtained from Figure 35. Figure 36 illustrates the dimensions used for defining the diameter (d) of a hexagonal cell (x). Table IX ia a tabulation of dimension a in Figure 35. Table X provides foil-edge-area-to-honeycomb-area ratios as determined both from actual gage measurements (Reference 9) and from those calculated. There was some difference in the values reported for gage-measured ratios and those calculated using Equation 25. The difference was significant and the source of major variations when predicted effective thermal conductivity values were compared with measured results. To determine the magnitude of these variations, the comparisons were made during the contract.

The prediction relationships given above refer primarily to honeycomb panels of which it is assumed that there are no other thermal resistive paths, as would be in brazed honeycomb panels. For aluminum honeycomb panels, which are adhesively bonded, the analysis does not hold, unless one considers the interface resistance that the adhesive offers to heat flow from the aluminum face plate to the core itself. Rather, the analysis would apply to the aluminum honeycomb core alone. Figure 37 illustrates the aluminum honeycomb core interface

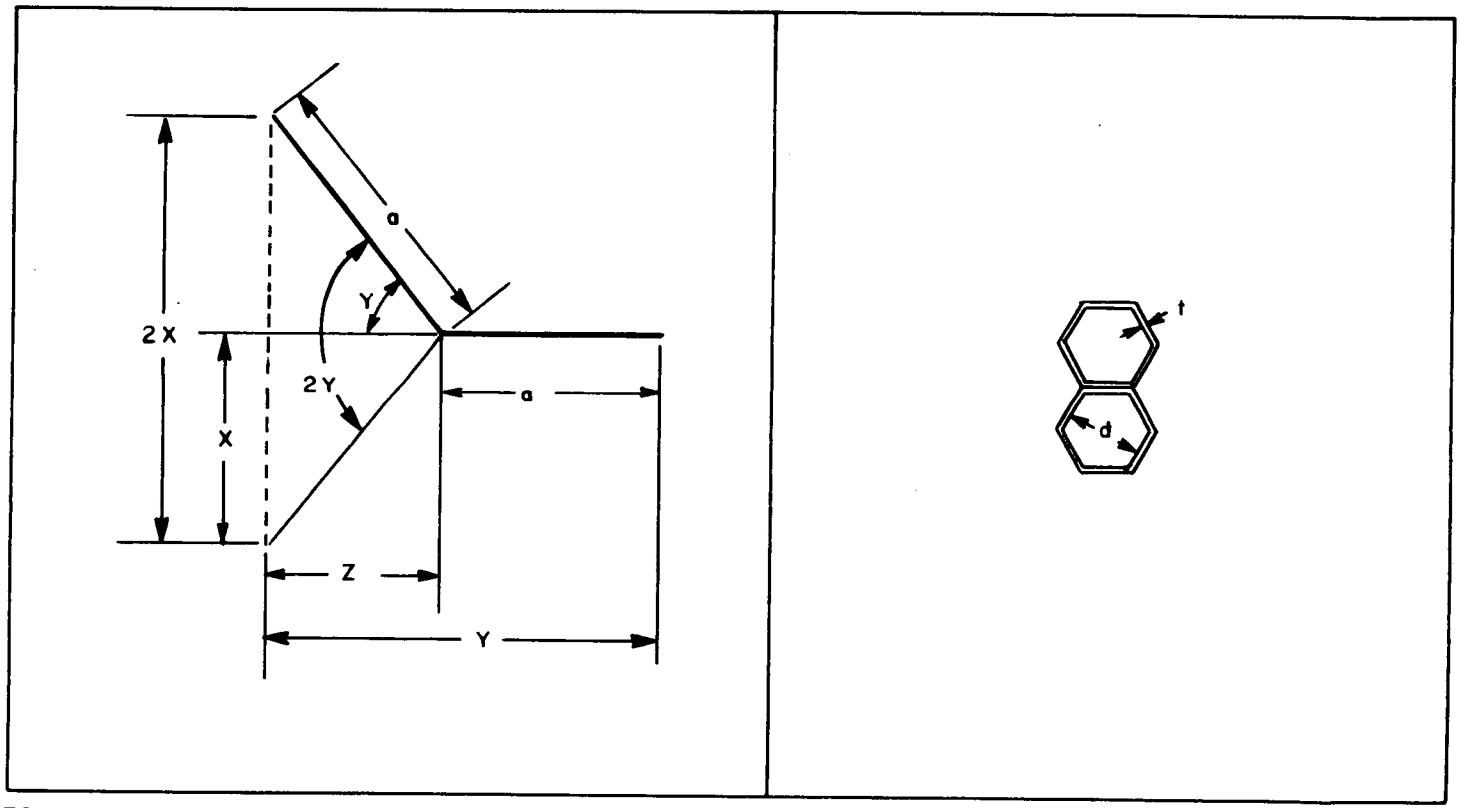

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Figure 35 HEXAGONAL CELL PARAMETERS, CELL GEOMETRY, AND PARAMETER DEFINITIONS

Figure 36 HEXAGONAL CELL PARAMETERS -- CELL DIMENSIONS

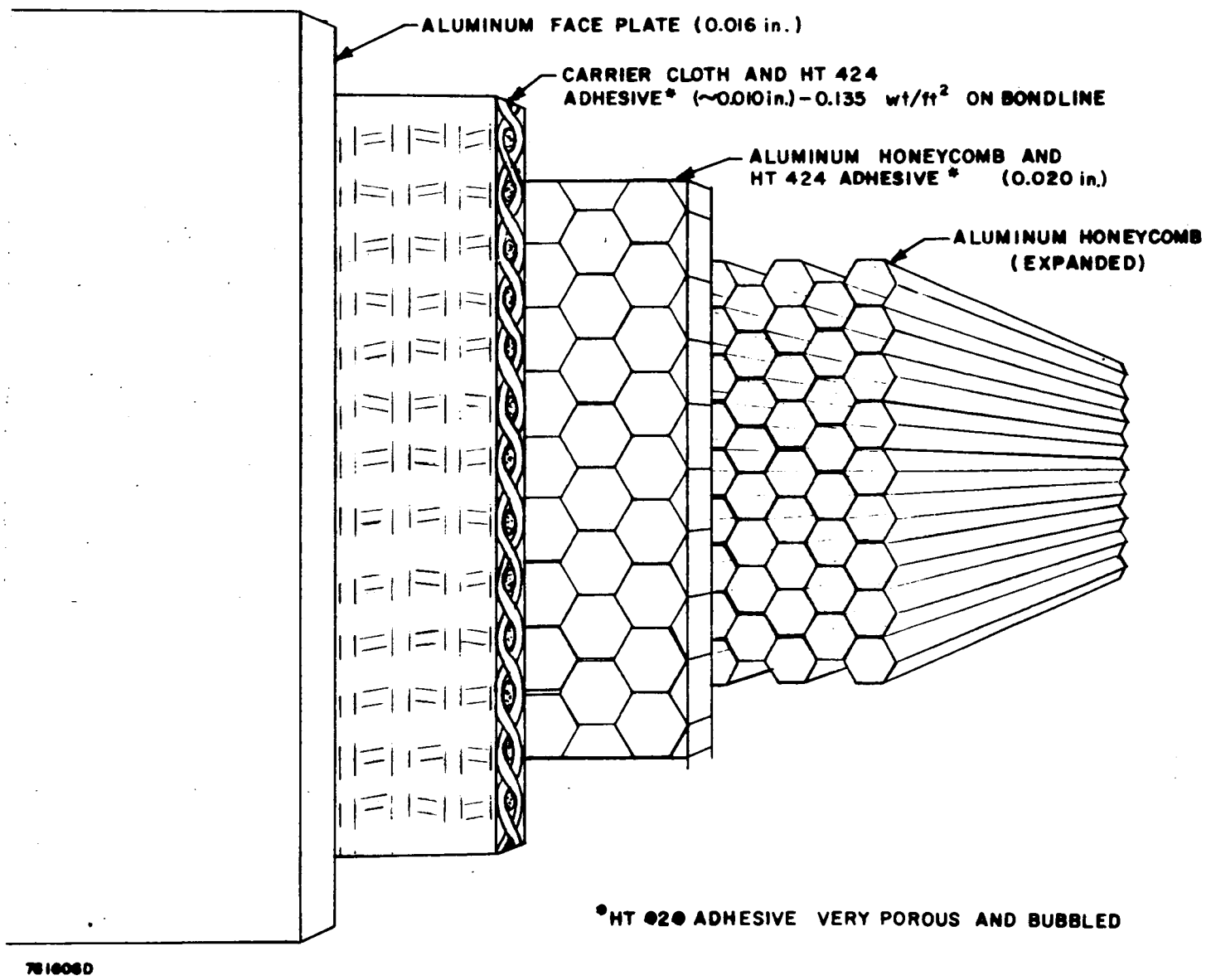

Figure 37 SCHEMATIC ILLUSTRATION OF BONDED ALUMINUM HONEYCOMB INTERFACE COMPONENTS

TABLE IX

CELL SIDE DIMENSION FOR VARIOUS COMMON CELL SIZES

Cell Size (inch)	Dimension A (inch)
1/8	0.0722
3/16	0.1082
1/4	0.1443
3/8	0.2165
3/4	0.4330

TABLE X

HONEYCOMB FOIL-EDGE AREA/HONEYCOMB AREA

Cell Size (inch)	Nom Gage (inch)	$\frac{\Delta A^*}{A}$	$\frac{\Delta A}{A(CALC)}$	Area* Covered (percent)	Area* Open (percent)
1/8	0.0010	0.028	0.021	3	97
	0.0015	0.038	0.032	4	96
	0.0030	0.074	0.064	7	93
3/16	0.0010	0.018	0.014	2	98
	0.0015	0.025	0.021	3	97
	0.0030	0.047	0.043	5	95
1/4	0.0010	0.014	0.011	1	99
	0.0015	0.019	0.016	2	98
	0.0030	0.037	0.032	4	96

*From actual gage

TABLE XI

PARAMETER VARIABLES USED FOR AVCO PREDICTION ANALYSIS

Core Depths (inch)	0.375, 100, and 2.00
Cell Sizes (inch)	1/8 and 1/4
Nominal Foil Size (inch)	0.001 and 0.003
Temperature Differential (° F)	5, 30, and 120
Emissivity	0.3 and 0.8

components. There are two composite layers in the heat flow path. These layers must be considered in any analytical prediction. Inward from the face plate, there is a layer of bond material (carrier cloth) impregnated with a very porous and bubbled HT 424 adhesive. The cloth-carrier layer prevents contact of the aluminum honeycomb and the face plate over the entire panel surface. Next to the cloth interface, there is a layer of honeycomb core material filled with the excess HT 424. The dimensions of these layers were found to be approximately 0.010 and 0.020 inch respectively. The dimensions are approximate because the adhesive was very irregular, bubbled, and non-uniform.

It was noted from a heat balance relationship that at elevated temperatures a convective heat transfer component was necessary. Post-test observations of samples subjected to high temperatures showed cell-wall and adhesive-bond discoloration. The node bond adhesive caused the most discoloration. It also caused the foil bond areas to be "pimpled" and some bonds were ruptured. Detailed studies were not performed during this contract due to the lack of time. Preliminary indications are that the node bond adhesive was the primary source of convection; further studies are suggested.

A very limited study of the correlations or predictability of effective thermal conductivity was performed during this program. In most references, where measurements were compared with predictions, it was found that some of the reports did not contain the thermal conductivity of the sandwich panel components used by Avco for predictions. Figures 38, 39, and 40 are the characteristic curves that were used for the preliminary Avco studies. The aluminum core thermal conductivity data were obtained from instrumented tests discussed later, because these were not available. Figure 41 illustrates the extreme variations reported for various aluminum alloys. In lieu of actual PH 15-7 Mo and PH 14-8 Mo data, the stainless-steel core-thermal-conductivity data were deduced from reference data noted on Figure 40. Analysis accuracy is limited to the accuracy of the basic information used for the prediction.

The magnitude of heat transfer was calculated using the extremes of all program conditions and are given in Table XI. The Table includes intermediate parameters for some conditions. Table XII presents the prediction extremes for evaluating the significance of each mode of heat transfer in the honeycomb core material only. As expected, it was found that the primary heat transfer component was the conduction of metal honeycomb core. The maximum conditions showed that the conductive heat transfer by air in the cell was only 0.3 percent of the metal,and that the radiation component was insignificant. The minimum conditions indicate that only 1.4 percent of metallic conduction was by air and that again the radiation was insignificant. The insignificance of radiation was expected since the maximum average temperature difference considered was 120° F.

Based on the component curves shown earlier, Figure 42 illustrates the predicted effective thermal conductivity of stainless steel. It was not possible to

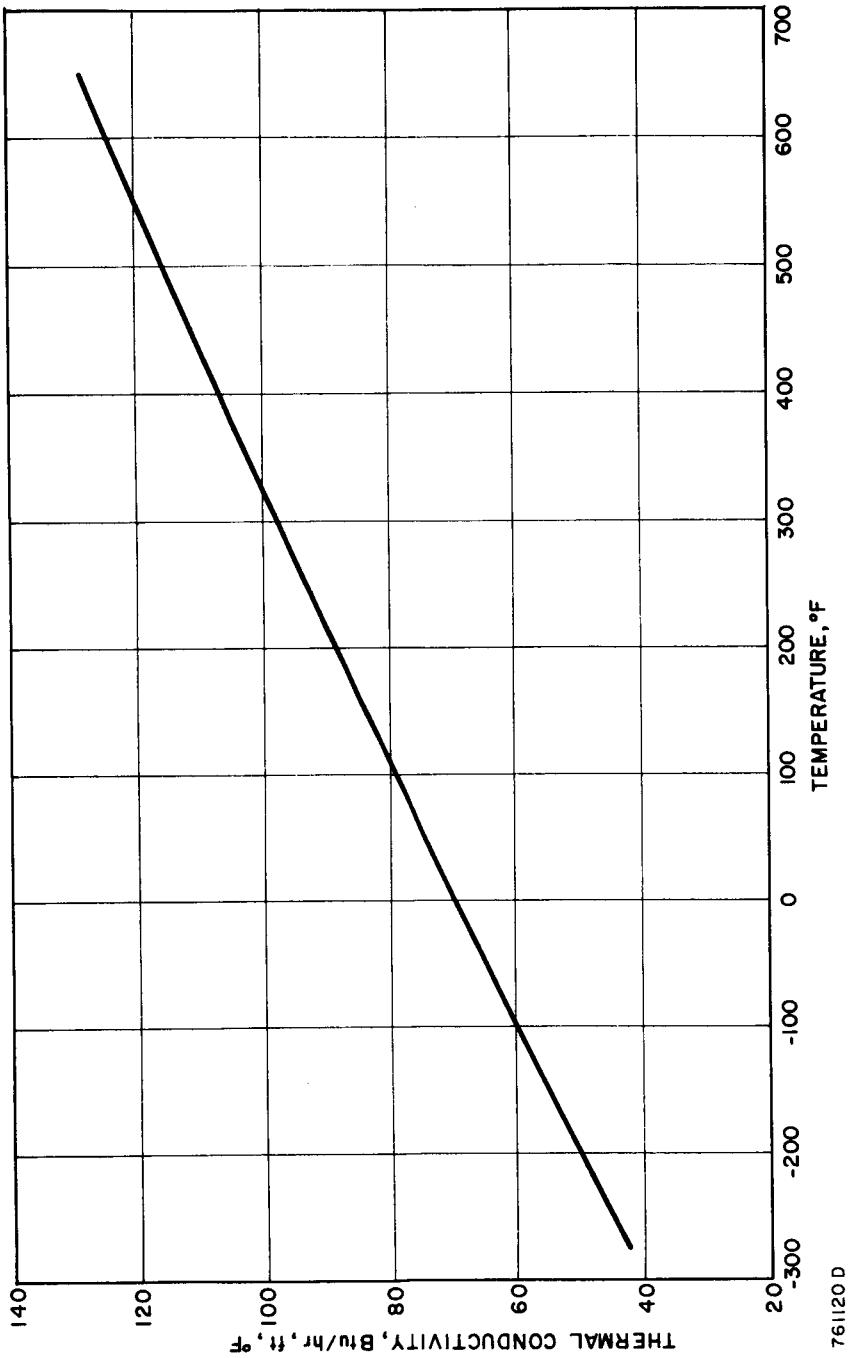

Figure 38 THERMAL CONDUCTIVITY OF ALUMINUM ALLOY 5052-H39

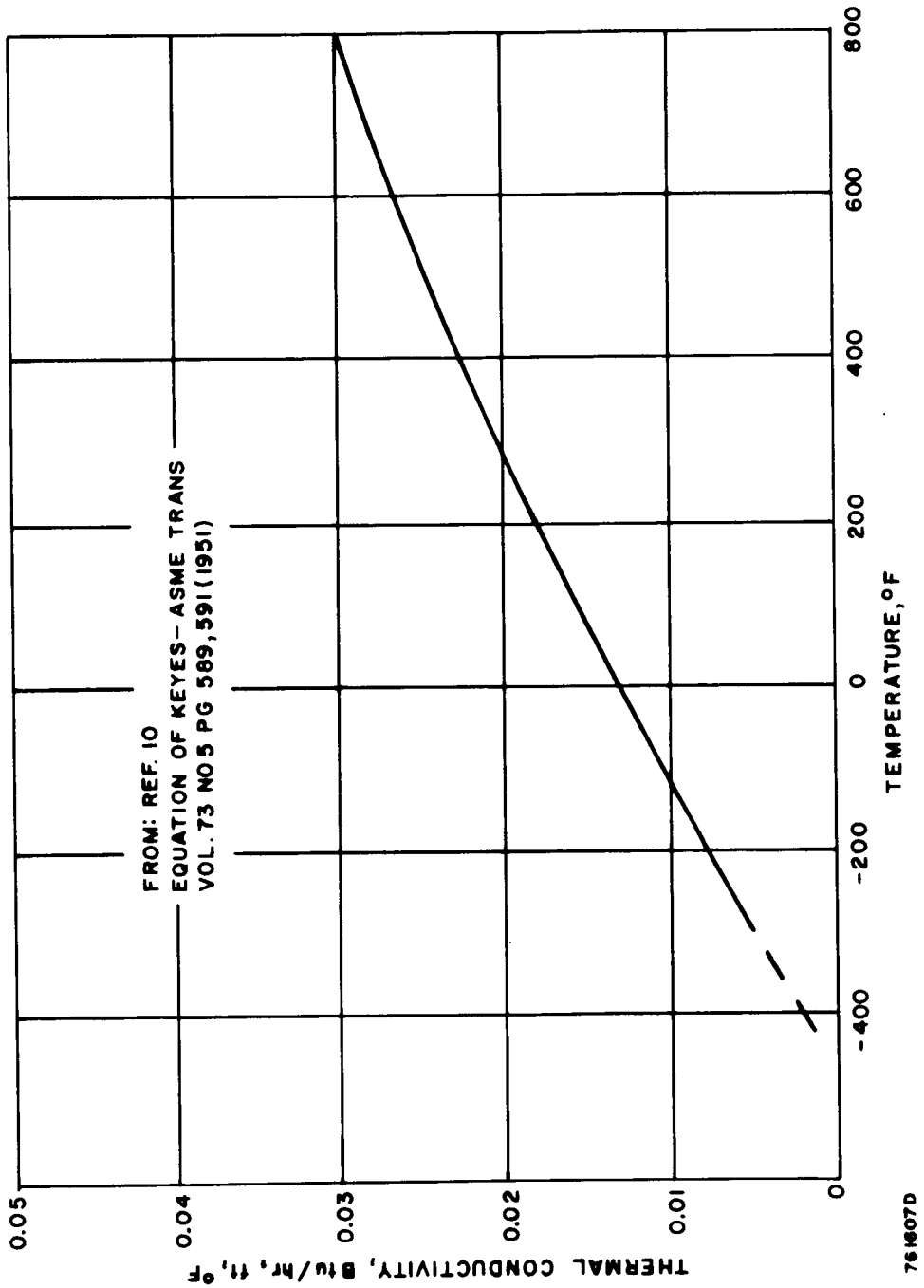

Figure 39 THERMAL CHARACTERIZATION OF AIR

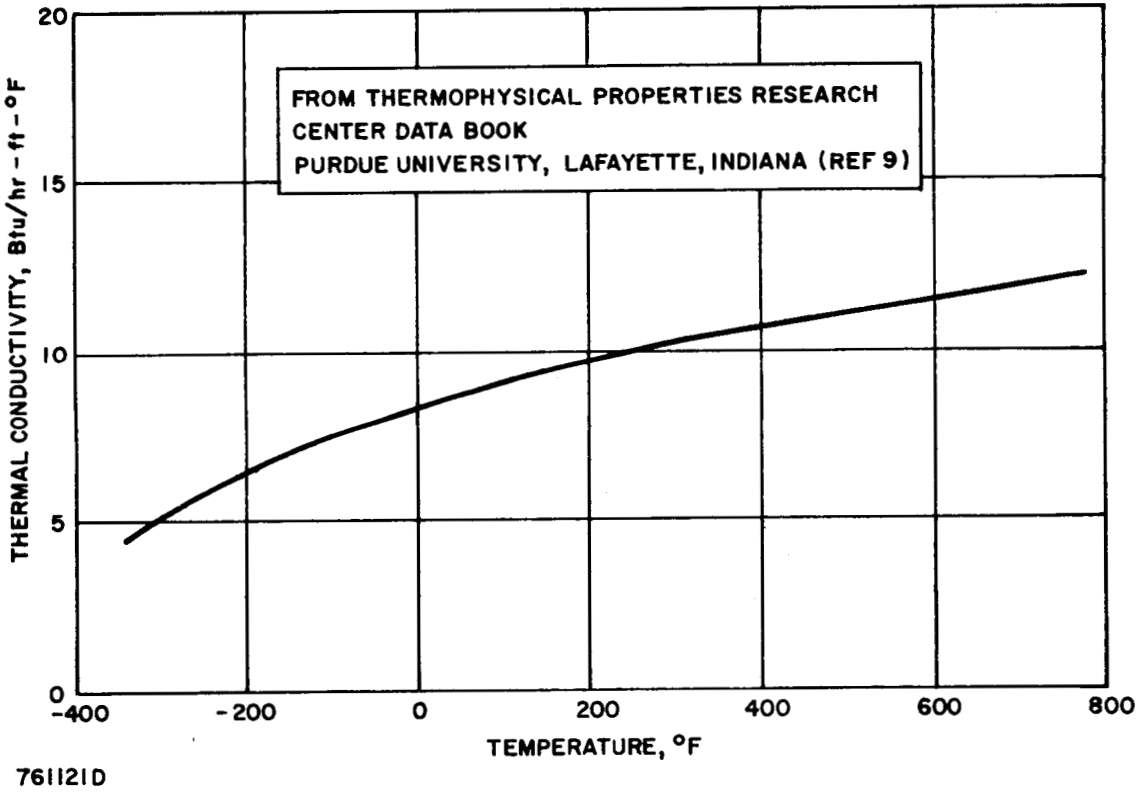

Figure 40 THERMAL CONDUCTIVITY OF STAINLESS STEEL

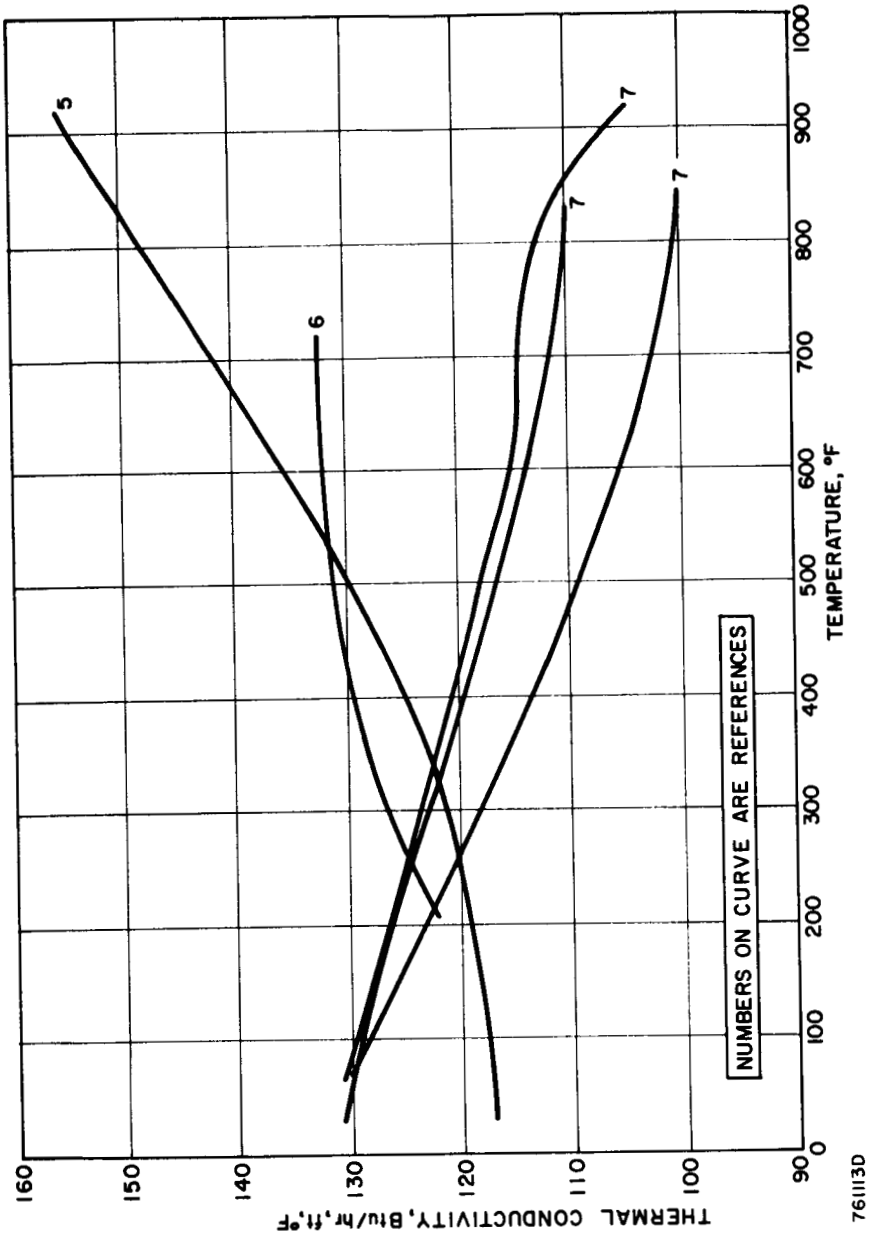

Figure 41 THERMAL CONDUCTIVITY OF ALUMINUM ILLUSTRATING MAGNITUDE OF REPORTED VARIATIONS

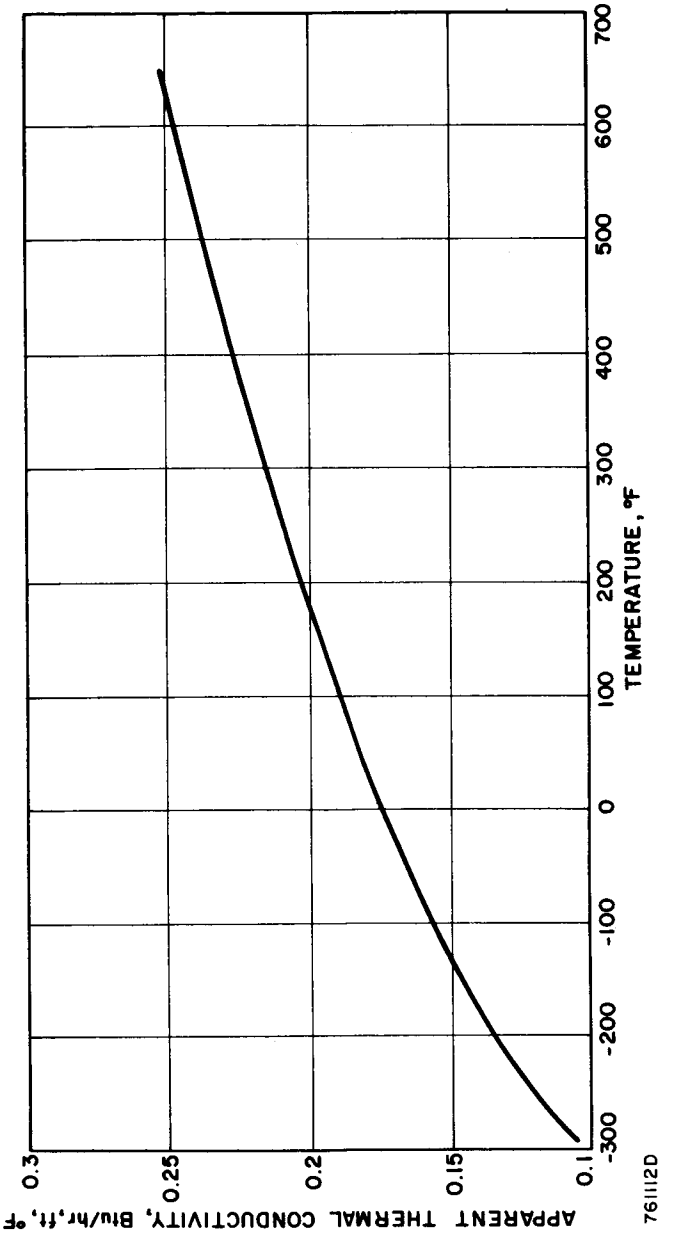

Figure 42 PREDICTED APPARENT THERMAL CONDUCTIVITY OF STAINLESS STEEL HONEYCOMB SANDWICH PANELS: 3/16-FIGURE 40-0.001P

TABLE XII

ANALYTICAL HEAT TRANSFER PREDICTION EXTREMES

Component	Heat Flux Btu/hr		Temp (° F)	Core Depth (inch)	Foil Sizes (inch)	Cell Size (inch)	Emissivity	Temperature (° F)
	Max	Min						
Metal foil	36,000		-100	3/8	0.003	1/8		120
Metal foil		50	600	2.0	0.001	1/4		5
Cell void	100		600	3/8	0.001	1/4		120
Cell void		0.73	600	2.0	0.003	1/8		5
Radiation	2.4×10^{-3}		600	3/8		1/4	0.3	120
Radiation		1×10^{-10}	100	2.0		1/8	0.8	5

use the reference analysis for a prediction of the effective thermal conductivity of aluminum honeycomb panels, and the analysis discussed in Reference 8 was beyond the scope of the contract.

3.2 ALUMINUM HONEYCOMB SANDWICH PANELS

3.2.1 General

A large part of the reported test program was directed toward measuring the thermal conductance of aluminum honeycomb panels. A literature search at the beginning of this contract disclosed a serious lack of these data, which were needed to meet the primary objective of correlating thermal conductance with the variables temperature, density, and pressure.

Evidence established that a significant factor in aluminum honeycomb panel data was the thermal resistance of the adhesive bond. It was also determined that bond layers from two vendor sources were different in porosity and porosity distribution. For additional evaluation, Avco prepared samples of HT 424 pressed to its carrier cloth. The evaluation consisted of measuring the total spectral reflectance, with reference to MgO, of this and other similar bond layers. The results of this test are shown in Figures 43 and 44. As expected, only the Avco sample of HT-424 bond material, being very dense (low porosity), exhibited high reflectance. A NASA MSC supplied sample appeared to have only a moderate distribution of porosity and showed a lower reflectance. The higher reflectance of the Albano Co. sample resulted from the method of measurement: the measurement of the NASA and Albano samples was made with the adhesive mounted on aluminum face plates; the very high porosity of the Albano adhesive allowed the aluminum back-up plate to contribute substantially to the reflectance. The measurements of Figures 43 and 44 were made using a Beckman DK-2 spectrophotometer, described earlier.

Sample fabrication from aluminum honeycomb panels proved to be a problem, which was discussed with panel suppliers. The suppliers explained special techniques used to avoid deformation of the foil or rupture of the adhesively bonded surface. Arrangements were made for finished thermal conductance samples to be supplied, conforming to Avco test specimen tolerances. The supplier "electric drilled" (arc-cut) the samples, thus eliminating the distortions that occur in machined (lathe or milling) samples.

With the exception of one set, all of the aluminum honeycomb panels used during this contract were obtained in two lots from the Albano Company of New York. The first lot consisted of all of the core depth variations in 1/4-inch nominal cell and 0.001-inch nominal foil sizes. The first lot was shipped to Avco as panels and was machined "in-house." The second lot consisted of all other variations, the one exception being a small panel of 0.96-inch core depth, 3/16-inch cell, and 0.0015-inch nominal foil size. This was supplied by NASA MSC

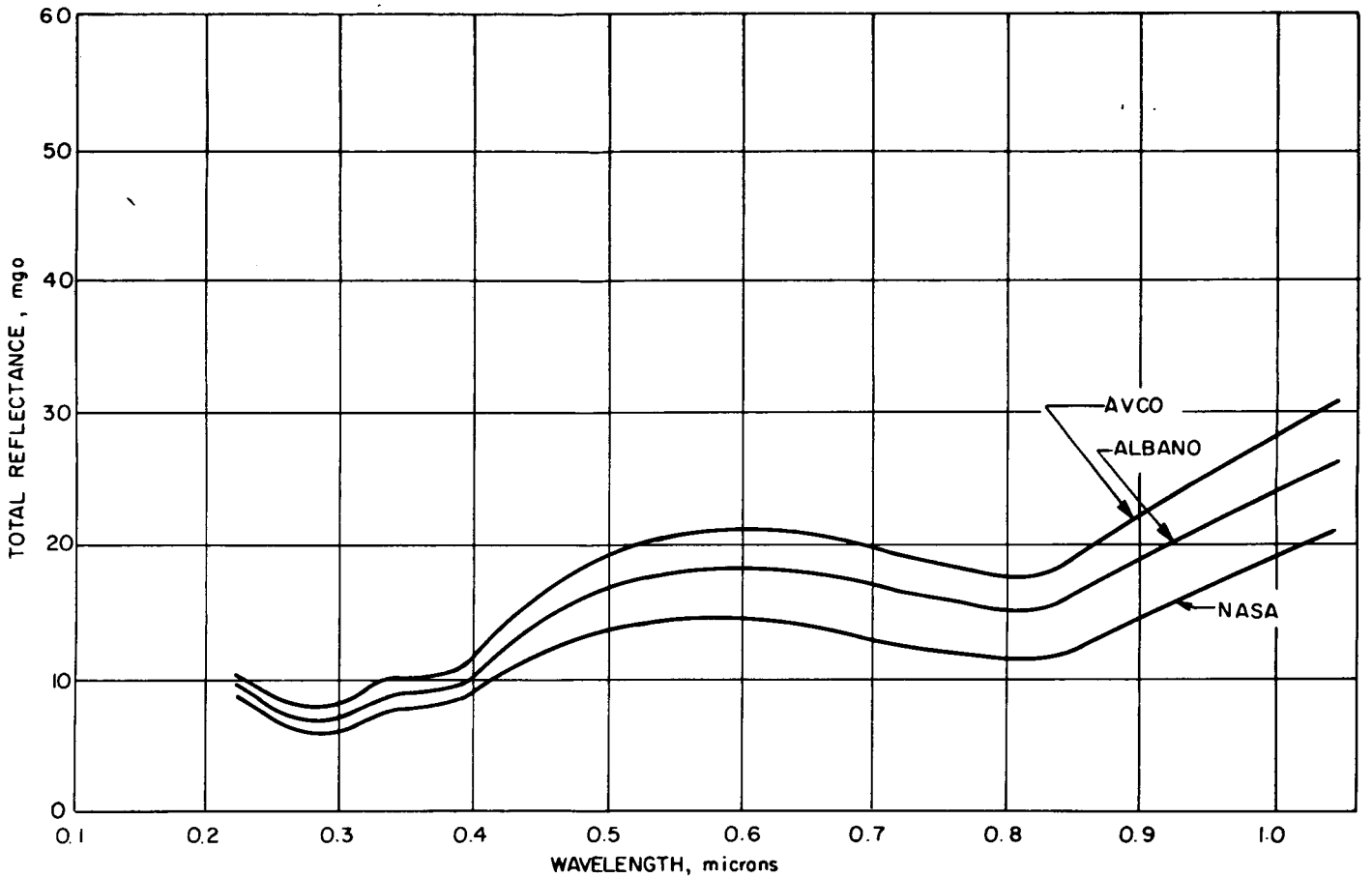

Figure 43 SPECTRAL TOTAL REFLECTANCE OF VARIOUS HT 424 ADHESIVES: ULTRAVIOLET AND VISIBLE RANGE

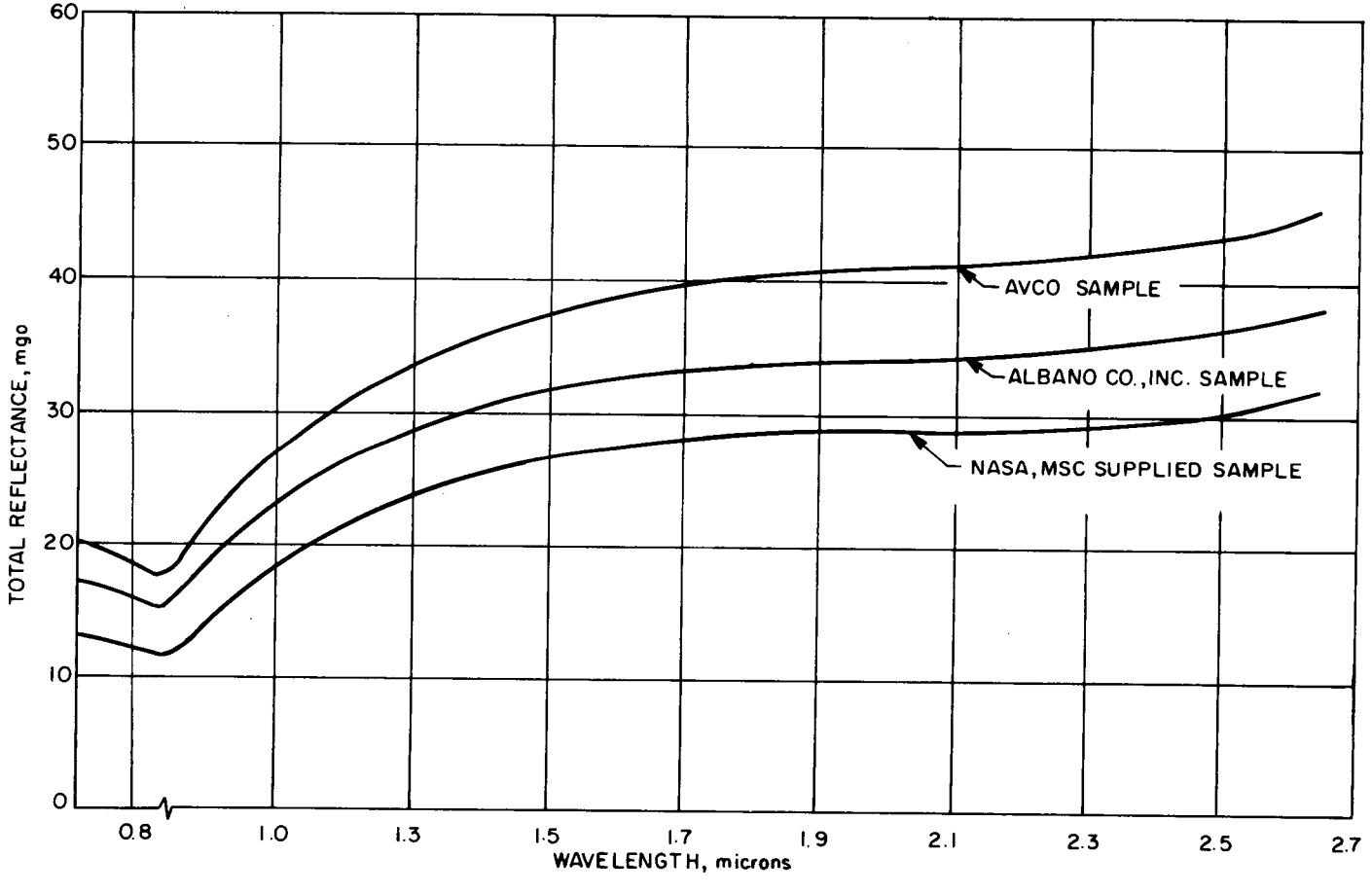

Figure 44 SPECTRAL TOTAL REFLECTANCE OF VARIOUS HT 424 ADHESIVES: NEAR INFRARED RANGE

and was sufficient in size to provide only one set of test specimens. The second lot of material was machined by Albano to Avco specimen drawings.

The only variation in all of the panels tested was the visual appearance of the face-plate adhesive discussed previously: the Albano interface material was lighter in color, bubbled, and the bubbles appeared to be broken. The variation was explained as possibly the result of a difference in the bond cure, the Albano being, supposedly, the proper cure.

3.2.2 Test Data

A large variety of aluminum panels was experimentally evaluated during this program. The criterion used to obtain test specimens was not restricted by close tolerances, since the object of the program was to evaluate the materials in a condition similar to those used in application. The panel materials were specified to be aluminum honeycomb core with hexagonal perforated cells and foil of nominal thickness, adhesively bonded with HT 424 to face plates of 2024-4T aluminum alloy, 0.016-inch thick and various core depths. Sufficient sets of specimens were prepared for concurrent tests to be run for repeat-evaluations at low and moderate temperatures under atmospheric and vacuum pressures. Data analysis of apparent thermal conductivity as a function of temperature and core thickness,for a particular cell and foil size, showed large data scatter that appeared dependent on core depth. It was noted that the curves of a specific core type showed a discontinuity, from the normal to the low temperature range. The experimental procedures were periodically verified, and the resulting data were used to study the variations and discontinuities. In addition to verifications with laboratory standards, a series of profile tests were performed on specimens having core depths of 2 inches. (See Figure 45, and 46.) The profile tests were useful to show that the temperature distributions along the core were linear (no center loss). Sufficient information was provided to determine the thermal conductivity of aluminum alloy 5052-H39; these data could not be located during an initial literature search. The thermal conductivity of the core material thus obtained, 182 specific data points were analyzed in detail. The analysis used the procedures described in Reference 6 to account for the temperature drop measured across the panel and that measured across the core was attributed to the temperature drop across the two interfaces (bond material). It was found that the interface material thermal conductivity was similar to measurements performed by this laboratory on the HT-424 adhesive. (See Volume II.) The core-and-interface apparent thermal conductivities and the data points mentioned above were used to calculate the foil edge to area ratio ($\Delta A / A$). It was found that the scatter and discontinuities over the temperature range were the result of slight variations in foil thickness. It was determined that the 1/8-, 3/16-, and 1/4-inch nominal cell sizes vary the foil edge to area ratio ($\Delta A / A$) 0.003, 0.002, and 0.001, respectively, for each 0.1 mil in foil thickness. As an example, this would mean that for a 3/16-inch cell, 0.001-inch nominal foil thickness, the ratio would vary 15% for each 0.1 mil foil thickness variation. The apparent thermal conductivity of the panel would vary accordingly.

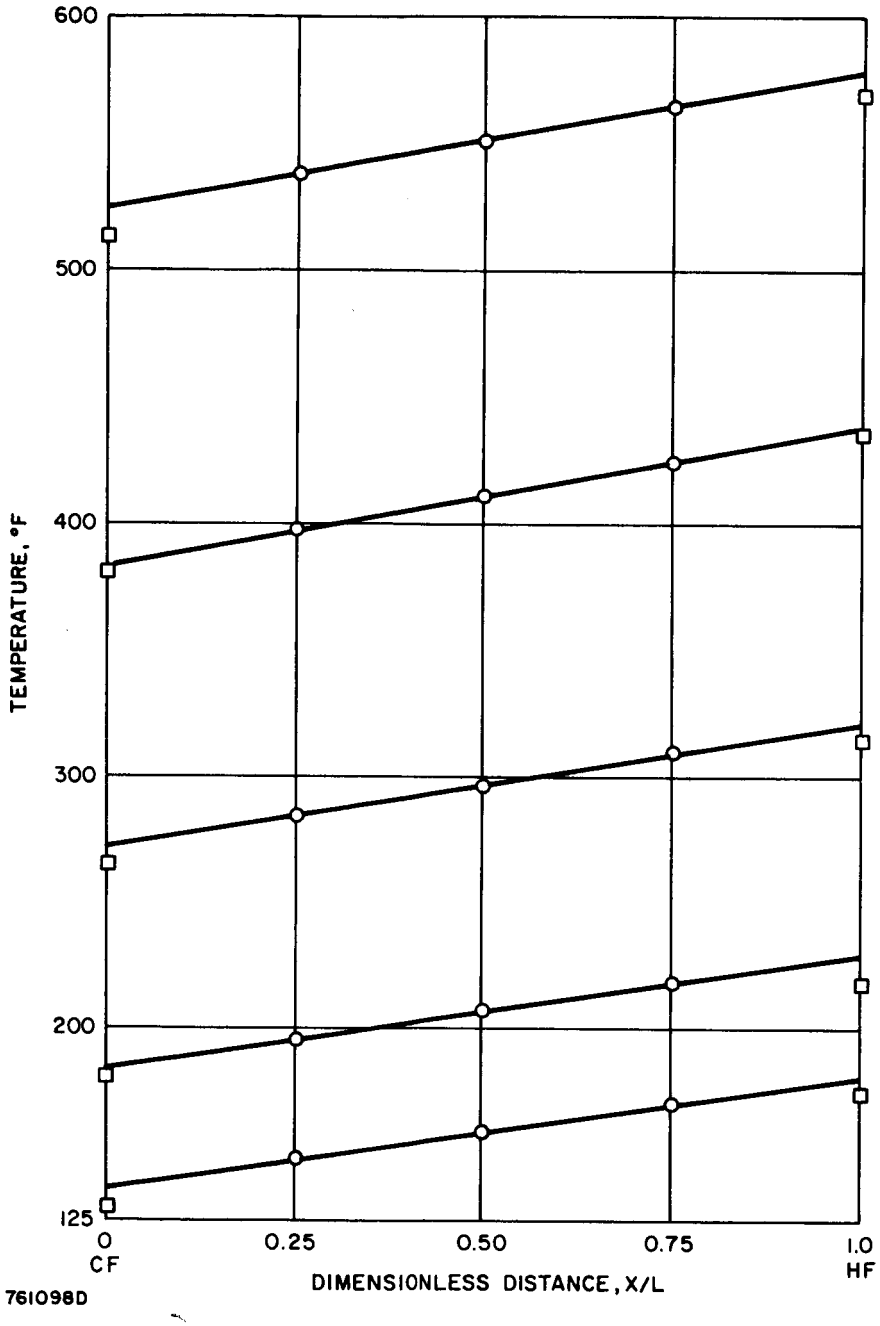

Figure 45 1.719-INCH ALUMINUM HONEYCOMB PROFILE, 1/4-5026-H39-0.001P

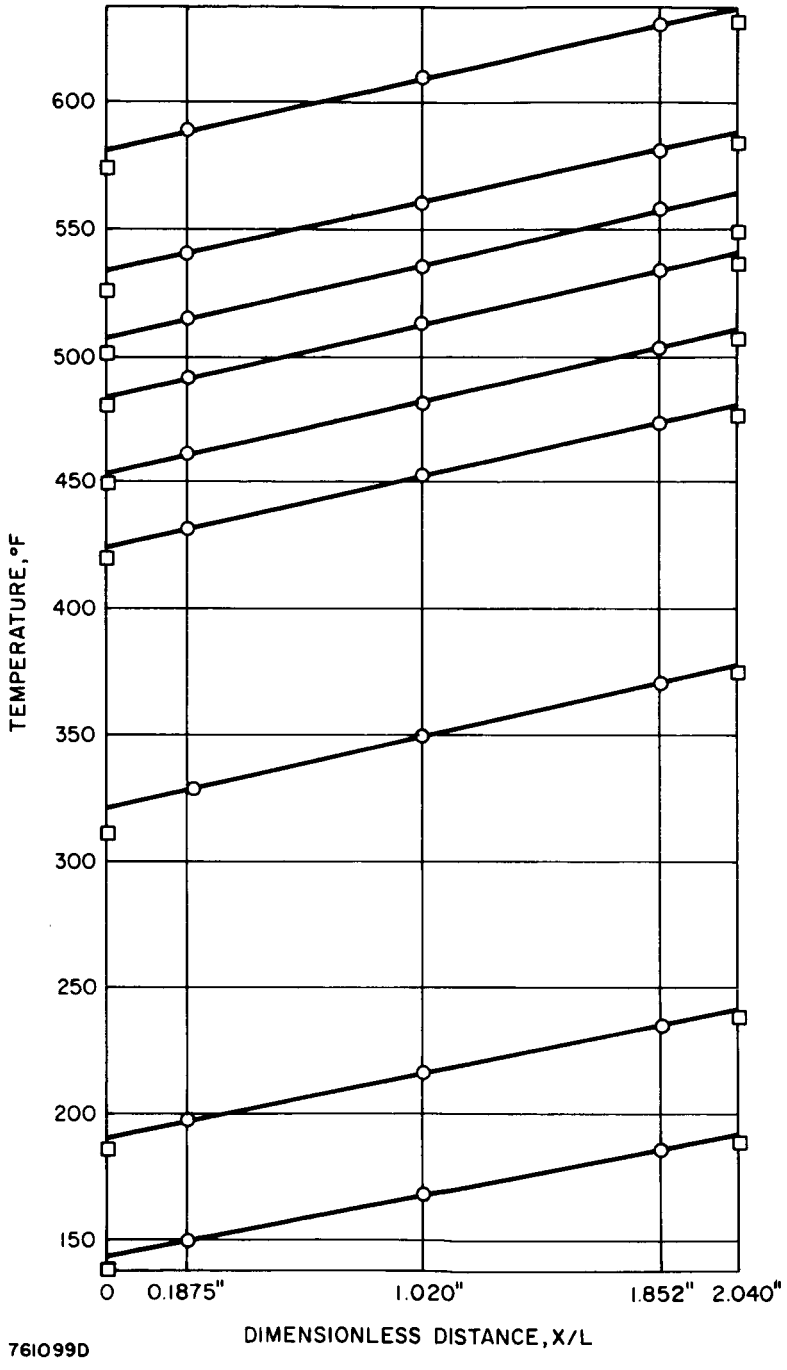

Figure 46 TEMPERATURE PROFILE TEST OF ALUMINUM HONEYCOMB, 1/4-5052-H39-0.001P, 2.00 INCH

Variations in panels from which samples were taken caused the effect noted. The objective set at the start of this program was not the measurement of apparent thermal conductivity, but that of apparent thermal conductance (k/l). Plotting families of thermal conductance of particular cell and foil sizes was found to be impractical. The most practical solution to the problem was to determine the mean thermal conductivity of a series,using least-square-fitting techniques, establish a standard deviation, and define curves for various core depths. This kind of a family of curves is valuable because it indicates the overlap that could occur due to material variations in the same type of panel. These curves are presented in Volume II of the report.

Consultations with the Hexcell Company application engineering group revealed that the node bond adhesive varied with cell type, foil size, and core depth. Such variations would cause variations in results, particularly at the higher temperature levels where node bond degradation occurs. The introduction of these variations made it difficult to establish the true effect of foil variation and node bond deterioration associated with the magnitude of heat transfer by convection.

3.3 STAINLESS STEEL HONEYCOMB SANDWICH PANELS

Open literature surveys found that stainless steel honeycomb sandwich panels, as with the aluminum, had not been measured to any extent. These panels have received some attention for analytical predictions, and a moderate effort is being devoted to other alloys. Most of the studies follow the prediction analysis of Reference 6, since in these cases the face plates are metal brazed to the honeycomb. The brazing eliminates the need for evaluating an interface component so long as its properties are not much different from the honeycomb metal. A variation from predicted values can be introduced in all kinds of core depths when the brazing material flows over a portion of the honeycomb core so that a ratio of foil edge to honeycomb area ($\Delta A/A$) cannot be practically established. The radiation transport at high temperatures can be also modified by the presence of the brazing alloy, which may produce emissivity different from that of the fabrication alloy.

It was found, as with the aluminum panels, that the component thermal properties of the alloys from which these panels were fabricated were not available. The properties used for prediction comparisons of stainless steel honeycomb were obtained from Reference 11. The stainless steel alloys 301 and 316 were the alloys primarily used,because they contained the same amount of chromium, about 7-percent nickel, and at least 2-percent molybdenum. The reference did not have properties of alloys even nearly similar to PH 15-7 Mo, from which the honeycomb sandwiches measured in this report were fabricated.

The material used for honeycomb panel tests were supplied by Aeronca Company of Middletown, Ohio, per B-70 specifications. All of the tests were performed on 1/2-inch core depth with face plates 0.008 inch and 0.015 inch in the one- and

two-piece configuration. The two face-plate thicknesses were used to evaluate the effect of these thicknesses and possible radial heat transfer when the face plate is in one or two sections.

The level of effort in this area was very small as compared to the aluminum honeycomb studies, and, as a result, correlations or any significant design comments could not be made.

A study like that performed on aluminum honeycomb is needed because of the increased use of stainless steel for higher temperature applications. It is postulated that the foil variations noted during the aluminum study, as well as the variation of $\Delta A/A$ due to brazing,alloy flow, is present in stainless steel panels.

3.4 ABLATORS

Experimental measurements of ablators were made in the virgin and charred states. The ablators measured were materials selected for this program by NASA MSC. Included in this selection were primary ablators for heat shields, compartment, pad, closeout, and edge members. In addition to these primary ablators, boost protective covers and ascent heat shield were considered.

The charred state of the primary ablator and the way in which the thermal properties were measured were dictated by the charring ablator program in which the data were to be used. The properties in this program were measured in a way compatible with NASA MSC contract NAS 9-432, which Avco performed during the same contract period. The development of an advanced analytical program for charring ablators was the objective of the latter effort.

The virgin ablator properties were measured in a one-piece sample configuration as discussed previously. The charred state required some clarification of preparation, since a variety of procedures could have been used. Ideally one would like to have the effective thermophysical properties under reentry conditions. Since ideal materials are unlikely,the second choice would be the evaluation of materials as formed in a simulating environment such as electric arcs. Simulation provides materials that have indistinct zones and may contain many gradations of chars. The most practical approach was laboratory prepared chars (described earlier) that provide samples that are somewhat homogeneous, that can be characterized for constituents, and that are of sufficient size to be measured using state-of-the-art techniques. The charring technique required programmed heating and cooling of materials to a predetermined temperature level (described earlier). The samples were then tested, using guarded hot plate or other applicable techniques, to a mean temperature for which a valid temperature differential could be established, and the hot face was not allowed to exceed the pre-char temperature.

Extensive experience of this laboratory with charred materials indicated that apparent thermal conductivity could be correlated with char temperature by the

use of a common mean measurement temperature. Sufficient tests were performed under this contract to provide a limited correlation.

The primary ablators considered in this program were the following:

a) Avcoat 5026-39 (Avco Corp.)

b) DC-325 (Dow Corning)

c) NASA, Purple Blend (NASA, Langley)

In all cases, the compositions of these materials were either not available, proprietary, or the formulation information was so sparse that its presentation would be of no value.

Data were obtained on the following materials that are usually used as ascent or protective covers:

a) Teflon

b) Polyethylene

c) Armstrong Cork A2755

d) Armstrong Cork A2755 fabricated as an ascent heat shield composite.

The materials measured during this phase of the program were non-homogeneous and of the diathermous type; consequently the data results are labeled only as "apparent" properties.

Materials whose decomposition characteristics were not known were subjected to TGA (thermogravimetric analysis). The material was then tested to a mean temperature not exceeding the hot face decomposition temperature. Measurements beyond the virgin material range were performed at incremental temperature levels by the use of the precharring procedure described earlier. Data obtained in this manner were considered more valid, since decomposition did not occur during steady-state measurement. The procedure also allows better physical characterization of the material measured.

Where some knowledge of the temperature variation of the specific heat was required, materials were evaluated using DSC (differential scan calorimetry). This procedure allowed quantitative evaluation of specific heat variation as a function of temperature. It also indicated reaction energies, should they occur within the instrument temperature range. Time permitted evaluation of the materials of the ablator type.

3.5 INSULATIONS

The insulations tested during this program were of the intercompartmental and laminated types. The intercompartmental insulations were TG 1500, a product of the H. I. Thompson Company, and NRC-2, a product of the National Research Corporation. The TG 15000 insulation is classed as matt-type and can be pre-formed to various configurations. The NRC-2 insulation is a laminated insulation (aluminized-mylar), which is usually used on cryogenic vessels. The third, and only other insulation that was a layer type, was SI-62, a product of the Linde Company. The material was made up of layers of low density insulation and aluminum foil.

The basic problems with insulations of the types measured (with the exception of TG 15000), is testing them under spacecraft conditions.

3.5.1 SI-62

SI-62 was tested in the Avco radial apparatus, the material wrapped around the heater assembly. Wrapping resulted in questionable density in the test area; thus, the test sample density has been reported with low confidence.

The maximum test temperature of this material was required to be 2000° F; it was found however, that the insulating layer between the aluminum foil deteriorated at a temperature less than 1000°F. The insulating layer deterioration resulted in conglomeration and serious sample swelling. The phenomenon was first noted during specific heat tests using drop calorimetry; as a result, the limiting temperature was not exceeded during thermal conductivity testing.

3.5.2 TG 15000

Thompsoglas 15,000 is a fiberglass matt bonded with silicone. It was tested in the guarded-hot-plate type apparatus. Test results at atmospheric pressure were no problem; vacuum tests, however, revealed unusual phenomena. Tests at moderate vacuums (3.5×10^{-2} torr) indicated a significant decrease in apparent thermal conductivity between 450 and 700° F. After 700° F, the apparent conductivity rose very rapidly to about 5 orders of magnitude within a 200° F (700 to 900° F) interval. Thermal conductivity at low vacuums (1.5×10^{-5} torr) exhibited the same trend, but the temperature range of the "dip" was reduced to 200° to 400°F. The tests were repeated several times, and the conclusion was that the silicone binder evaporated from the sample. The conclusion was tentatively verified when it was found that the apparatus components were coated with a thin layer of a transparent material. Time did not permit an analysis of the deposited material; during the subsequent test, however, the deposit re-occurred. The significance of these tests is that if the binder is released at lower temperatures with decreasing pressures, the binder may be the source of a convective heat transfer component during a mission.

3.5.3 NRC-2

Testing of NRC-2 was undertaken rather late in the program because the measurement techniques available were not compatible with the application configuration. NASA MSC decided upon, and approved of, the Avco standard low-temperature procedure using test samples made from various numbers of layers. The layers were stacked, and, after each test series, the thickness of the layers was reduced in preparation for the next series. The change of thickness provided data relationships as the number of sheets per inch of thickness varied. The ensuing tests were performed on decreased numbers of sheets so that data at the lowest number of sheets per inch practical were measured. Typical application test information can be obtained from test data extrapolations.

Post-test analysis of the NRC-2 test samples indicated a tendency for delamination of the aluminum layer from the substructure. Delamination was preferential and occurred on complete sheets and in many other localized areas. Sample deterioration was not expected, since the maximum hot face temperature did not exceed the specification (MB0135-016) maximum use temperature of 200° F. The maximum temperature of exposure was 130° F.

The material was exposed to a variety of environments that could have occurred during testing, and it was noted that exposure to conditions of high humidity caused the same phenomenon. There was no attempt to verify the phenomenon further. It was observed that the sheets should be in very light contact for delamination to occur. Verification that the phenomenon occurred when not in contact was not attempted.

The details of this phenomenon was not determined during this program. Tests at vacuum pressures were not performed on this material, because of the extended number of test performed at atmospheric pressure to determine if the loss of material was a real factor. It is assumed that the condition would be aggravated by a vacuum environment.

3.6 SEALS AND ADHESIVES

The tests on seal and adhesive materials were evaluated over the temperature range requested and, in some cases, to the decomposition temperature. There were no particular anomalies detected from the measurements (thermal conductivity and specific heat). The number of tests did not allow detailed analysis or correlations.

The materials evaluated were the following:

Adhesives	Seals
HT 424	RTV 560
Sylgard 182-2	
Epon 931	

4.0 REFERENCES

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5.0 APPENDIX: SCOPES OF ASTM-RECOMMENDED PRACTICES USED DURING CONTRACT

The ASTM-recommended practices listed below govern definition of terms, data handling, and sampling procedures as used by the Avco SSD Thermal Properties group. Although the procedures related to sampling are not entirely applicable to the limited testing reported in this document, they are presented and were utilized where relevant.

1. ASTM E177-61T. -- Tentatively recommended practice for "Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material".

SCOPE

The purpose of this recommended practice is to make clear some general concepts regarding the terms "precision" and "accuracy" as related to some physical property of a material.

2. ASTM E12-61T. -- Tentative definitions of "Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases"

SCOPE

No scope is presented since the specification deals with density definitions.

3. ASTM E29-60T. -- Tentatively recommended practice for "Designating Significant Places in Specified Limiting Values"

SCOPE

These recommended practices are intended to assist in the use of uniform methods of indicating the number of places of figures which are considered to be significant in specified limiting values; for example, specified maximum values and specified minimum values. Their aim is to clarify the intended meaning of specified limiting values with which observed values or calculated values obtained from tests are compared in determining conformance with specification.

4. ASTM E178-61T. -- Tentatively recommended practice for "Dealing with Outlying Observations"

SCOPE

This recommended practice deals with the problem of outlying observations. An outlying observation, or outlier, is one that appears to deviate markedly from other members of the set in which it occurs.

5. ASTM E105-58. -- Recommended practice for "Probability Sampling of Materials"

Definition

Probability sampling plans make use of the theory of probability to combine a suitable procedure for selecting sample items with an appropriate procedure for summarizing the test results so that inferences may be drawn and risks calculated from the test results by the theory of probability. For any given set of conditions there will be several possible plans, all valid, but differing in speed, simplicity, and cost.

6. ASTM E122-58. -- Recommended practice for "Choice of Samples Size to Estimate the Average Quality of a Lot or Process."

SCOPE

This recommended practice presents simple methods for calculating how many units to include in a sample in order to estimate, with a prescribed precision, the average of some characteristic for all units of a lot of material, or the average produced by a process.

7. ASTM E141-61. -- Recommended practice for "Acceptance of Evidence Based on the Results of Probability Sampling."

SCOPE

This recommended practice presents and defines a rule by which to accept or reject evidence based on samples. Such evidence may come from results of tests of samples of materials or from other sources pertinent to the decision to be made.

In the experimental evaluation of material property under some defined set of test conditions, various representative samples of the material are subjected to a standardized test procedure resulting in measurements which quantitatively describe the particular material property of interest. The evaluation is complicated by the fact that such measurements, when made relatively precise, exhibit variation. For the cases where the tests performed are of the destructive type, the variability observed among the measurements can usually be classified into one of two major categories as follows:

Process Variability. -- The variability due to a multitude of minor factors wherein the physical composition of the material differs to some degree from one batch to another, one manufacturer to another, even one sample from another sample of one batch processed by one manufacturer. This type variability simply illustrates the fact that perfect homogeneity of material can be thought of only in the abstract.

2. Measuring Variability. -- The variability due to another multitude of minor factors which are not functions of the material as such, but rather relating to such things as precision of measuring instrument, control of test conditions, environmental variations, human factors, and the like.

Although some exploratory work has been done for analytically estimating the relative contributions of each of these two major factors to the total variability observed (see reference 1A), in the general case the two cannot be separated analytically with the result that the conservative approach is used and the total observed variability is attributed to category (1) above.

A logical question then pertains to how a meaningful evaluation of the material property can be made on the basis of a relatively small number of samples whose respective property values vary, representing a much larger "population" of possible samples which possess the same characteristics of variability. The answer is in the field of Statistics which can be defined as "the science of the collection and classification of facts on the basis of relative number of occurrences as a ground for induction" (reference 2A).

The statistical evaluation recognizes that although the individual samples yield measurements which fluctuate randomly, taken in aggregate (i.e., the "population" of all possible samples theoretically), such measurements in many cases exhibit a consistent and predictable pattern. Thus, if an infinite number of samples were tested and a frequency distribution formed, such a distribution could be approximated by the familiar normal distribution function defined by:

$$f(x) = \frac{1}{\sqrt{2\pi\sigma}} \, e^{-\frac{(x-u)^2}{2\sigma^2}} \qquad (1A)$$

where u is the measurement value that occurs most frequently and represents the average or expected value of the distribution function, where σ^2 is a measure of variability and is a parameter characterizing the particular distribution function.

It should be noted that although the normal distribution function is a common mathematical model for approximating the true distribution function being studied, in particular situations other frequency distribution functions may be more valid representations (i.e., lognormal, Poisson, etc.).

Once the proper mathematical model has been selected, the statistical evaluation proceeds, on the basis of the relatively few samples tested, to made inferences concerning the characteristics of the mathematical model or the material property distribution function which it represents. For example, if the normal

distribution function is being used as the model, then inferences can be derived for the u and σ^2 parameters on the basis of a relatively small group of representative measurements. These inferences can be made in the form of point estimates as follows:

$$u = \bar{x} = \frac{\Sigma x_i}{n-1} \tag{2A}$$

$$\sigma^2 = s^2 = \frac{\Sigma(x_i - \bar{x})^2}{n-1} \tag{3A}$$

where

x_i = are the individual measurements

n = is the sample size or number of measurements

x = is the point estimate of μ

s^2 = the point estimate of σ^2.

The inferences can also be made in the form of confidence intervals which for estimating μ would be:

$$\bar{x} \pm t \frac{s}{\sqrt{n}} \tag{4A}$$

The t is a statistical tabular value associated with the Student or "t" distribution function which relates expected variability in the $\bar{x}$ and s^2 estimates by sample size when such samples are drawn at random from a normal distribution function. By the use of such statistical tables, confidence intervals at defined probability levels may be derived. For example, a 95 percent confidence interval estimate of μ would be derived by computing $\bar{x}$, s^2 and n from the measurements observed and selecting the appropriate t value from the tables available. The interpretation of the confidence intervals would be that the true μ parameter will be enclosed within the limits derived with 95 percent confidence. It should be noted that there isn't any confidence associated with the point estimate of μ unless the sample size is the population size. Similar theory and tables are available for estimating the σ^2 parameter and parameters of other theoretical frequency distribution functions (i. e., lognormal, Poisson, etc.).

Another type of inference made on the basis of a statistical evaluation is to derive a range, the bounds of which are called tolerance limits such that a certain percentage of the population individual measurements may be expected to

1A Mandel, J., The Measuring Process, National Bureau of Standards, Technometries (August 1959).

2A Webster's New Collegiate Dictionary, 2nd Ed.

3A Techniques of Statistical Analysis, Eisenhart, Hastey and Wallis, McGraw-Hill Book Co., Inc., 1947.

fall between these limits. Relative to the normal distribution function, the familar 3σ limits are of this type. It is well known that in this distribution function, "3σ" describes limits which enclose approximately 99.7 percent of the individual values. A useful inference, therefore, would be one which predicts on the basis of a representative sample, the limits within which a given percentage of future measurements can be expected to lie with a specified degree of confidence. For the normal distribution function, such tolerance limits are simply:

$$\bar{x} \pm Ks. \tag{5A}$$

The K is a statistical tabular value associated with the non-central T distribution function which relates the expected variability in the $\bar{x}$, s^2 estimates and the minimum percentage of the population values by sample size which the limits are intended to enclose. For example, 99 percent tolerance limits with 95 percent confidence would be derived by computing the x and s^2 from the measurements observed and selecting the appropriate K value from the tables available. The interpretation of these limits would be that 99 percent of the population individual values will lie within the limits derived with 95 percent confidence.

It should be noted that inference in terms of confidence limits or tolerance limits can be derived as <u>two-sided limits</u> or <u>one-sided limits</u>. The latter is interpreted in the tolerance limit case as: 99 percent of the population individual values will be above some derived limit, $\bar{x}-Ks$, with 95 percent confidence or, if appropriate, 99 percent of the population individual values will lie below some derived limit, $x-Ks$ with 95 percent confidence.

The above described confidence intervals or tolerance limits can be derived for any sample size greater than one. It is clear from an understanding of how they are derived that their usefulness depends upon the precision of estimate (width of limits). For example, the utility of tolerance limits for the one-sided lower limit example given above on the basis of a sample size of two is questionable since the appropriate K factor is 24.167. The appropriate sample size that should be used in particular situations is dictated on the basis of several factors which include the degree of precision required, the tolerable risks of being in error, and obvious economic considerations. It should be noted that RAD 16015, Rev. 1, specifies the required risks, which is to derive tolerance limits which include 99 percent of the population individual values with 95 percent confidence unless a proof test is performed on each delivered item. In certain cases, the requirement is modified to deriving tolerance limits which include 90 percent of the population values with 95 percent confidence. As stated above, this requirement can be met with any sample size. It is clear then that the actual sample size to be used depends on the other factors. Table A-1 presents a guide for determining the gain of precision against increased sample sizes. For realistic preliminary estimates of design parameters, a sample size of the order of 10 <u>recommends itself.</u> From Table A-1, it is noted that small

gain in precision is gained by sample sizes of 12 as compared to 10, whereas basing an estimating procedure on sample sizes less than 10 compounds the conservativeness already noted in the assumption of considering measuring variability as essentially material variability.

Although the above could be considered a practical procedure for deriving preliminary material property estimates, it must be emphasized that definitive estimates of these properties is a different problem. It will be recalled that the preliminary estimates are based on the assumption that a valid mathematical model was used for the actual population frequency distribution. For definitive estimates, therefore, it would be imperative to establish the validity of the assumed mathematical model. The samples sizes required for this type of information is appreciable and statistical studies of establishing definitive materials property values on the basis of sample sizes of the order of 300 can be cited. Nevertheless it can be restated that the determination of the proper sample size for particular situations relate to similar factors as described above for the preliminary estimates case. Of importance in the definitive estimates is that the distribution form of the individual values must be studied whereas in the preliminary estimate situation, the distribution form may be assumed in most cases.

TABLE A-I

TOLERANCE FACTORS FOR NORMAL DISTRIBUTION FUNCTIONS (See also Figure A-1)

M	K
2	24. 167
4	6. 440
6	4. 870
8	4. 278
10	3. 959
12	3. 758
14	3. 618
16	3. 514
18	3. 433
20	3. 389
α	2. 576

Note: The above table shows the K factors such that at least 99 percent of the population individual values will lie above $\bar{x}-Ks$ with 95 percent confidence, where $\bar{x}$ and s^2 are estimates of μ and σ^2 computed from a sample of size n reference E3 .

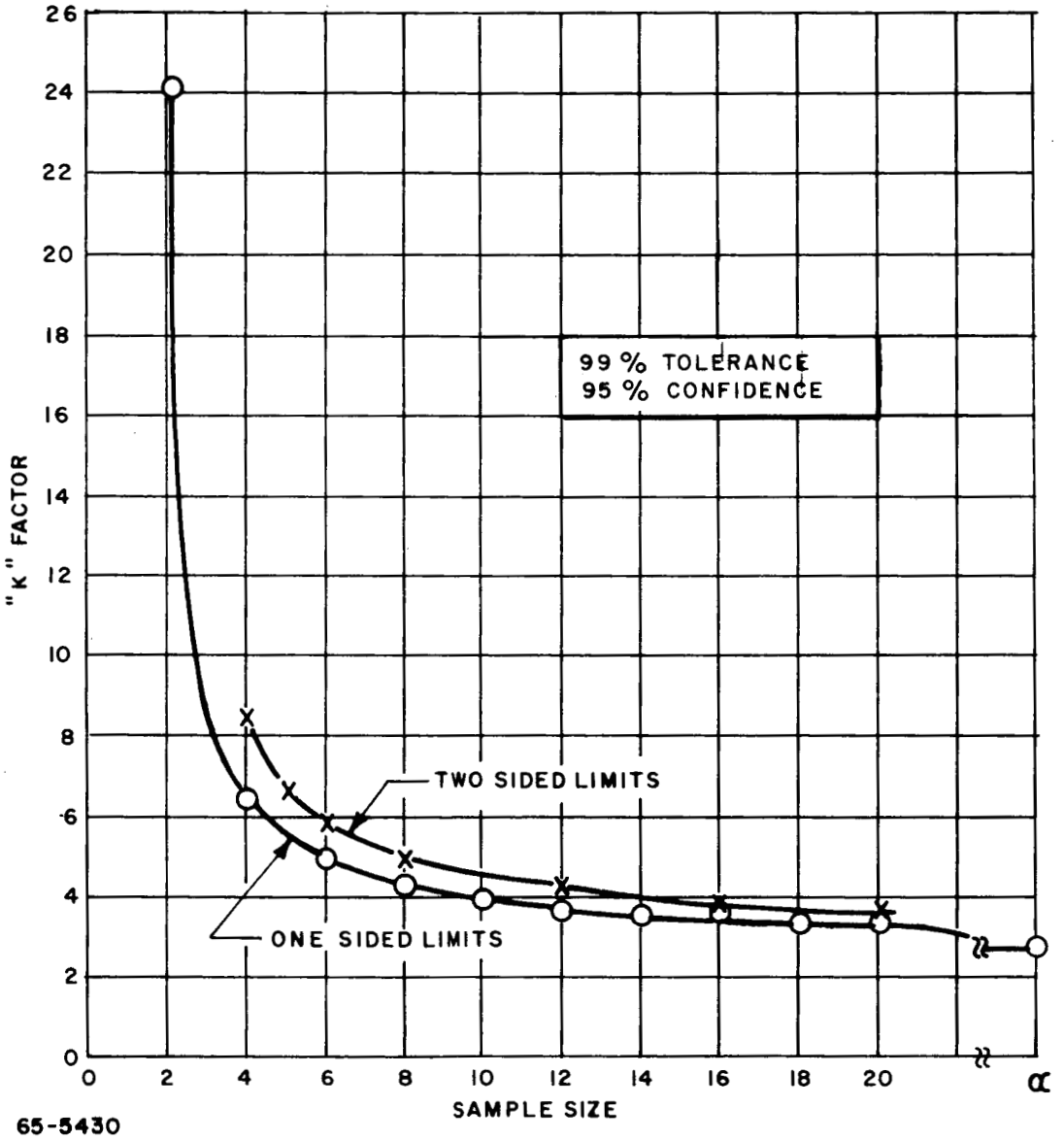

Figure A-I TOLERANCE FACTORS FOR A NORMAL DISTRIBUTION VERSUS SAMPLE SIZE