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Design And Development Of A Transient Thermal Conductivity Apparatus For Water And Other Fluids

William Thomas Trick

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DESIGN AND DEVELOPMENT OF A TRANSIENT THERMAL

CONDUCTIVITY APPARATUS FOR WATER AND OTHER FLUIDS

by

William Thomas Trick

Department of Mechanical Engineering

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ABSTRACT

The report hereto is concerned with the mathematical analysis and associated mathematical models, the experimental apparatus and the data reduction techniques used in applying the constant heat flux, finite source technique to thermal conductivity determinations of substances. It was shown to be a versatile experimental method.

Of the two mathematical models, namely the line source model and the finite source model, it was shown that the former was inappropriate except in the case of measurements with insulation materials. The latter model must be invoked for investigations of air and water.

Within the report is presented the detailed description of three experimental systems, used for insulation material, for air and water, and for water respectively. The latter system was designed to operate with light and heavy water from atmospheric pressure and 72 Deg. F to the respective critical points of the liquids.

A computer programme based on the finite source model was completed which enabled convenient and consistent data reduction.

Although the purpose of the study and research was

to investigate the source technique from a broad basis,
preliminary results were obtained for expanded asbestos
insulation, for air and for water, all under ambient
laboratory conditions. Comparison of the data obtained
in this work to accepted values from other sources, under
the same temperature pressure and densities showed + 4%
difference for the expanded asbestos insulation, + 6%
for the data of air, and between - 1% to + 4% difference
for the water data.

The method of data recording and reduction used in
this work has not been previously reported. Mechanical
details of the test apparatus, resulting from experimental
and analytical optimization, form a satisfactory basis for
extended researches with this technique.

(Approved by the Examining Committee. 17 April
    1972).

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List Of Equipment And Materials

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1	Potential Comparator	Guildline Instruments Ltd.	9800	25889
2	Four Terminal Variable Resist-or	"	9801	26124
3	Galvanometer	"	9461-A	26605
4	Galvanometer Amplifier	"	9460	180641
5	Precision Ice Bath	Rosemount Engineering	–	–
6	Thermocouple wire-Cu.-Const.	Thermoelectric Canada Ltd.	–	–
7	Potentiometer	Leeds & Northrup	K-3	153801
8	Galvanometer	Honeywell	3100	G-528
9	Regulated Power Supply	Harrison Lab.	865C	–
10	Timer	Precision Scientific	69235	–
11	Standard Shunt, 1 ohm	Julie Research Labs.	–	21
12	Pure Nickel Wire, .010" Diam.	Sigmund Cohn Corp.	–	–
13	Decade Resistor .1 to 1000 ohms	Honeywell	1013	1016
14	Decade Resistor 1 to 10,000 ohms	"	2281	H-2292
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16	Chart Recorder	"Electronik"	–	–
17	Inshop Power Resistor	U. W. O.	–	–
18	Inshop Power Resistor	U. W. O.	–	–
19	Resistance Wire (Karma)	Driver-Harris Corp.	–	–
20	Shielded Conductor	Beldon Canada	–	–
21	Copper Bar – 100% conductiv-ity, electrolytic, tough pitch	Drummond-McCall		
22	Electrolus Gold Plating solution		–	–
23	Electrical heating wire, fiberglass covered, 24 gauge	Driver-Harris Corp.	–	–

Item	Name	Manufacturer	Model	Serial No.
24	"Inconel" sheathed "Ceramo" heating cable			
25	Temperature Controller	Thermoelectric Canada	—	—
26	Microvolt Potentiometer	Honeywell	2779	P-3574
27	Conductivity Meter	Johns-Manville		
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29	Standard Resistor, 100 ohm	Guildline Instruments Ltd.	9221	26091
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31	Standard Cells And Temperature Controlled Enclosure	"	2778	P-1940
32	Standard Cell			
33	Standard Cell			
34	Mercury Wetted Relay			

NOMENCLATURE ASSIGNED

Symbol	Definition	Units	Page
A	Area	$Ft.^2$	.
C	Euler's Constant	—	
C_1	Specific Heat of Finite Source Heater	B.T.U./Pound Deg.F.	
C'	Specific Heat	B.T.U./Pound Deg.F.	
D	Density	$Pounds/Ft.^3$	
d	Thermal Diffusivity	$Ft.^2/Hr.$	
E(i)	Exponential Integral	—	
H	Surface Heat Transfer Coefficient	$B.T.U./Hr.Ft.^2Deg.F.$	
K	Thermal Conductivity	B.T.U./Hr.Ft.Deg.F.	
m	Mass/Unit Length Finite Source Heater	Pounds/Ft.	
Q	Thermal Energy Rate	B.T.U./Hr.	
q	Thermal Energy Source Strength	B.T.U./Hr.Ft.	
R	Radial Co-ordinate	Ft.	
R_0	Radius of Thermal Conductivity Cell	Ft.	
R_1	Radius of Finite Source Heater	Ft.	
T	Temperature	Deg.F.	
t	Time	Hrs.	
Z	Axial Co-ordinate	Ft.	

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Symbol	Definition	Units	Page
$\dfrac{dT}{dX}$	Temperature Gradient X Direction	Deg.F./Ft.	
$\dot{\theta}$	Angular Co-ordinate	Radians	

CHAPTER 1

INTRODUCTION

There is at the University of Western Ontario, Department of Mechanical Engineering, a small, but active group of personnel engaged in thermo-physical property investigations. Notable work has been carried out (Nowak 1, 2)* and is continuing at present on the experimental determination of heat transport properties of Deuterium Oxide**. Furthermore, viscosity measurements, heat transfer near the critical point and boiling heat transfer characteristics for water and D_2O, are being investigated at present.

As an adjunct to these researches, the writer, who joined this group in October 1966, embarked upon a programme of experimental thermal conductivity measurements.

The sources of motivation for the above researches were many, but one in particular was the economical design of the reactors of the Candu type which use D_2O for primary heat transport and for moderating. Since a significant part of the capital cost of these reactors was the D_2O inventory, accurate data for the heat transport and heat capacity of D_2O was of considerable financial consequence.

*Represents references listed at end of this report.

** Hereafter abbreviated D_2O.

Early literature surveys showed that such data was in many cases, not available. The property of thermal conductivity was of direct importance in the design of heat transport rates in the primary heat transfer system of the Candu reactor. A significant contribution to engineering and to Canada would result from better thermo-physical property data for D_2O, liquid phases.

At present thermal conductivity data for D_2O and in fact for most liquids, must be determined experimentally, if engineering accuracy is required. The complexity of the water molecule prevents prediction of this property from the basic data at the molecular level. The molecular forces are less complex for simple gases, on the other hand, and reasonable predictions can be made from the physical level. Undoubtably accurate experimental data for D_2O will be required, as was the case with gases, to assist those physical chemists and physicists working on this problem, (Horrocks et al., (64, 1963)

The thermal conductivity of a substance can be defined as the proportionality factor relating the heat flux and temperature gradient through a particular thickness of that substance. Mathematically, this can be expressed as,

$$dQ = - K A \frac{dT}{dx}$$

where dQ is the thermal energy transferred.

K   is the value of the thermal conductivity.

A   is the area perpendicular to the sense of the thermal gradient.

$\dfrac{dT}{dx}$ is the thermal gradient.

All the above must be expressed in consistent units.

This expression is the Fourier heat conduction equation, the basis of conductive heat transfer analysis.

For fluids, the heat energy transmitted by conduction implies an absence of bulk fluid movement, or alternately implies energy transfer at the molecular level. As will be discussed later, the absence of bulk fluid movement was extremely difficult to achieve experimentally, and has plagued almost without exception, every experimentalist who has attempted to measure the thermal conductivity of fluids.

Traditionally, time independent heat transfer models and experimental systems to simulate the mathematical model were used for the majority of thermal conductivity studies. All used the Fourier relation, selecting mathematically and physically resolvable geometries and boundary conditions. Sufficient variables were experimentally measured to permit the solution of the Fourier equation for the thermal conductivity. The literature well documents the commonly used geometries, namely the parallel plate, the concentric cylinder, and the concentric sphere systems.

During the initial stages of the writer's work, a rather comprehensive bibliography of the previously published work was made available to him. Dr. E. S. Nowak and previous graduate students (Yin, 3) had in the past reviewed the complete field of thermal conductivity research, both from the point of view of collecting together all available data for water, and also of experimental apparatus. Dr. E. S. Nowak had himself, an invaluable collection of European and Russian work, in many cases translated by himself or his associates. Some recent literature was incorporated by the writer, and altogether this formed an excellent and comprehensive literature survey of the field of thermal conductivity research for water.

As time passed, and the writer assimilated larger amounts of the background information, he experienced a growing disillusionment with the traditional steady-state methods. Successful apparati had required many years of painstaking development, requiring great expenditures of time and material resources. Ziebland et al., (9) showed the extensive discrepancies in the data of light water in the period 1900 - 1960. Extensive development obviously had been done, and further refinements to produce a system as good or better than had already been built appeared a most formidable task. It was felt by the writer that such a system was quite possibly beyond his time

resources and the engineering department's material resources.

It was at this point that the decision was made to look into some less complex technique overlooked by others, or develop a non steady-state method, the latter not having experienced the same degree of development as steady-state methods.

Following further study, it appeared that a steady-state system could not be simplified significantly, and that experimental difficulties were inevitable. As was previously stated, the steady-state systems simulated the Fourier Equation mathematical model, whether it be geometrically rectangular, cylindrical, or spherical. Using any of these geometries required the experimental determination of a minimum of four primary variables, usually two absolute temperature measurements, an energy input and at least one geometry constant. Additionally, the mathematical boundary conditions required the physical creation of isothermal surfaces, and adiabatic enclosures, these latter two being particularly demanding experimentally. In some cases, researchers had used a comparative system, whereby the geometry constants of the steady-state system had been determined by calibrating their system with a "reference" liquid. The writer and his advisor felt that an absolute measurement technique rather than a comparative one should be employed if at all possible.

When the impractability of utilizing a simplified
steady-state system became apparent, the writer reviewed
the documented work involving transient methods. The
published reports, while small in number compared to the
steady-state research reports, were nevertheless, encourag-
ing. The writer submitted progress reports (4, 5) to his
supervisor at this time, recommending that additional
emphasis be placed on the use of transient methods for
thermal conductivity measurements.

Three transient systems were investigated, the line
source technique being selected for further development.
This method, as did all transient methods, required the
accurate recording of the temperature-time history of a
test substance under an impressed, but known, heat source.
The response characteristics were consequently related to
the thermal properties of the test substance.

In general, the advantages of the transient methods
over the steady-state techniques were that measurement
times were shorter, reducing the difficulty in maintaining
adiabatic enclosures. Dimensional constants, particularly
for the line source method were not required to a high order
of accuracy. Finally, fewer primary variables required
monitoring than in a steady-state system. Balanced
against this was the added complexity of measuring trans-
ient voltages accurately and of additional data reduction
complexity.

In both the writer's and his advisors opinion, it

was strongly felt that some preliminary investigations
should be embarked upon, in order to point out physical
and technical problems which might not have been evident
in the technical reports and other background literature.
This course was pursued.

The first investigations were completed using a
very simple apparatus. Expanded asbestos insulation,
commonly known as "Vermiculite" was chosen as the test
medium. Results were encouraging.

Next, a series of tests were completed on water
and air using again a simplified apparatus. It was during
this series of preliminary tests that technical difficult-
ies first began to appear. Some development in the analysis
of the data was initiated. Again after sufficient refine-
ment of equipment and methods, encouraging results were
obtained.

At this stage, a decision was made by the writer and
his advisor that the design and development of an experim-
ental facility permitting the measurement of data of 1%
absolute error or less be undertaken. The writer was unable
to achieve this objective accuracy due to equipment problems
and limitations in readout equipment imposed by financial
restraints.

CHAPTER 2

SURVEY OF PREVIOUS WORK

An engineer or scientist involved in the area of thermal conductivity of substances, whether he be an experimentalist or concerned with screening of the available data, must be initially astounded and perhaps later overwhelmed by the abundance of documented investigations and reports of studies which have been completed in the past. Laubitz of the National Research Council reported in Tye (6) that eight hundred references existed, all of which were concerned with the thermal conductivities of solids, whereas the reported investigations concerning the thermal conductivity of water, liquid and gaseous phases, exceeds 300. It of course would be presumptuous for the writer to state that he has surveyed the field completely. Rather, approximately two hundred reports have been perused, some in detail, the most cursorily.

As was pointed out in Chapter 1, a platform for a background study had been well prepared by the writer's project supervisor, and previous graduate students. The international reports, particularly those describing the Russian work, and their invaluable translations, were of particular worth. This literature review represented a rather comprehensive review of the data and experimental

technique for water.

Additions were made by the writer of the most recent work and an exhaustive search for transient heat conduction reports was completed. Partly through the project, it became obvious that natural convection was to play a part in the experimental work, and the published work on natural convection phenomena for fine vertical wires was surveyed. Particular areas of instrumentation as were required for the experimental aspects of this work were reviewed in the appropriate journals.

2.1 Selected Steady-State Experimental Techniques For The Measurement Of The Thermal Conductivity Of Light And Heavy Water

Using as an approximate guide the number of publications relating to each technique as indicative of the amount of effort devoted to that technique, the following breakdown results.

Sixty percent of the investigations were completed using a cylindrical co-ordinate system with either two concentric cylinders of moderate size (3/4" to 3" diameter) as heat receiver and emitter, or the hot wire type format, wherein the heat emitter consisted of a fine wire, usually platinum, surrounded by a precision bore glass or quartz tube.

Twenty percent of the documented investigations were completed using the parallel or flat plate system, subjecting a thin lamina of liquid to a heat flux applied in a vertical sense.

Five percent of the remaining investigations utilized a concentric spherical geometry, the inner element forming the heat emitter, the outer, the receiver.

The remaining 15% of the investigations were based on transient heat conduction models, such as the line source method, or comparative techniques.

2.1.1 Coaxial Cylinder Systems

Bridgman (7) has been credited with the development of the first coaxial-cylinder apparatus, his work being documented in 1923. The work range extended over high pressures, up to 3,000 atmospheres, with a temperature excursion of ambient to 75°C.

The German investigations by Schmidt & Sellschopp (8) 1932, produced at that time the only data for water over 80°C. Their system utilized an inner cylinder of 1.18 inch diameter, an annular gap of .0196, with an overall gap height of 4.72 inches. The measurement range extended from 0°C to 270°C, under pressures up to 90 atmospheres. The estimated error was $1\frac{1}{2}\%$, but their data served as a basis for comparison for other investigators for 30 years.

A recent coaxial cylinder system was that of Ziebland & Burton (9) reported in 1960. Heavy water was investigated over a range from 75°C to 260°C at up to 300 atmospheres pressure. Their work represented at the time of publication, the only reported data for heavy water over 82°C. Although the system was designed to operate up to 400°C, an unfortunate problem developed, limiting the useful results to 260°C.

The mechanical arrangement consisted of a copper cylindrical emitter, 1.3016 inches diameter, surrounded by an outer copper receiver; 1.3147 inches inside diameter. It

was not stated by the authors if any electroplating of the surfaces had been carried out. The design was unique in that the emitter heater could be removed, an advantage which permitted the insertion of a standard grade platinum thermometer into the heater cavity, thereby permitting calibration and recheck of the thermocouples used for primary temperature difference measurement. Guard heaters controlled end-heat loss from the emitter. The temperature match between emitter and guard heaters was monitored by differential thermocouples and was manually controlled. The entire system was encased in a steel pressure vessel, further housed in a cylindrical steel thermostat, whose temperature was controlled automatically. Variations of the thermostat temperature were less than $.01^{\circ}C$ over a two hour period.

Their investigations with this apparatus was terminated by the unfortunate occurrence of a cell leak, when operating close to their design limits. Upon rechecking the emitter dimensions they found it had increased in diameter, a defect attributed to lack of annealing after final machining. Accordingly they rejected all data above $260^{\circ}C$ excepting one final data point recorded immediately before disassembling.

The authors did not report an overall accuracy estimate, but detailed several areas of possible error. They felt that mismatch in the end guard heaters due to thermocouple error could result in an error up to .4%. Heat transfer by radiation was considered negligibly small. Natural

convection, and axial heat loss effects were considered to introduce less than 4% error, after due consideration by the authors.

Venart (10), while at Glasgow, reported in 1964 on the development and testing of a concentric cell system, in which he performed measurements on a series of gases and liquids including water over the temperature range $15\,^{\circ}C$ to $75\,^{\circ}C$. An error of 2% was estimated, and supported by reference to data of other investigations. The outside diameter of the brass emitter was .786 inches, the inside diameter of the glass receiver, .823 inches, leaving a nominal gap of .018 inches.

The preceding comments briefly describe representative investigations wherein the coaxial cylinder geometry has been utilized. Those selected have been used for investigations with water. Similar geometry has been extensively used for investigations with other liquids, gases, and vapors. Regarding the latter, it would be improper not to mention the work of Mason (11) 1953, whose subject liquids were a large range of industrial non-metallic chemicals. Guildner's work (12, 13) reported in 1962 extensive investigations for carbon dioxide, making careful note of the possible sources of error when using this technique.

2.1.2 Hot Wire Systems

The hot wire format can be considered a modification of the coaxial cylinder arrangement in which the inner cylinder or heat emitter has been replaced by a fine single wire, usually of platinum. In this arrangement, the heater wire served both as heat emitter and electrical resistance thermometer.

It was early realized that axial heat loss was a serious problem affecting the accuracy of the method. Consequently three variations were devised each with the object of solving the problem.

An early method was the compensated hot wire method wherein two cells differing in length, but otherwise similar, were used in the opposing arms of a bridge network. The electrical output across the bridge can be made to reflect only the differences in resistance of the two cells. Ideally, the effects of end losses in one cell could be equally compensated by end losses of the companion cell.

A second method was known as the potential lead technique. Potential leads were joined to the heater wire, partway in from the ends of the cell. The section between the potential leads became the active section, and was relatively unaffected by end loss effects. This has been the variation used by the majority of recent investigators.

The third variation consisted of designing into the cell, end conditions for the hot wire attachments which were explicitly known. It was then possible to calculate the axial heat loss by the assumption of boundary conditions at the hot wire terminations which were consistent with the actual cell. This arrangement was known as the thick wire cell.

In all of these investigations, the heater wire served as both the heat emitter and as a resistance thermometer.

It would be wise to note the comments recorded by Taylor & Johnston (14), 1946, who, before selecting a potential lead variation, made a careful study of the axial heat flow problem. In the compensated type of hot wire cell, from 5% to 10% of the total heat input to the heater wire required compensation by the second cell. It necessarily implied a geometrical, mechanical and thermodynamic similarity between the cells if compensation was to be achieved. The thick wire cell required absolute calculation of up to 50% of the total heat input, this quantity flowing to the end connections. The potential lead cell required the correction for perhaps 1/2% of the total energy input to the heater wire; this small amount being axial heat loss. In conclusion, the authors felt that the possibility of systematic error was much reduced with the potential lead arrangement.

An early investigation of the thermal conductivity of air and other gases was conducted by Gregory & Archer (15)

in 1926, using a compensated hot-wire cell. Two sizes of
receiver tubes were employed, to discern convective effects
on the experimental data. Platinum heater wire was used,
.005 inch diameter. The system was encased in a liquid
thermostat for temperature stability. While all work was
completed near 0°C, the authors estimated their possible
error to be not more than .33%.

Milverton (16) further investigated air in 1934,
covering the range from 0°C to 100°C, under low pressures.
He, too, used a compensated type hot wire cell, the critical
dimensions being the diameter of wire which was .004 inch,
diameter of receiver tube, .016 inch.

The Russian investigators have for many years used
a hot wire system in their investigations with water and
steam. One of the earliest reports was that of Vargaftik (17)
who reported in 1937 the results of their tests with steam,
other vapors and gases. This equipment as described in 1937
was capable of pressures up to 40 atmospheres, and temperat-
ures to 475°C, the results obtained in this region being
unique at the time.

For the early work, a compensated type arrangement
was used. Platinum wire, .004 inch diameter was used as
the heat emitter, thin walled quartz tubing, .032 inch
inside diameter served as the receiver. In order to determine
convective effects, Vargaftik duplicated each data point
using two different heat rates. In the 1937 work, reasonably
satisfactory agreement for nitrogen data was achieved, when

compared to previous data, but a systematic discrepancy in his results encouraged Vargaftik to introduce a 1.3% system correction, this hereafter being applied to steam measurements.

Taylor & Johnston (14) reported in 1946 their results for air obtained with a potential lead hot wire apparatus. Special features included the use of a steel cylindrical receiver, and the application of a small weight on the end of the platinum heater wire, to maintain a uniform tension in the wire under conditions of thermal expansion. The authors predicted an absolute error of not more than .5% from 80° to 380° Kelvin.

The recent Russian investigations for water, Vargaftik et al., (18) 1960, have been completed using a potential lead type of cell.

A quartz tube, approximately .040 inch inside diameter enclosed a .004 inch diameter platinum wire. The receiver temperature was determined by constructing a platinum resistance thermometer on the outside of the receiver. By connecting the end of the heater wire to the end supports with a spring, a tension was placed in the wire at all times. It was interesting to note that the authors found this constant tension affected the temperature coefficient of the platinum, when compared to an unstressed wire under the same temperature conditions. This small but consistent change in the temperature coefficient of the platinum heater wire necessitated the in situ calibration, it being intercompared to the

receiver platinum resistance thermometer.

As a conclusion to this section, the listing of the variables measured by Vargaftik necessary to permit the absolute determination of the thermal conductivity will be made. These parameters were typical of all similar systems. Of primary importance, was the absolute temperature difference between the heater wire and the inside surface of the receiver cylinder. A geometry constant was required, this being a function of the ratio of the diameter of the hot wire and the inside diameter of the receiver tube, as well as the overall effective length of the heater. Accurate measurements of electrical power input to the heater wire were necessary. Of importance as secondary parameters was the determination of axial heat loss along the heater wire, and radial heat loss out the potential leads. The effects of thermal resistance through the receiver tube wall were determined. Finally, heat transmitted by radiation and by natural convection had to be distinguished from conductive heat transfer.

The preceding reports were concerned with the hot wire technique, and while representative of this method, cannot be considered comprehensive.

2.1.3 Parallel Plate Systems

In general, the parallel plate system consisted
of two flat plates, usually circular in cross-section,
positioned horizontally and enclosing between them the
thin lamina of liquid or gas under test. On first contem-
plation, the system appeared to offer considerable advant-
age, in that the upper plate can be made the isothermal heat
source, the lower, the isothermal sink. For this format,
the convective effects in theory were nil. However, upon
constructing a prototype, it became obvious that most
experimentalists experienced recurring problems. Isotherm-
ality of the parallel surfaces was achieved with great
difficulty. Unaccountable heat loss from the adiabatic side
of the heater plate plagued investigators. Finally, deform-
ation of the surfaces under pressure and high temperatures
affected the system geometry constant. In spite of these
problems, a considerable number of investigations, including
some recent studies have been completed using this arrange-
ment. Its inherent advantage over cylindrical systems
regarding natural convective effects, made the method applic-
able for critical region studies, in this region convective
forces being very large.

In 1918, Hercus & Laby (19) used a parallel plate
system for air, at ambient pressures and temperatures. At

the end of their paper, the authors showed the then
available data for air, illustrating that the average
departure from the mean of the 14 available investigations
was 7%.

A second investigation was reported by Hercus &
Sutherland (20) in 1934. From 1918 to 1934, additional
data for air was measured by others using the hot-wire
method (see Gregory & Archer), (15). At $20^{\circ}C$ excellent
agreement between the data was determined from the flat
plate system and the hot-wire technique. A comment by
Hercus & Sutherland was most interesting. The authors
considered their parallel plate system had verified the
accuracy of the hot-wire method, and they consequently
were prepared to do further work using the hot-wire system
rather than the parallel plate arrangement. This might
have indicated the relative experimental and mechanical
difficulties of the two techniques.

The parallel plate method was used by Bates (21, 22)
(1933 and 1936 respectively) for experimental investigat-
ions with liquids. In his first system he utilized a thick
film arrangement, the fluid layer being approximately 1.93
inches thick. His results for water were considerably
higher than other data available at the time. In the
1936 paper he reported redesigning his cell, in order to
enable varying the fluid layer thickness, from .125 to
4 inches. Uneven temperature distribution in his first
arrangement (21) had affected the accuracy. Results from

the second system were consistent with previous data.

Additional results with liquids were reported by Challoner & Powell (23) in 1956. Using a copper parallel plate apparatus, with a liquid thickness of .080 inch, the authors completed experiments with D_2O over the temperature range from 2°C to 82°C. Suggested absolute error was 1%.

The extensive investigations on the critical region properties of carbon dioxide, as reported by Michels et al., (24, 25, 26) 1962, illustrated the elaborate experimental equipment required for even moderate pressure studies. The problem of natural convection heat transfer was carefully investigated, and as a result led to the utilization of the parallel plate apparatus, although demanding high mechanical sophistication.

Fritz & Poltz (27) using a parallel plate system with variable liquid thickness, carried out studies with water and methanol at atmospheric conditions. They found that for particular film thicknesses and temperature differences, definite indications of convective heat transfer were evident.

The preceding investigations cited have described the absolute measurement of thermal conductivity. The parallel plate method has been extensively used for comparative measurements wherein a particular fluid under investigation has been paired with a reference fluid in a two layer arrangement. This may simplify to some extent the heat flow accounting. It implied, however, that a

reference fluid introduced inherent uncertainty regarding its own properties. Bonilla & Wang (28) completed in 1953 some of the early investigations for D_2O, using a flat plate comparator system, the reference fluid being water in this case.

2.1.4 Other Systems

A spherical geometry has been used in a thermal
conductivity measurement system built by Schrock &
Starkman (29) in 1958. Two concentric copper spheres
of unspecified diameter were assembled (being machined
separately as hemispheres and bolted together), the inner
sphere serving as the heat emitter, the outer as receiver.
The liquid film thickness between the spheres was approx-
imately .100 inch. When using this system for measure-
ments on viscous fluids, the authors predicted a system
error of approximately 2%.

The elaborate multi-purpose cell of Leidenfrost (30)
who reported in 1963, consisted of a vertical cylinder with
hemispherical ends as the heat emitter, surrounded by a
geometrically similar cavity in a pressure vessel. The
technique used to determine the geometry cell constant
consisted of exploiting an analogy between electrical
capacity and the cell constant, the former was accurately
determined under vacuum conditions within the gap. Prelim-
inary thermal conductivity results were obtained for nitrogen
gas, with investigations of water vapour, nitrogen and
other fluids and gases being planned. The author estimated
the possible absolute error to be .1%.

2.2 Transient Heat Conduction Experimental Techniques For The Measurement Of The Thermal Conductivity Of Liquids And Solids

Various transient heat transfer techniques have been evolved for the determination of the thermal conductivity of substances, and a few have been applied to liquids. Their utilization has been encouraged to some extent by the recurring and difficult problems encountered in the steady-state systems. The maintaining of adiabatic conditions, the production of heaters providing isothermal surfaces, the machining and measuring of critical mechanical surfaces and the maintaining of the system geometry constants under extremes of pressure and temperature have been a few of the details requiring the meticulous attention of the steady-state experimentalist. The transient systems, depending on the particular one selected, did not involve these problems to the same degree.

There are a considerable number of experimental transient systems possible which utilize transient heat transfer phenomena. The majority permit the direct determination of the thermal diffusivity of the test medium.

Only one, the line source constant heat flux method

permits a direct determination of the thermal conductivity. Since it has been selected as the topic of investigation by the writer, a separate literature section, Chapter 3, has been devoted to this technique.

The reader is referred to Tye (6), Volume 2, Chapter 3, for an extensive review of the investigations of thermal conductivity of solids by non-steady state methods.

A recently developed technique for the absolute measurement of the thermal conductivity of liquids is the transient hot-wire frequency response method. First reported at Columbia University in 1952, subsequent refinement of the technique resulted in a system which provided data with less than 3% probable error. A recent publication, Navarro et al., (31) pointed out some of the advantages of the method compared to alternate hot-wire techniques, these being simple cell design and reduced temperature stability requirements.

In general, the advantages of the transient techniques compared to the steady-state methods are these:

1. A particular test can be completed in a few seconds rather than hours as are required for steady-state systems.

2. Cell complexity is reduced due to the absence of guard heaters.

3. Reduction in the number of primary measurement variables, notably, the cell geometry constants in particular are not required as a primary variable.

4. The line source technique need utilize a much smaller sample of test medium than other available methods.

2.3 Natural Convective Heat Transfer From Small Cylinders

It would not be an exaggeration to state that the single common problem which has plagued most experimentalists in thermal conductivity studies of gases and liquids has been natural convective heat transfer during testing. All of the presently available measurement techniques are susceptible to a greater or lesser degree to heat transfer by convection. Almost without exception the various investigators have had to design their equipment and methods around constraints imposed by the necessity of reducing or rendering insignificant spurious heat transfer by natural convection.

The writer was early made aware of this problem and consequently completed a study of the past investigations of heat transfer to fine wires or small cylinders, which would be relevant to thermal conductivity studies.

For the purpose of this report, the previous reported investigations have been somewhat arbitrarily divided into four areas.

The first area includes the reported investigations which provided design data for electrical light filaments. This group includes the oldest research, and was in general, applied research rendering empirical relations between heat transfer and filament temperature. General conclusions were

relevant to the thermal conductivity problem.

A second area of investigation was, again, applied studies which lead to the successful application of the hot-wire anemometry technique for air velocity measurement. Those investigations dealing with low velocity applications were relevant to the thermal conductivity problem.

The third area included the reports of the phenomena of thermal overshoot, as observed by Bosworth (35) 1946 and later experimentalists. The results and conclusions were particularly relevant to transient thermal conductivity methods.

Finally, the fourth and largest group encompassed the results of a number of investigators, some experimentalists, others analysts, who have studied the natural convection problem from a broad general standpoint. Their intention of mathematically describing the convective heat flow has been achieved under particular conditions.

Within the first group above, Langmuir (32) 1912, undertook experimental and analytical investigations of the heat flow characteristics of electrical light filaments. In the initial section of his report, Langmuir summarized the conclusions of the few previous investigations conducted up to that time on the subject of convective heat transfer. The previous researches had been predominantly empirical, the earlier workers having been obviously overwhelmed by the complexity of suitable analytical relat-

ions between heat transfer and variable densities, viscosities, fluid thermo and fluid property inter- actions. An earlier assumption, that of negligible viscosity of air with respect to the convective heat transfer, was challenged by Langmuir. He realized that. in the vicinity of the hot filament, the viscosity was of extreme importance. Langmuir then proposed a heat transfer model which included a stagnant film of gas surrounding the hot-wire wherein conduction alone applied. The thickness of the film was dependent upon the wire diameter and ambient gas temperature, but relatively in- dependent of the wire temperature. He further concluded that radiation heat transfer was small compared to con- duction heat transfer for wire temperatures less than 200 $^\circ$C above ambient gas temperature. The viscosity of gases increases with temperature and in the immediate vicinity of the hot wire, the viscosity was so large that convective motion would be impeded, making conduction the predominant heat transfer mechanism. He was able to support his conclusions experimentally.

Elenbaas (33) 1948, presented experimental heat transfer formulae of the Grashof-Prandtl-Nusselt type which were applicable to the high temperatures experienced in electric light filaments. He furthermore conducted Schlieren tests, making visible the isotherms surrounding the wire, their arrangement supporting the conduction model of Langmuir's. While he completed most of his experimental

investigations on horizontal wires, he measured differences of approximately 15% in heat transfer coefficient between horizontal and vertical arrangements of the same specimen, the vertical arrangement having the lower coefficient.

The work of Langmuir & Elenbaas was conducted with gases only, and consequently the Prandtl number effect was not sufficiently studied to permit the application of the data to liquids.

Within the second area, that of hot-wire anemometry, was the work of Collis & Williams (34) 1959, and their studies of heat transfer to fine wires at very low velocities. They determined the critical Reynolds number at which natural convective heat transfer became significant, and developed a method of determining the minimum velocity that could be measured using a hot-wire anemometer.

The third area of investigation concerning natural convection of small wires was the so called "Thermal Inductance" phenomenon reported by Bosworth (35) 1946. When a fine wire, immersed in a fluid was subjected to a constant electrical heat input, Bosworth observed the characteristic logarithm of time temperature rise of the wire. The temperature reached a peak, then fell to a lower steady-state value. Tha analogy between the electrical and thermal system was exploited, the temperature overshot being analogous to the voltage transient observed in an electrical inductive network. Bosworth pointed out that

this finding was contrary to accepted views that a thermal system possessed only resistive and capacitive characteristics. Initially, he explained this inductive-like characteristic by relating it to the kinetic energy stored in the natural convective flow field surrounding the wire.

Bosworth (36) in 1948 reported again on the same phenomenon, but expanded it to include "mutual inductance", also modifying his experimental technique to permit direct wire monitoring by resistance measurements of the heating wire. In his first researches, he had attached a fine thermojunction to the heater wire, possibly introducing spurious effects.

Bosworth et al., (37) 1959, and Bosworth (38) 1959, presented the equivalent electrical network which simulated the thermal system. They reached a surprising conclusion that the deduced inductive energy magnitude exceeded by 10^2 times the conceivable kinetic energy associated with the convective current flow field. He concluded at this time that some form of energy storage such as surface evaporation or chemical phase change must have been present to explain the inductive energy storage of such magnitude. The conclusions and data of Bosworth do not have universal acceptance. It appeared Bosworth attempted to draw an analogy with an electrical circuit which in fact did not necessarily have a relationship to the thermal system. It is intuitively obvious that some degree of overshoot in a thermal system

could be experienced due to a time lag in building up a
convective current flow field. The actual mathematical
model which closely describes the thermal response of the
system has been presented by Goldstein & Briggs (48) and
is considerably more complex in format than Bosworth's
analogous electrical model. Bosworth reported no serious
study of the extensive fluid and thermodynamic interactions
which occur in the onset of a fluid convective field, and
he could by no means compare this case to the relative
simplicity of a magnetic field within an electrical inductor.
The comparison of one physical system to another by analogy
necessarily implies a simularity in the respective mathemat-
ical models. An oversimplified model for one physical system
can lead the investigator into completely erroneous conclus-
ions, which appeared to be the case with Bosworth, in partic-
ular with respect to his prediction of the kinetic energy
stored within the convective field.

Ostromov (39) 1957, reported on his detailed investig-
ations involving natural convective studies for horizontal
wires immersed in alcohol, water and transformer oil. A

method of flow visualization employing fine aluminum
powder in suspension in the test medium in conjunction
with time-lapse photography permitted him to determine the
velocity and direction of the flow currents about the wire.
Simultaneously he monitored the temperature of the heater
wire using wire resistivity measurements. Overshoot was
observed for water and alcohol for all but the lowest heat
flux magnitudes. The visualization technique illustrated
the formation of a "characteristic cap" above the wire,
the size being a function of time from initiation of the
heating current. The velocity of ascent of the cap was
shown to be directly related to the heating power. Ostromov
was successful in describing the size and velocity of the
"cap" by mathematical relations, inputing fluid property
data, and additional hydrodynamic and dynamic criterion.

In 1966 Dring & Gebhart (40) reported on their invest-
igations of transient natural convection for thin vertical
cylinders immersed in silicon fluids and air. The test
results showed much less overshoot than those of Bosworth.
Dring & Gebhart felt that the temperature measurement
technique of Bosworth was in question, suspecting that a
thermocouple on the heated wire severely distorted the
flow field introducing serious temperature measurement
errors. It should be noted that Bosworth used horizontal
wires, Dring & Gebhart, vertical.

To correlate their experimental data, they compared
the experimental data of wire temperature to the conduction

solution of Carslaw & Jaeger (41) for a finite source
in a semi-infinite medium. Good agreement was observed
up to the time when fluid motion became important. The same
analysis applied to experimental data for air could not
be solved except for longer times, whereupon the Carslaw
& Jaeger solution converged, and satisfactory agreement
was obtained.

At the time of writing of this report, the observ-
ations of Bosworth remain in question. The phenomenon of
thermal overshoot has been observed by independent invest-
igators, but of less magnitude. The present explanation
is that the overshoot of temperature is due to the kinetic
energy stored in the convective current flow field.

The fourth group of investigations of heat trans-
mission by convection relevant to the thermal conductivity
problem comprised the work of a series of investigators
whose object was solely the understanding of natural con-
vection heat transfer. Experimental and analytical studies
were included.

A majority of the reports of thermal conductivity
researches using the hot-wire method have referred to the
early work of Beckmann (42) and Kraussold (43), 1931 and
1934 respectively. These German investigators studied the
steady-state heat transfer from small cylinders under a
wide range of heat fluxes and with different fluids.

Beckmann utilized a cylindrical emitter, .320 inch
diameter, horizontally positioned inside receivers of .380

inch to 2.592 inch inside diameter. Thermocouples and appropriate readout ancillaries permitted the observing of temperatures of the emitter and receiver, and by calculation, the temperature difference. Beckmann reduced his data by plotting to logarithmic co-ordinates the Grashof number on one axis, the thermal conductivity ratio on the other.

The thermal conductivity ratio as developed by him consisted of the ratio of the effective thermal conductivity (calculated by using the conduction solution between concentric cylinders) and the fluid thermal conductivity. For the case of significant heat transfer by natural convection, the ratio would exceed unity, but would tend to unity for no natural convective effects.

Beckmann's results indicated that the effects of fluid characteristics were not correctly correlated since he obtained different curves for air, carbon dioxide and hydrogen when the thermal conductivity ratio was plotted against Grashof number.

His experimental range covered Grashof number from 800 to 80,000,000 over which he observed a variation in thermal conductivity ratio of unity to 16. An interesting observation was the effect of the receiver diameter on the thermal conductivity ratio. As the ratio of diameter of receiver/diameter of emitter increased, progessively lower values of Grashof number were required for the thermal conductivity ratio to approach unity.

It should be noted that the test fluids were exclusively gases in Beckmann's work.

Kraussold (43) 1934, completing further investigation with apparatus which appeared to be similar to that of Beckmann, reported on his data for several liquids including water and oil. He, however, observed that a correlation of the Grashof-Prandtl product rather than the Grashof number alone as in Beckmann's work, better accounted for the fluid characteristics.

Kraussold further recalculated Beckmann's work with gases and found that a unique relation existed for both experimental data, remarkable in that the former work involved gases, while Kraussold's work utilized liquids, involving a five fold range in Prandtl number.

In Kraussold's work, the dimensional parameter in the redefined Grashof number consisted of the annular gap thickness while Beckmann had used the inner cylinder diameter. Kraussold's experimental data at low Grashof numbers was quite limited, the lowest Grashof number being approximately 50, this test data being for machine oil. For his tests of water, the lowest Grashof number for which data was presented showed a value of 57,000. In the case of the machine oil, the thermal conductivity ratio was 1.009, while for the latter, the ratio was 4.50.

Kraussold deduced two empirical relationships which expressed the thermal conductivity ratio as a function of Grashof and Prandtl numbers, the first valid for the value

of Grashof-Prandt product between 6,300 and 1,000,000 and the second valid for Grashof-Prandt product above 1,000,000.

While the data of Kraussold and Beckmann has been extensively applied to thermal conductivity researches for establishment of natural convective criterion, this direct application should be questioned on several points.

The German investigations utilized a horizontal arrangement of cylindrical heater. Almost without exception, hot-wire thermal conductivity investigations have been completed with vertical heater wires. While Elenbaas (33) showed that the vertical or horizontal arrangement exhibited small differences (less than 15%) in the heat transfer for hot filaments, it could not be concluded that this would be true for the low heat fluxes experienced in thermal conductivity experiments.

The criterion used by experimentalists when applying the results of Kraussold to thermal conductivity cells (see Van der Held (44, 45)) involved the selection, somewhat arbitrarily, of a value of Grashof-Prandtl product equal to 2,000 as the point at which conduction heat transfer alone occurred. If the thermal conductivity cell was operated with the Grashof-Prandtl product at a value of 2,000 or less, it has been assumed by the experimentalist that no correction for convection need be applied.

The experimental data of Kraussold does not support this. Such a value was definitely below any of his measurement points, and the lower region represented a definite

extrapolation below his test range. Furthermore, Kraussold himself made no empirical prediction below a value of Grashof-Prandtl product of 6,300.

While the selection of the annular gap thickness as the dimensional parameter in the Grashof number was reasonable and justifiable for Kraussold's work for horizontal concentric cylinders, the same dimensional parameter could not be physically rationalized for the vertical arrangement. Schlichting (46) in the chapter "Thermal Boundary Layers In Laminar Flow" presented an abundance of data and analysis for the vertical plate heat transfer. For the Grashof number to have physical and perhaps any significance, the dimensional parameter must be measured in a plane parallel to the line of action of the body forces. Schlichting's compilation of data support the physical significance of the Grashof number without exception. Application of Kraussold's data to the vertical cylinder case could only be contrary to conclusions reached for the vertical plate problem.

Nevertheless, criterion reached for vertical cylindrical systems for natural convection, based on Kraussold's data has experimental support. Van der Held (44, 45) obtained predictions of the onset of convection using the Kraussold experimental data and verified this prediction through testing with his vertical cylinder thermal conductivity probe. Many other experimentalists used the same approach to establish design conditions for not-wire cells or concent-

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ric cylinder thermal conductivity apparati. That natural convection criterion established with this approach should produce useful knowledge, appeared to the writer to be most fortunate and probably coincidental.

Successful solution of the boundary layer and hydro-dynamic equations for the cylindrical case have been recently developed, under particular boundary conditions. The application of this analysis to the fine vertical wire geometry should enable the establishment of more definite design and operating criterion for thermal conductivity measurement systems.

Sparrow & Gregg (47), using a boundary layer analysis for laminar flow showed a derived functional relationship between the Nussult number and the Grashof number, which would be valid for Prandtl numbers of .73 and 1. While their analysis was developed for vertical plate geometry, it was possible to utilize this approach for vertical cylinders with sufficient accuracy provided particular limits were not exceeded. A qualitative criterion to establish these limits was presented, but the authors pointed out that existing systematic experiments on the laminar free convection about cylinders was quite limited and recommended additional test programmes. Their solutions were expressed in terms of infinite series which required computer solution.

A more recent publication by Goldstein & Briggs (48), 1964, presented these authors' work on transient natural

convection about vertical plates and cylinders. Unlike
the previous report (47) the vertical cylinder solution was
developed exactly without recourse to vertical plate geom-
etry. Necessarily this analysis was of more relevance to the
problem of convection about the fine wires used in thermal
conductivity studies since the departure from the cylindrical
and vertical plate geometry becomes larger with smaller
cylinders.

Goldstein & Briggs proposed that in the vertical
cylinder case, the onset of natural convective effects
initiated at the lower end of the heater wire, and progressed
up the length of the wire. The interface between the con-
vective front and the unaffected fluid above the front was
defined by the authors as the leading edge of the natural
convective stream. Their analysis permitted the calculation
of the distance with time relation of the leading edge as a
function of Prandtl number of the fluid, heat flux in the
heater wire, and thermal capacity of the heater material.
The application to thermal conductivity measurements would
involve the determination of the time at which the leading
edge of the convective field reached the lower potential tap
of the heater wire. If all testing were completed in a
shorter time than predicted above, it would be reasonable
to conclude that only conduction heat transfer existed in
the length of heater wire between the potential taps. As in
the case of Sparrow & Gregg (47) the above solutions were
also presented in integral format, amenable only to computer

calculation. When the present writer compared a sample calculation supplied by Goldstein & Briggs to a calculation performed by the writer, using the Kraussold criterion inserting in the latter the same data, the prediction of time by the Goldstein & Briggs analysis was approximately 25% of the time using the Kraussold criterion. The writer did not invoke the Goldstein & Briggs analysis in the final design of the apparatus described in the present report, since he predicted a lengthy computer analysis study would be required before determining design data.

The writer was able to show the effects of natural convective effects in his testing by a more direct experimental method, these results being subsequently presented in this report.

As a summary for this section, the following points may be presented,

A. There is little recent experimental work on natural convection heat transfer to vertical cylinders under the range of heat fluxes experienced in thermal conductivity studies, under both steady-state and transient conditions.

B. It is the writer's opinion that the work of Kraussold & Beckmann should be applied to these studies with great caution, since their measurement range was considerably above the heat fluxes experienced in thermal conductivity studies, and were studies with horizontal cylinders only.

C. The writer presents a hypothesis that there exists two modes of convective flow about a long vertical heater

wire. The first mode may be a smooth streamlike, creeping flow along the wire in which case the effective heat transfer mode is predominantly conductive, followed at later times and/or at higher heat fluxes by a gross fluid movement in a less organized flow fluid, in which case gross fluid movement transfers heat by mass transfer as well as conduction. This is not to be confused with the fully turbulent type of convective flow wherein heat transfer is predominantly by mass transfer. An inspection of the plates presented by Ostromov (39) tends to support this hypothesis.

Thermal conductivity measurements must be made while the vertical hot-wire systems experience the streamlike flow **mode.** Even under this condition it is probable that only an apparent thermal conductivity value is obtained since there would be some net heat transfer through mass transfer of the streamlines. The only basis available at present for discerning the difference between the apparent thermal conductivity as measured by hot-wire systems and the absolute thermal conductivity would be to carefully intercompared data obtained with a horizontal flat plate apparatus, a steady-state hot-wire system, and a transient finite source method.

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CHAPTER 3

REVIEW OF THE SOURCE METHOD FOR DETERMINATION
OF THE THERMAL CONDUCTIVITIES OF SUBSTANCES

Source methods of thermal conductivity measurement
are characterized by a long cylindrical heat emitter,
immersed in a test medium, the extent of the medium being
infinite in a radial direction with respect to heat transfer
of the emitter. The heat emitter is then subjected to a
constant internal heat generation, by electrical energy input.
The subsequent transient temperature response of the emitter
can be related to the thermal conductivity of the test medium.

While the temperature response of the emitter is
largely related to the thermal conductivity of the test medium,
for a given magnitude of internal heat generation, there are
secondary variables which enter into the response character-
istics. These variables include the diameter of the emitter,
and thermal properties of the emitter and test medium other
than the thermal conductivity.

In general, the rise of temperature with time approximates
a logarithmic type function.

There exists two mathematical models which describe the
temperature response. The first, the line source model implies
that heat generation occurs in a line emitter. The response
of a physical system will depart from the predictions of the

above model due to the finite size of the source emitter. The line source model can be used under some conditions as discussed later, where the deviation of the response of the physical system and the mathematical model predictions are not significant.

The second mathematical model, the finite source model, permits the introduction of the source heater and test medium thermal property data to correctly account for the effects of the thermal capacity of the finite source heater. This is the model used for experimental testing of liquids, and for work of the highest accuracy.

For the purpose of clarification, the following ranges of measurement test times will be presented.

Immediately after initiation of the electrical input to the source heater, the temperature of the source rises very quickly. In this very short time period, neither of the above mathematical models accurately describe the transient temperature response. The line source model is inapplicable since it does not account for the effects of the thermal capacity of the source heater. The finite source model is equally inapplicable since it is described in terms of infinite numerical series expansions which require excessive numbers of terms for convergence at short times. Physically, the experimental apparatus usually lacks the frequency response in the readout equipment to accurately respond to the rapid temperature rise in this time region. For the writer's test

systems, this time range extended from initiation of the test to approximately one half second later.

The next time range, the intermediate region extended from a one half second to six seconds after test initiation, for a typical test cell. The finite source model accurately describes the system response in this region. The line source model would be still inapplicable due to the source thermal capacity effects. Furthermore, the frequency response requirements of high precision laboratory readout equipment can be met with "off the shelf" equipment in this region. It is during this period of time that most transient source experimental systems are designed to operate.

The third time range, the long time region, extends from the end of the intermediate region to infinity. The line source model is applicable with suitable accuracy. The testing of solids and viscous liquids is generally carried out in this third time region, since instrumentation frequency response requirements are easily met even when using laboratory galvanometers as readout devices. For testing gases and liquids of low viscosity, the onset of natural convection around the source heater renders testing at long times impossible.

The origination of the source technique is commonly credited to Stalhane & Pyke (49), who first reported the phenomenon in 1931. They determined the thermal conductivity of water and glycerin at ambient temperatures, after developing a simple apparatus. Their apparatus consisted of a mercury thermometer, upon the bulb of which was coiled an electrical

resistance heater. They observed the characteristic logarithmic type rise of temperature with time under constant heat input. Further noting that the rate of temperature rise was dependent upon the test medium in which the thermometer was immersed, they deduced that the instrument could be used to determine the thermal conductivity of the test medium. An expression relating the thermal conductivity, the heat flux, temperature and time, was developed experimentally by the investigators, which can be shown to be identical to the line source model, as will be presented later.

Pfriem (50) in 1938 developed the line source mathematical model, complementing the work of Stalhane & Pyk, approaching the problem from an analytical basis. He further accounted for the effects of finite heat capacity of an experimental source heater, realizing that the line source model implied a source heater of zero radial dimensions.

Weishaupt (51) 1940, made use of Pfriem's work and completed experimental investigations for a series of liquids, including water, finding close agreement with Schmidt & Sellschopps (8) work of 1932, over the temperature range 20.8 C to 59.3 C. The line source heater consisted of a gold wire, .003 inch diameter, it and the test fluid being enclosed in a glass container. Weishaupt was the first of several later experimentalists to use the heater wire as a resistance thermometer as well. An unbalanced resistance bridge, having the heater wire as one arm permitted Weishaupt to monitor the resistance change in the gold wire. Weishaupt used a galvan-

ometer as the final readout device across the bridge
diagonal, but noting the lag inherent in the response of
galvanometers, performed a series of calibrations of slew
rate on the galvanometer. With these slew rate calibrations,
he was able to correct his galvanometer readings for the
inertial effects of the galvanometer. It appeared the
author was completely familiar with the effects of finite
wire size.

A series of papers were presented by Van der Held
et al., (44, 45, 52) in which they documented their work
with the source method. Their first paper appeared in
1949. Departing a considerable extent from the equipment
designed by Weishaupt, they developed a rather large source
heater, electrically heated, encased by quartz capillary
tubing, and with thermocouples for temperature monitoring.
An objective of their work was the measurement of thermal
conductivities of highly corrosive acids, precluding the
use of a single wire heater. The effects of the thermal
capacity of their relatively large source was corrected by
a graphical method.

The reader is referred to Van der Held's reports for
a detailed explanation of this correction technique.

Van der Held (44) was credited with first applying
the work of Kraussold (43) to the natural convective effects
in the line source system. He felt that the transient
system had a distinct advantage over the steady-state methods
regarding natural convection, due to the short measurement

time, this being sufficiently short that the density differences developed by the hot wire could not cause disturbing convective currents. In a subsequent paper (45) he showed that convective currents could result under some conditions.

Van der Held's results for water over a limited temperature range compared well with the Schmidt & Sellschopps data of 1932.

In a 1953 paper by Van der Held et al., (45), continuing investigation had shown that the simplified methods of correcting for the finite heat capacity of the line source were inappropriate. While the graphical correction method was valid for sufficiently long times, at short measurement times significant error resulted. When better methods of correction were applied, demanding more rigorous mathematical treatment, some disillusionment was expressed by Van der Held, since the simplicity and directness of the method was lost.

The 1955 paper by Van der Held (52) was prompted by the appearance of some discrepancies in results when testing fibrous insulation materials, using the line source method. Experimentally, a minimum in thermal conductivity was observed when the fibrous insulation was tested at the same temperature, but at variable densities. He attributed this to the contribution of inter-fibre heat radiation.

The subject method has been extensively applied for measurements on insulation materials, soils, and rock. The work at the University of Toronto in the 1950's was devoted to investigations of insulating materials and soils. Hooper

& Lepper (53) described in 1950, the development of a
semi-portable instrument called the thermal conductivity
probe for use in soil thermal conductivity measurements.
The method appeared to be ideally suited to this application,
since the measurement time was short, and during testing the
soil properties were not affected, the moisture content in
particular being unchanged. A typical probe consisted
of a 3/16" diameter tube, 1½ feet long, electrically heated
internally, with thermocouples used for temperature monitor-
ing.

Hooper & Chang (54) reported again in 1952, further
describing the technique, equipment and analysis required
to permit measurements on soils either in the laboratory or
in the field. Working charts were provided for analysis,
enabling the method to be used as a practical engineering
tool, providing for the user data of adequate engineering
accuracy for the intended investigations in soil heat
transfer.

Later, Lenz (55) applied the probe to materials of
low thermal conductivity such as slab cork and glass fibre
insulations. For these substances, the corrections of
Hooper & Chang were inappropriate since the thermal capacity
of the probe differed to a large extent from that of the
medium, whereas in the case of soils, the thermal capacities
did not differ to such a large extent. He did find that a
single fine wire source was satisfactory with these materials
provided care was taken with end heat losses.

Vos (56) in 1955 reported working with insulations following the work of Van der Held. He felt the source method was most appropriate for high temperature investigations of insulation materials, or for materials which would be changed physically by prolonged heating, as moist soil samples would experience in a steady-state system.

Blackwell (57, 58, 59) at the University of Western Ontario, completed both experimental and analytical work on the source method. When involved with the possible utilization of the probe method for in situ investigations of geological materials, certain inadequacies of the existing mathematical treatment for the finite probe became evident. He pointed out (44) that at that time, assumptions regarding axial heat flow along the length of the emitter were in doubt and that the presence of a thermal resistance at the probe wall had been assumed negligible. Length/diameter ratios selected for probes used up to that time failed to have a firm mathematical support and finally data analysis technique could be improved.

He consequently completed the mathematical analysis for the finite thermal probe, considering probe capacity and probe surface resistance to heat flow, providing two series solutions, one relevant for intermediate test times, and the other applicable to short test times.

For analysis of data, he proposed a "least squares" approach be used to correlate the observed data to the complete mathematical defining equation.

The papers (58) and (59) by Blackwell, gave a sound mathematical basis to axial heat loss calculations, and to values of optimum length/diameter ratios for thermal probes.

Gillam et al., (60, 61) conducted experimental work on liquids in 1955. Using a potential lead cell, with a platinum heater wire of .004 inch diameter as both heater and resistance thermometer, the authors tested water, glycerol, ethyl alcohol and sucrose solutions, reporting errors of the order .3%. An unbalanced Kelvin bridge was used to permit measurement of resistance of the heater wire, in conjunction with a galvanometer, calibrated for self inertia. Corrections for the finite heat capacity of the heater wire were made, and the data corrected graphically. In the latter report (61) the test cell was mounted in different positions, with the heater wire vertical in one test, at 45° to the vertical in another. This enabled them to check the onset of natural convection, noting that the vertical arrangement was the best.

They concluded that the method was highly accurate for viscous liquids or for less viscous liquids of low thermal expansion.

Allen (62) used the line source method to determine the thermal conductivity of transformer oils, claiming an error of not more than $\pm$ 5%.

Underwood & McTaggart (63) conducted experiments on the measurement of thermal conductivity for polystyrene and polyethylene from 40° F to 400° F. Effects of natural

convection were suppressed since these substances were either solids or very viscous liquids over this temperature range. Their line source system consisted of a .010 inch diameter heater wire about which was twisted thermocouple wire and a thermojunction.

In 1962 Horrocks & McLaughlin (64) reported using the source system for measurements on some liquid poly-phenyls, and toluene. Error of measurement was estimated to be $\pm$ .25%. The platinum heater wire, .0024 inch diameter was suspended in a glass enclosure, being fastened rigidly at one end, the other end being spring connected to the enclosure. Potential leads were spot welded to the four inch long heater wire about one half inch from each end. After installation of the heater wire, it was calibrated in situ by comparison to a standards platinum thermometer. Two tests were made for each data point, since a recording potentiometer monitored the change in voltage across the potential leads in the first run, the same recorder monitor-ing the change in heater current in the second run. The change in voltage and current were then transcribed into the change in resistance that the heater wire had undergone, permitting the determination of the temperature rise of the wire. The particular recorder used was insufficiently rapid in response necessitating a calibration of slew rate with consequent data correction for time lag.

Grossman (65) in 1963 used the line source method in a comparative technique whereby two line source cells

were connected to a dual resistance bridge network. A
co-ordinate recorder was connected to both bridges, and
thence responded to the both cells. When similar liquids
were used in both cells, the recorder pen traced a path at
45° from the horizontal pen axis, indicating that the
temperature rise for each cell at the same time was iden-
tical. If an unknown fluid were placed in one cell, and a
reference fluid in the other, the recorder response would
deviate from the 45° path, the deviation being directly
related to the thermal conductivity ratio of the two fluids.
Grossman recommended using a thermo-setting plastic material
in the reference cell to provide long term stability.

Jobst (66) 1964, completed an extensive investigation
of organic liquids over a wide temperature range from -100° C
to 200° C, using an absolute line source method. Experimental
results were estimated to be within 2% of absolute values.

Quite recently Tauscher (67) 1969, used a relative hot
wire system for rapid measurements of the thermal conductivity
of a family of refrigerants. The hot wire used for both
reference and test cell was very small, approximately .0008
inch diameter.

As a fitting conclusion to this review, the writer
notes the paper of Cremers (68), who used the source technique
to determine the thermal conductivity of a small sample of
Lunar soil particles recently returned by the Apollo 11
crew members. The method was selected for several reasons,
one being the limited size of the soil sample, another being

the necessity of obtaining preliminary data in a limited time. As a point of interest, the results obtained indicated that the lunar fines exhibited the same thermal conductivity as terrestial basalt fines of the same gradation.

CHAPTER 4

ANALYTICAL BACKGROUND

There are a number of physical systems wherein
the heat transfer and temperature responses exhibit the
subject characteristics. It is appropriate to describe
several of these examples prior to the presentation of
the mathematical model.

A common example is that of an insulated electrical
conductor which upon initiation of current flow experiences
internal self heating due to its' resistivity. When the
conductor is surrounded by some substance which could be ice,
as is the case of power transmission lines, or earth, as in
buried electrical conductors, or insulating material, as in
electrical resistance heaters buried in a refractory, the
temperature and the heat flux response of the conductor
closely approximates the line source constant heat flux
mathematical model.

Water pipes, or refrigeration pipes buried in the
earth exhibit the same characteristics upon initiation
of water or refrigerant flow.

These examples are characterized by a long, thin
cylindrical heater immersed in a substance which is infinite
in extent with respect to the heat transfer of the cylinder.

Furthermore, the cylinder is of essentially uniform temperature across its section at any instant, unlike the surrounding material which is of relatively high thermal resistance. If the temperature of the cylinder were monitored it could be observed that it increased approximately logarithmically with time from initiation of the heat transfer. It further could be observed that the rate of increase of cylinder temperature was related to the thermal conductivity of the surrounding material.

Just this was observed by Stahlane & Pyk (49) who recognized the possibility of measurement of thermal conductivities using the technique.

The system utilized by later investigators for thermal conductivity measurement generally consisted of a heater wire of length/diameter ratio reaching several thousand, immersed in the test sample. Electrical current was applied to the wire, resulting in a constant heat flux from the wire. Electrical input was monitored by appropriate equipment. Simultaneously the resistance of the heater wire was observed, this being converted to a temperature by the introduction of the temperature coefficient of resistance of the heater wire material. Knowledge of the temperature-time history, the heat flux and certain thermal and dimensional parameters enabled the determination of the thermal conductivity through application of a mathematical model which simulated the heat transfer process.

It was realized that the developed mathematical

model simulated the physical system accurately under particular boundary conditions and limits. Consequently the experimental system was designed for and operated within constraints imposed by these boundary conditions and limits.

4.1 Line Source, Constant Heat Flux Model

The mathematical model first derived to describe the response characteristics of the previously described systems and also the thermal conductivity experimental systems will first be presented. While this model can be shown to be inadequate under certain operating conditions, it was successfully applied to the writer's investigations for insulating materials.

The general differential equation for heat conduction in a homogeneous medium can be written in radial co-ordinates as follows:-

$$\frac{dT}{dt} = \frac{K}{CD}\left(\frac{d^2T}{dR^2} + \frac{1}{R}\frac{dT}{dR} + \frac{1}{R^2}\frac{d^2T}{d\theta^2} + \frac{d^2T}{dZ^2}\right) \quad (1)$$

Where

T is temperature	Deg. Fah.	
t is time	Hours	
K is the thermal conductivity	B.T.U./Hour-Foot-Deg.Fah.	
D is the density of the medium	Pounds/Foot^3	
C' is the specific heat of the medium	B.T.U./Pound/Deg.Fah.	
R is the radial co-ordinate	Feet	
θ is the angular co-ordinate	Radians	
Z is the axial co-ordinate	Feet	

For the case of a continuous line source extending to

infinity in an axial direction, with a constant energy
generation within this source, with the source heater
surrounded by an isotropic medium of infinite radial and
axial extent, the following boundary conditions apply:-

(1) $t = 0$, $R \neq 0$; $T = 0$

(2) $t > 0$, $R = \infty$; $T = 0$

(3) $t > 0$, $R \to 0$; $-2\pi K R \dfrac{dT}{dR} = q = constant$

where q is the line source strength   B.T.U./Hour/Foot

Regarding equation (1) above,

$dZ = 0$

$d\Theta = 0$

Under these conditions, equation (1) can be shown, follow-
ing Carslaw and Jaeger (41) page 261, to yield:

$$T(R,t) = -\frac{q}{4\pi K} \; Ei \left(\frac{-R^2}{4dt}\right) \tag{2}$$

Where

q is the source strength   B.T.U./Hour-Foot

K is the thermal conductivity

     of the surrounding medium   B.T.U./Hour-Foot-Deg.Fah.

Ei is the exponential integral

d is the thermal diffusivity

     of medium $Foot^2$/Hour

$T(R,t)$ represents the temper-

     ature at any radius

and at any time after initiation of the source constant
heat flux.

The exponential integral does not lend itself to explicit integration and its value is usually approximated by a numerical series solution. Abramowitz & Stegun (69) Page 229, show the conventional numerical solution as the following:-

$$Ei(x) = C + Ln(x) + \sum_{n=1}^{n=\infty} x / n \, n! \qquad (3)$$

where C is .57721... - Euler's Constant

The above series solution is valid for $x \rangle 0$. Progressively more numbers of terms in the expansion must be used for accuracy if the magnitude of the argument, x, is large.

Inherent in the solution of equation (2) above, was the concept of the mathematical line source, a line of limiting small radial extent wherein the constant heat flux was generated. This idealization could be only approximated in any physical system. Consequently application of the above model to a physical system would be incorrect due to the finite heat capacity of the emitter.

4.2 Line Source Model Used For Insulating Material

The preliminary analysis of data obtained for insulating material in this work used the mathematical model as described by equation (2). Expansion of the equation yields:

$$T(R,t) = \frac{q}{4\pi K}\left(-C + Ln\frac{4dt}{R^2} + R^2/4dt - \frac{1}{4}(R^2/4dt)^2 + - \cdots\right)$$

(4)

This expression has been utilized by Hooper & Lepper (53) and by Vos (56) for analysis of data on measurements of thermal conductivities of soils and insulating materials. It was rewritten to provide a temperature difference at two different times, at a fixed radial distance.

Assume the fixed radial distance to be R_o Ft. and the time of observation of temperature to be t_1 , and t_2 , during which the temperatures were T_1 and T_2 respectively.

Then
$$T(R_o,t) = \frac{q}{4\pi K}\left(-C + Ln\frac{4dt_1}{R_o^2} + \frac{R_o^2}{4dt_1} + - \cdots\right)$$

(5)

and
$$T(R_o,t) = \frac{q}{4\pi K}\left(-C + Ln\frac{4dt_2}{R_o^2} + \frac{R_o^2}{4dt_2} + - \cdots\right)$$

(6)

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Subtracting equation (5) from (6) one obtains

$$T(t_2, R_o) - T(t_1, R_o) = \frac{q}{4\pi K}\left(Ln(\frac{t_2}{t_1}) + \frac{R_o^2}{4d}(\frac{1}{t_2} - \frac{1}{t_1}) + \cdots\right) \qquad (7)$$

and succeeding terms have been truncated by many previous investigators, leaving:

$$T(R_o, t_2) - T(R_o, t_1) = \frac{q}{4\pi K} Ln(\frac{t_2}{t_1}) \qquad (8)$$

Rearranging

$$K = \frac{q}{4\pi K} \frac{Ln(\frac{t_2}{t_1})}{(T(R_o, t_2) - T(R_o, t_1))} \qquad (9)$$

Upon experimental measurement of t_2, t_1, $T(R_o, t_2)$ $T(R_o, t_1)$ and q, the value of K may be determined. The value of R_o need not be determined, but as may be seen from the form of the truncated terms, small values of R_o introduce less truncation error.

There are several sources of error inherent in the analysis as presented by equation (9), their magnitude

being dependent on the particular physical system simulated, and its operating conditions.

The first error due to truncation of the succeeding terms of the series expansion for the exponential integral, equation (3) was shown to not introduce significant error for the range of argument experienced in experiments undertaken by the writer. Computer compiled calculations, completed early in the writer's programme are presented in Appendix 1 of this report.

The second error due to the effect of a source of finite dimensions can introduce significant error, dependent upon the thermal capacity ratio of the test medium and of the finite heater wire. The more complete finite source model eliminates this error through introduction of the system thermal capacity ratio. It is possible, however, to utilize the simple mathematical model and correct the subject error by a graphical means as described by Hopper & Chang (54). For the portable thermal conductivity probe developed by these authors for field work with soils, the utilization of the more complete mathematical model would have introduced unnecessary complexity.

4.3 Finite Source, Constant Heat Flux Model

Approximate solutions have been developed which include the effect of the finite size of heat source. Carslaw & Jaeger (41), or Blackwell (57) have shown the analysis and resultant mathematical model for this case.

Blackwell used boundary condition and limits as follows:-

1) $t > 0$, $R_1 < R > \infty$; $\dfrac{d^2 T}{dt} + \dfrac{1}{R}\dfrac{d T_2}{dR} = \dfrac{1}{d}\dfrac{d T_2}{dt}$

where R_1 is the radial co-ordinate of the

    finite source.   feet

    d is the thermal diffusivity of the

    surrounding medium. $feet^2$/hour

    T_2 is the temperature of the surround-

    ing medium.   Deg. Fah.

2) $t > 0$, $R = \infty$; T_2 is bounded.

3) $t = 0$, $0 < R < \infty$; $T_2 = T_1 = 0$

where T_1 is the temperature of the finite source   Deg. Fah.

4) $t > 0, R_1 = R \; ; \; -K\dfrac{dT_2}{dR} = H(T_1 - T_2)$

where K is the thermal conductivity of the

surrounding medium.   B.T.U./Hour-Foot-Deg.Fah.

*H is the surface conductance between

the finite source surface and the

surrounding medium.   B.T.U./Hour-Foot^2-Deg.Fah.

This latter boundary condition permits the intro-

duction of a surface resistance between the finite source

and the medium. When the resistance is negligible, $H \rightarrow \infty$.

5) $t > 0 \; , \; R_1 \gg R > 0 \; ; \; T(R_1,t) = T(R \rightarrow 0,t)$

This condition implies the perfect conductance of the

finite source.

6) $t > 0 \; , \; R_1 = R \; ; \; -2\pi R K \dfrac{dT_2}{dR} = q - M_1 C_1 \dfrac{dT_1}{dt}$

where M_1 is the mass/unit length of the

finite source.   Pounds/foot

C_1 is the specific heat of the

finite source.   B.T.U./Pound-Deg.Fah.

q is the finite source strength B.T.U./Foot-Hour

*Blackwell was interested in applying the probe to

measurements of thermal conductivity in soils where in fact

there is an air film between the probe surface and the soil.

This condition imposes the effect of the finite heat capacity of the source.

Blackwell (57) Page 138, using Laplace transformations of the boundary conditions and of the differential equations, was able to derive the solution in real infinite integrals. He evaluated these integrals by approximate series expansion for two cases, that of short time and that of long time.

As applied to thermal conductivity studies, the response of the system very quickly passes from the short time to the long time solution, and only the long time approximate solution has direct application.

Blackwell's long time approximate solution is presented below.

$$T(t) = \frac{-q}{4\pi K}(C - Ln\frac{4dt}{R} - \frac{2K}{H}$$
$$+ \frac{R^2}{2dt}(C - Ln\frac{4dt}{R^2} + 1 - W(C - Ln\frac{4dt}{R^2} - \frac{2K}{H}))$$
$$- \frac{R^4}{d^2t^2})).$$

$$(10)$$

where **C** is .5772......   -Euler Constant

    H is the surface conductance at the finite source surface.

    W is the thermal capacity ratio, thermal capacity

of probe/thermal capacity of test medium.

R is the radial co-ordinate.

For the case of negligible thermal resistance at the surface of the finite source,

$$T(t) = \frac{-q}{4\pi K}(C - Ln\frac{4dt}{R^2}$$

$$+ \frac{R^2}{2dt}(C - Ln\frac{4dt}{R^2} \div 1 - W(C - Ln\frac{4dt}{R^2})) - \frac{R^4}{d^2 t^2}).$$

$$(11)$$

If "W" were to assume the value unity (thermal capacity of the finite line source equivalent to the thermal capacity of the test medium) equation (11) resolves to the solution for the mathematical line source as given by equations (2) and (3).

4.4 Finite Source Model Used For Experiments
Of Highest Accuracy

When experimental determinations of thermal
conductivity are undertaken, one must use the mathem-
atical model of equation (11) for highest accuracy.
The data should then be correlated against the equation
using least squares or a similar technique to reduce to
a minimum the random error associated with the experim-
ental equipment.

It is furthermore essential to show that the
experimental system and mathematical model are consistent
with respect to the boundary conditions of the model.
The conditions are listed below, referenced to the boundary
conditions presented in Section 4.2.

1. Boundary Conditions 1 and 2 - Test medium is infinite
in extent with respect to radial heat transfer at all
times during a test.

2. Boundary Condition 1 - Heat transfer mode must be
radial conduction only.

3. Boundary Condition 2 - Test medium is homogeneous with
respect to heat transfer.

4. Boundary Condition 3 - System must be isothermal through-
out before a test is started.

5. Boundary Condition 4 - The assumption of negligible

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thermal resistance at the source - test medium interface must be shown valid, or if present, shown to introduce negligible effect.

6. Boundary Condition 5 - The finite source must remain at constant temperature across its diameter at all times.

7. Boundary Condition 6 - The sole heat source must be the finite source. There shall be no heat sinks.

8. Boundary Condition 7 - The heat source must remain at constant magnitude throughout a test.

9. It must be shown that the long time mathematical solution is valid for the operating conditions of the experimental system.

These above conditions are required for proper similarity between the mathematical model and the experimental system. Any experimental errors due to equipment calibrations, or data reduction, would be in addition to the forementioned and must be considered as well in an overall error analysis.

Each of the previous conditions has been considered independently and the results indicated below:-

1. Test medium is infinite in extent with respect to heat transfer.

Since a thermal conductivity cell must be thermostated, and enclosed in a pressure vessel if the data for

liquids are to be investigated under various temperatures and pressures, it is obvious that the test medium must be bounded. It is necessary to deduce the limits of time during which the cell appears as an infinite reservoir with respect to the heat transfer from the emitter. The thermal conductivity testing must be carried out within this time interval.

For a bounded cell, there is eventually established a steady-state temperature distribution across the cell.

Fortunately, there is available a mathematical solution to this problem, (see Carslaw & Jaeger (41), Page 334). One solution, commonly known as Fischer's solution, was used by Horrock's & McLauglin (64) to determine the length of time before the effect of the boundary was apparent.

Horrock & McLaughlin (64) formed a ratio of temperatures as predicted by Fischer's Solution and by the semi-infinite finite source solution. They determined the magnitude of this ratio for a range of parameters, and were able to utilize the information for subsequent equipment design.

Their conclusions are summarized below:-

$$\frac{d\,t}{R_o^2} < \cdot 12 \qquad \frac{T(R_1,t,L\cdot S\cdot)}{T(R_1,t,B\cdot S\cdot)} < 1\cdot 0001$$

where $(T(R_1,t,L\cdot S\cdot))$ is the temperature as predicted by the semi-infinite finite source solution (Sect. 4.3 –

equation 11).

$T(R_I, t, B \cdot S \cdot)$ is the temperature as predicted by the bounded line source solution (Carslaw & Jaeger (41) Sect. 13.4 - equation 12).

d is the thermal diffusivity of

the test medium. $Foot^2/Hour$

t is the time.   Hours

R_o is the radius of the bounded

test cell.   Feet

R_I is the radius of the finite

line source.   Feet

The same criterion was utilized by the writer in equipment design. For the substances tested, and with the cell geometry adjusted consistent with the above limits, no effects of the bounded cell would be expected.

2. Heat transfer mode must be radial conduction only.

This requirement necessitates showing that axial conduction through the finite line source is negligible, that no significant heat transfer by radiation is present, and that heat transfer by natural convection is insignificant.

Regarding axial conduction through the finite source, the writer completed a series of calculations assuming that the ends of the finite source at their attachment

points experienced the initial equilibrium temperature

of the cell. Since the potential lead arrangement has

been generally utilized for precise experiments, and was

used by the writer, the active section of the finite source

heater would be quite isolated from end effects. Results

of the mathematical analysis as presented in this report,

Appendix 2, confirmed that axial conduction by the finite

source heater would be negligible.

Heat transfer by radiation from the finite line

source has been investigated by several previous experim-

entalists. Horrock's & McLaughlin (64) showed that heat

transfer by radiation represented approximately 1/1000 the

magnitude of conductive heat transfer for their particular

test system, using conservative assumption for the radiative

heat transfer.

It should be noted that Horrock's & McLaughlin's

(64) work was concerned with liquids, for which radiation

was negligible with respect to conduction. For certain

insulation materials, however, Van der Held (52) found

that radiation could, in fact, be important, and would

need to be considered when determining thermal conductiv-

ities of these mediums.

Natural convection remains as the sole heat trans-

fer mechanism which for liquid and gaseous media must

be shown to introduce negligible effect on the radial

conduction. The approach used previously by other investig-

ators for both transient and steady-state hot wire systems

has been referred to the early work of Kraussold (43) or Beckmann (42). The writer has pursued their criterion as a guide, the method and analysis being presented in Appendix 3. Fortunately for the finite source method, when testing fluids, it is possible to observe experimentally significant departure from pure conduction, by two methods. The first is to observe the departure of the temperature rise from the mathematical model. The second consists of observing the test date when the cell is operated at differing angles of inclination from the vertical.

3. Test medium is homogeneous with respect to heat transfer.

The writer can foresee no possibility of the test medium experiencing inhomogeneities when that medium is a pure fluid.

When testing granular insulation materials, the possibility exists of the material segregating. No general conclusion can be made in this case, as it necessarily is a characteristic of the particular granular medium.

4. System must be isothermal throughout before a test is started.

The maintaining of an isothermal thermal conductivity cell imposes two requirements. The first is the

design, testing and subsequent operation of an enclosure

which will provide an isothermal environment for the cell

under all expected operating conditions. For a partic-

ular enclosure utilized by the writer, the design and

testing is discussed in Section 5.3-3, this report.

The second requirement is the determination analytic-

ally, or experimentally of the length of time necessary

for the test medium to equilibrate in a radial direction.

Analytically, the approach is a straight forward problem

in transient heat conduction. Provided the thermal diffus-

ivity of the test medium of the enclosure is known and the

magnitude of the thermal resistance between the cell and

enclosure is estimable, a prediction of the time for over-

all equilibrium can be made. Experimentally, the operator

of the thermal conductivity apparatus can monitor at any

time the temperature difference between the finite source

and the enclosure. Experimental observation of this temper-

ature difference for several different times after a test

enables the operator to easily establish just how long he

must wait before equilibrium is reached.

5. Effect of thermal resistance at the test medium-

finite source interface.

When one is involved with thermal conductivity

experimental determinations of the highest accuracy, it

is prudent to consider the effect of an interface resist-

ance at the finite source-test medium interface. This
could be of significance in some cases, depending on
the test medium.

Solids -

Blackwell (57) pointed out that for in situ tests
with rock, the presence of a thermal resistance between
the finite source and test medium was of importance, and
of important consequence for the short time approximate
solution for the finite probe. It was of less importance
for the long time approximate solution. He further showed
that the magnitude of this thermal resistance coefficient
could be determined by interpretation of the experimental
results for the short time tests.

Other workers using very small finite sources wherein
the short time approximate solution is not relevant since
the system very quickly passes into the long time region,
have seen fit to neglect the effect of the interface
resistance. Lenz (55) found a single fine wire source
gave satisfactory results when testing insulation materials,
even when neglecting both the probe thermal capacity effects
and the interface resistance.

For the tests of insulation completed by the writer,
no thermal interface effects were present. The temper-
ature sensor, in this case, was a thermocouple junction
which was positioned a small distance away from the finite
source heater. The thermocouple junction would experience
only a small thermal flux into it, due only to its thermal

capacity. No significant temperature difference between the medium and junction could be expected.

Liquids -

Provided the test medium wets completely the finite source, there is no evidence that a thermal resistance appears at the source-medium interface. Years of experimental work with steady-state thermal conductivity systems of the hot wire type have supported this conclusion.

Gases-

Unlike the results of liquid steady-state experimental work, there is a considerable volume of results which have indicated that a finite temperature difference can exist between a fine wire and the gaseous test medium. The magnitude of this temperature difference has appeared to be largely a function of the density of the gaseous medium, as well as its other thermal properties, but needs to be considered only at very low densities.

Application of the transient finite source method to gaseous mediums must be considered carefully both from this aspect and from additional problems arising from the magnitude of the finite source/test medium thermal capacity ratio.

Only preliminary investigations were conducted by the writer involving gaseous medium, and he must present this area as a subject of further investigations.

6. The finite source must remain at constant temper-

ature across its diameter at all times.

For the diameter and heat flux rates experienced
in the writer's investigations, the finite source remained
at a near uniform temperature across its diameter at all
times. This conclusion is supported by analytical calcul-
ations, shown in Appendix 4.

7. The sole heat source must be the finite source.

The only possible heat source other than the finite
source could be contributed by a non-isothermal enclosure.
The enclosure used by the writer was designed to provide
a uniform temperature along its length. Test results for
the enclosure are discussed in Chapter 6.

8. The heat source must remain at constant magnitude
throughout a test.

During tests of the highest accuracy, the equipment
designed and used by the writer permitted the monitoring
of the ohmic heat flux at all times. In general, the
heat flux during a test varied less than 1 part in
10,000. This can be shown to introduce less than .005%
error in the value of the measured thermal conductivity.

9. The long time mathematical approximate solution
is valid for the operating conditions of the experimental
system.

Appendix 1 contains calculations which illustrate
the magnitude of the truncation error for the line source
solution. For the value of the argument experienced in
the writer's tests, the truncation error was negligible.

When completing tests of highest accuracy, the
finite source model, equation 11, was utilized. The reader
is referred to Blackwell (57) for a discussion of the
possible magnitude of the truncation error in this case.

CHAPTER 5

DESIGN AND DESCRIPTION OF EQUIPMENT

The equipment used by the writer is described within this chapter. Furthermore, relevant design criteria necessary in apparatus design are presented.

In order to minimize the extent of footnotes, the writer has compiled a complete list of the laboratory equipment utilized during testing and refers to same by item number. This list appears at the front of the report.

As was consistent with both the writer's and his advisor's research philosophies, the testing programme was broken into discrete phases. The first phase was carried out in under two months and consisted of fabricating the simplest possible arrangement of equipment which could show the source technique of thermal conductivity measurement. The second phase involved an extension to different test media with improved equipment. This period of testing involved considerably more time, approximately one year. The final phase included an extensive review of secondary factors of importance in equipment design and selection, and ultimately led to a final system capable of standards laboratory accuracy.

This philosophy of progressing from the simple to

the more complex was beneficial from several aspects. It provided the writer with an increasing awareness of and experience in the subject technique. It provided a sound basis for equipment development, and finally, it permitted a termination at a preliminary phase if the experimental technique should prove in general, not feasible.

The experimental technique and calibration results are covered in Chapter 6, whereas the following Chapter 7 includes the results of testing. For consistency in interpreting a particular phase of the programme, the reader should study, for example, Section 5.1, Section 6.1, and Section 7.1.

5.1 Simplified Apparatus For Testing Insulating
Materials

Under the initial phase of the testing programme,
a very simple apparatus was built and utilized by the
writer to determine the thermal conductivity of dry
granular insulating materials. Eight tests were conducted
during this work.

Illustration 1, Page **249** shows diagrammatically
the equipment and arrangement used to conduct tests on
the granular asbestos insulating material, commonly known
as "Vermiculite".

The heater wire consisted of "Nichrome V" resistance
alloy, the diameter being .010 inch nominal. The effective
length between the potential leads was measured and found
to be 10.16 inches at ambient room temperature. Over this
length, the average resistance was determined to be 4.20179
ohms, the latter value being measured by means of a potent-
ial comparator system (Item 1, 2, 3, 4, Page **XI**)*. The
resistance determination was completed while the wire
experienced the laboratory temperature of $70^\circ F \pm 1^\circ F$.

In immediate proximity to the heater wire, the
active or hot junction of an absolute thermocouple pair

* A complete list of equipment is described on
Page **XI** of this report.

was attached. The reference junction was immersed in an agitated ice bath (Item 5, Page XI).

The thermocouple wire, (Item 6, Page XI) of gauge 30, met type TT specification. The junctions were formed by twisting together the respective wires, and silver soldering.

During a test, the potential difference of the differential thermocouple was monitored by a potentiometer and galvanometer (Item 7, 8, Page XI).

The electrical power to the heater wire was supplied by a regulated power supply (Item 9, Page XI), which operated in the constant voltage mode.

A small portable electric timer (Item 10, Page XI) served as a visual indicator of elapsed time during a test.

As is shown on Illustration 1, Page 249, the heater wire, the active thermocouple, the test medium and the assorted electrical connectors were contained in a glass vessel of approximately 1/3 cubic feet volume.

A standard shunt (Item 11, Page XI) was connected in series with the power supply. The same potentiometer as above was used to monitor the voltage drop across the shunt, enabling the determination of current flow.

5.2 Apparatus For Preliminary Tests Of Fluids

Consistent with the testing philosphy, an extension
of the first phase of experimental work to fluids was
followed. Instrumentation and test cells of improved
design were used, based upon experimental feedback and
more complete analysis.

Within this second phase of testing, four thermal
conductivity cells and two electrical networks were dev-
eloped for use with water and air. In total, 91 thermal
conductivity tests were undertaken, as well as separate
equipment and calibration tests.

5.2.1 Thermal Conductivity Cells

Test Cell A

The first cell constructed was used for thermal conductivity measurements of air under ambient pressure and laboratory temperature. The source heater consisted of a single length of pure nickel wire, .010 inch diameter, (Item 12, Page XI). It was suspended from a laboratory retort stand, and was for the initial test, not enclosed. Later, significant erratic temperature changes of the wire indicated air currents were affecting the wire, and it was encased by a paper cylinder, approximately 8 inches diameter and open at the top.

The total length of wire was approximately 12 inches. Two potential leads, of .005 inch diameter nickel were spot-welded to the main heater about 3 inches from each end. The active test length of 6.00 $\pm$ .01 inches was measured by vernier calipers.

This first cell exhibited several problems. The first was the difficulty of maintaining a draft-free environment about the source heater.

A second problem involved the transmission of vibration through the laboratory table to the retort stand, through the heater wire and to the tensioning weight, resulting in a change in the wire resistance due to

changing stress conditions in the wire. The writer was unable to assess the relative effects of these two disturbances at the time of testing. Another disadvantage was the impossibility of testing liquid mediums with this cell.

Test Cell B

This cell was used for thermal conductivity tests of both air and water.

The source heater consisted of a single length of pure nickel wire, .010 inch diameter. Two potential leads, of .005 inch diameter gold wire, were attached to the heater wire leaving an active length of $6.10 \pm .01$ inch between them.

A plastic framework was constructed to hold the source heater. It consisted of two 1/2 inch thick "Perspex" discs, 10 inch diameter, spaced and supported by three 1/2 inch diameter "Perspex" rods. The heater wire was rigidly fastened to a central binding post on one of the discs, and was attached to a flat copper leaf spring at the other disc. The leaf spring maintained a tension in the heater wire at all times.

The whole assembly of heater wire and plastic framework were then set inside a cylindrical glass vessel, approximately 12 inch inside diameter by 12 inch height. (See Illustration 1).

All current and potential leads were brought to suit-

able copper binding posts mounted on the topmost "Perspex" disc.

With Cell B, the first tests with water were possible. It was, however, impossible to minimize convective flows in the glass vessel since it was very large compared to the wire size. Preliminary tests with dye showed that circulatory currents always existed within the vessel, even though the source heater was not energized, possibly due to laboratory temperature changes. The writer further suspected that the nickel-gold potential lead joint to the source heater introduced spurious thermo-voltages, since there existed bias voltages at the readout equipment when the heater was de-energized. This could not be absolutely supported, however, since development of a less noisy electrical network was being continued at the same time, this noise shown later to be caused by inadequate shielding and grounding.

Test Cell C

This test cell was utilized in both air and water testing. The heater wire consisted of a length of pure platinum wire, .010 inch diameter. It was centrally contained within a 12 inch length of 1.25 inch diameter glass tubing.

Two potential leads were spot-welded to the central source heater and consisted of .003 inch diameter pure platinum wire.

The active source length between the potential taps was $10.031 \pm .010$ inch, measured by a vernier height gauge.

The test cell was assembled by moulding a ceramic

cement plug into each end of the glass tube, after the source heater and potential leads had been installed. Electrical terminations for these leads and fluid charging connections were embedded in the ceramic end plugs.

The source heater was placed under tension by suspending a small weight on the end of the current electrical termination before the ceramic cement had set, the lead wires consequently experiencing a permanent tension.

It was discovered during initial checking of Cell C, that the ceramic cement had deteriorated due to the constant immersion in water.

Test Cell D

Test Cell D was utilized for measurements of the thermal conductivity of water. The source heater wire consisted of pure platinum wire, .0025 inch diameter.

A glass tube, .75 inch inside diameter served as the enclosure and provided structural support for the source heater.

Two potential leads, of .0025 inch diameter platinum were spot welded to the source heater, leaving $11.560 \pm .010$ inch length of active source heater.

An epoxy type bonding agent was used to cap the ends of the tubing and further insulate, support and seal the electrical lead-in wires and hypodermic tubing. The latter served as charging access for the test medium. On occasion, the complete cell was immersed in a one gallon container of

transformer oil, to dampen the room temperature variations.

5.2.2 Electrical Networks

For the tests undertaken as described under
Section 5.2, the finite source heater served as both the
heater for the uniform heat flux and also as the temperature
monitor, using the resistivity variation of the heater
material as the indicator of heater temperature. The
instrumentation problem could be described as the measure-
ment of small changes in current and voltage superimposed on
a large bias current and voltage. Direct currents were
utilized throughout. The first approach exploited the
resistance bridge technique whereby the finite source heater
formed one arm of the bridge. Subsequent testing resulted
in a revised network in which the bridge system was replaced
by potentiometers which were used to reduce the bias voltage
and current to zero at the readout instrument.

Bridge Network

The circuit illustrating the bridge arrangement is
shown on Illustration 2 of this report. The right hand
upper arm consisted of a two decade resistance box
(Items 13, 14, Page XI), providing resolution to .1 ohm.
The lower right resistance arm was formed by utilizing the
variable branch of a Mueller Bridge (Item 15) as a variable
resistor.

The upper left bridge arm was fabricated from a
group of commercial power resistors, connected in series

and in parallel to provide a total resistance of 33.630 ohms. The power level in this bank of resistors during a test represented approximately 1/10 the commercial rated power level of the resistors.

The lower left arm consisted of the thermal conductivity cell heater wire and potential taps, connected as shown.

A standard shunt (Item 11) 1.000 ohm was inserted to measure the total bridge current.

The power dropping resistor was composed of a collection of commercial 50 watt resistors, arranged to provide taps of different resistance, from 1 to 300 ohms. These resistors during a test operated at less than 1/20 commercial rated power level.

For preliminary initial experiments, a regulated power supply (Item 9) was used to energize the bridge. This was later replaced by a bank of six volt 120 ampere hour lead-acid storage batteries.

The bridge voltage was monitored by an electronic chart recorder (Item 16) with a chart speed of 1 inch/second and a full span sensitivity of 100 microvolts.

The bridge current was monitored by a potentiometer and galvanometer (Item 7, 8, Page X I).

The bridge current ratio was maintained at 100 to 1. Maintaining this ratio posed some difficulty, and to insure accurate current measurements, the 1 ohm standard shunt was placed in the upper left hand arm. The value of resistance

on the upper right arm was then increased by 100 ohms, to maintain the bridge ratio.

All bridge connections consisted of #14 gauge unplated single conductor copper wire.

The bridge network presented an array of operational difficulties. It required a considerable time period to balance the bridge accurately, since it necessitated several checks and adjustments each time, occupying in all approximately 15 minutes. Since none of the cells utilized in this part of the programme were thermostated to a high degree, the source heater reflected the ambient changes in laboratory temperature, resulting in an unbalanced bridge by the time the bridge was ready for a test run.

During the preliminary experimental work with the bridge arrangement, it was deduced that the power resistor and the dropping resistor, (see Illustration 2) were exhibiting resistance changes with time during a test. It was further concluded that this was a result of internal self-heating effects, although their commercial power ratings exceeded greatly the operating power level.

To minimize the temperature instability, two new variable resistors (Item 17, 18) were constructed. For these, the resistors consisted of "Karma" wire (Item 19) wound on porcelain mandrels. "Karma" wire exhibits an extremely low temperature coefficient of resistance.

The mandrels and resistor wire were rigidly attached to the underside of a 1/2" thick "Perspex" disc. The copper

binding posts used for terminations of the resistor bank
and taps were attached to the top of the disc, with all
connections being silver-soldered. The disc was machined
on its underside to provide a check which fitted over
the rim of a five gallon metal container. A hole was prov-
ided in the disc through which a thermometer was inserted,
its bulb being immersed six inches in the transformer oil.

The completed resistor banks were then immersed in a
five gallon container of transformer oil to temperature
condition the resistors. The resistance was measured for
these units, the results and technique being described in
Chapter 5.

The complete bridge arrangement, consisting of many
separate components separated over approximately 16 square
feet of laboratory table, was persistently electrically
noisy. This noise was not satisfactorily removed, even
after a considerable degree of development.

The writer subsequently looked into other techniques
to monitor the current and voltage relations of the potent-
ial lead type thermal conductivity cell.

A variation of the potential comparator technique was
developed for the subject problem. This arrangement was
used subsequently for all further testing.

Modified Potential Comparator Technique

The general arrangement of the electrical circuit
used is shown on Illustration 3.

On the left hand side is shown the power supply, standard shunt, and power dropping resistor associated with the thermal conductivity cell finite source heater.

On the right hand side **is** shown the two potentiometers which provide the constant voltage required to buck the bias values of voltage across the standard shunt and the potential taps of the cell.

The two channel chart recorder (Item 16, Page **XI**) is interposed between the potentiometers and the standard shunt and potential taps in such a way as to monitor the change in current and voltage through the finite source heater to enable its resistivity determination.

Associated with the network was a number of interlocking relays which provided the following functions:-

A. During the times that a test was not being conducted, a resistor, equivalent in value to the cell, was connected across the power supply, to present a uniform load to the batteries. This resulted in a constant battery voltage.

B. During the time that a test was not being conducted, the potential lead - potentiometer - chart recorder circuit was disconnected, to prevent the chart recorder attaining the excessively large potentiometer bucking voltage value, which would have overloaded the amplifier.

C. Simultaneous with B above, the chart recorder input was shorted to facilitate zero adjustment of the pen.

D. One master switch energized the above relays, but a resistance-capacitance circuit was interconnected between the master switch and relays A and B above, to enable the imposition of a time lag. The latter time lag was necessary to isolate the recorder amplifier from voltage transients.

Based upon the problems experienced in the bridge arrangement previously described, precautions were taken to suppress electrical noise. These included:-

A. All connecting leads consisted of shielded electrical conductor (Item 20), single copper conductor.

B. The potentiometer and chart recorder internal shielding connections and the shields of interconnecting wires were brought to a common point.

C. A #14 gauge single copper conductor was connected to the metal chassis, and to a copper water line in the laboratory. The ground connections were mechanically made, then silver-soldered.

D. All equipment comprising the electrical network which required 110 volt alternating current for operation, were separated from each other through power line isolation transformers.

E. To minimize spurious thermo voltages at connectors, the minimum number of mechanical connections were made. Where possible, silver-soldered joints of similar conductor materials rather than mechanical connections between conductors were utilized.

5.3 Apparatus For Tests Of Fluids Capable Of High Accuracy

As the final phase of the experimental programme, the writer and his advisor decided to embark upon the construction and operation of a thermal conductivity measurement system which would be capable of providing data on liquids over a range of pressures and temperatures. All previous apparatus had been designed for ambient laboratory conditions. **Design limits were 0 to 3,000 psia, 70° to 700° F.**

It was the opinion of the writer that sufficient background experience had been accumulated to assist bringing this to pass in a reasonable time interval.

To bring this final phase to a satisfactory conclusion, it was necessary to investigate the following aspects of the equipment design:-

A. Select the most suitable material for the source heater, and determine the optimal dimensions.

B. Investigate and select a satisfactory physical arrangement of the thermal conductivity cell, including the method of suspension of the source heater.

C. Design, fabricate and test a pressure vessel-thermostat which would provide the proper environment for the thermal conductivity cell.

D. Investigate, assemble and test a suitable electrical readout system.

E. Describe the limits and undertake the testing of purity for the test medium and evaluate the contamination, if any, experienced by the medium after prolonged testing.

In the design of a system for experimental thermal conductivity system, one must consider the thermal properties of the test medium being investigated. In this final phase of the programme, the fluid chosen was water. It was the intention of the writer and his advisor to initially study H_2O, then at a later date investigate deuterium oxide. The chemical and thermal simularities of the two liquids would permit the same thermal conductivity cell being used for either media with no change in apparatus or operating conditions.

5.3.1 Selection Of Material And Optimizing The Geometry
Of The Finite Source Heater

Material Selection

Important to the successful application of the
finite source method of thermal conductivity measurement
was the selection of the optimal material for the source
heater. In the method used by the writer, the source heater
served as both an electrical resistance heater for gener-
ation of the heat flux, and also as a resistance thermo-
meter. The dual functions of the heat emitter are not
unique to the subject method, as similar techniques have
been used in steady-state hot wire thermal conductivity
cells for many years. There was consequently, some back-
ground information available regarding the important con-
siderations for material selection.

An important factor was the magnitude and stability
of the temperature coefficient of resistivity of the
heater material. To accurately determine the temperature
of the finite source, it was necessary to monitor the
resistivity of the source, and thence convert the resist-
ance change to the associated temperature change through
the temperature coefficient of resistivity for the material.
The stability of this factor should be of an extremely
high order if a resistance-temperature calibration was to

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be carried for a considerable length of time in the
course of experiments. Pure reference grade platinum
was known to be adequate in stability of resistance, having
long been utilized in the measurement of temperature through
resistance measurements and largely as a result of its applic-
ation to this area, has been studied thoroughly with respect
to the temperature coefficient and the stability of the
same. Pure nickel could be considered as the next best
material from this standpoint.

The magnitude of the temperature coefficient of resist-
ivity was also of importance, insofar as it affected the
gain specifications of the readout equipment. A large value
would require less instrument gain.

In Appendix 5 is shown a list of pure metals, with
their respective thermal, electrical and chemical character-
istics, of importance to thermal conductivity studies. Pure
platinum possesses a temperature coefficient of an inter-
mediate magnitude, while nickel exhibits the highest value
for the metals listed.

Another factor to be considered was the degree of
chemical activity between the metal and the test medium,
water in this case. All the materials in Appendix 5 are
relatively inert to water. Chemical attack of the finite
source heater would be detrimental to the thermal conduct-
ivity cell through its effect on the dimensions of the

wire and the resultant resistance change. Corrosion would further contaminate the test fluid sample.

A factor of importance to the subject thermal conductivity cell was the value of the thermal capacity ratio of the finite heater material to the test medium. Due to inherent limitations in the mathematical model of the thermal system, it was advantageous that this ratio be as close to unity as possible. For experiments with water as the test medium, nickel rated highest with respect to this factor, while gold was the least desireable.

Several mechanical properties of the material selected were important. While ultimate strength of the material would not be of consequence, the material should be ductile, with a freedom from strain hardening. The assembly of the apparatus necessitated a considerable degree of cold working of the material, and some movement could be expected in service. As a result, a non-ductile material could not be considered satisfactory. It has been the writer's experience that only platinum and gold exhibit sufficient ductility.

Satisfactory terminations and potential taps must be made to the heater wire. In the case of the latter, spot-welding has appeared to be the only satisfactory method of attachment. It was advantageous if the material selected could be easily welded.

The ideal material for the finite source heater would possess no resistance change due to variations in stress

level within that material. Since none of the metals shown
in Appendix 5, or in fact, any pure metals possess this
characteristic, it would be desireable that the resistance
change due to stress change be small compared to resistance
change due to temperature change.

The extent of strain sensitivity would dictate limits
for permissible strain variations within the heater when in
service. This in turn would determine the degree of mechan-
ical sophistication within the cell to meet those limits.

A factor of minor consideration in the heater material
was the magnitude of the thermo voltage against copper.
The terminations of the thermal conductivity cell to the
electrical connecting leads could, if not at a uniform temper-
ature, introduce a thermo voltage which would appear as a
bias voltage at the readout devices. Since the connecting
leads would be copper, the magnitude of the thermo voltage
of the pure metals against copper is shown for reference in
Appendix 5.

After due consideration of the above mentioned factors,
the best material was concluded to be pure platinum, refer-
ence grade. Of major importance was the stability of the
temperature coefficient of resistivity and the ductility of
that metal.

Selection Of The Optimum Geometry For The Finite

Source Heater

The optimal diameter and active length of the source heater can only be determined after a detailed study of many factors. The experimental equipment design, the boundary conditions and limits from the mathematical model, and the writer's experience should all be considered in the final decision.

Following a brief introduction, each factor will be considered in detail in this section of the report.

Upon referring to equation (11) Chapter 4, one will observe that the smaller the value of the finite heater diameter, the less significant the thermal capacity ratio. Furthermore, the truncation error decreases as the fourth power of diameter. Consequently, for very small emitter diameter, the finite source model approaches the idealized line source solution. Further consideration will, however, show that in the limit of small diameter, both the mathematical model and the experimental system become meaningless since the model approaches a singular point and the physical system approaches infinitely large temperature rise for the source heater.

Before this condition of mathematically small diameter is approached for a platinum-water system, the finite heater becomes too small for manipulation.

Another condition is imposed by the response characteristics of the data acquisition equipment. As the frequency response requirements of the data acquisition equipment

increase with smaller diameter emitters, microvolt
resolution is increasingly more difficult and expensive
to attain.

These factors will be presented below:-

1. Effect Of Diameter On The Finite Source Mathematical Model

Equation (11) Chapter 4 is rewritten for reference
below:-

$$T(t) = \frac{-q}{4\pi K}(C - Ln\frac{4dt}{R^2} + \frac{R^2}{2dt}(C - Ln\frac{4dt}{R^2} + 1 - W(C - Ln\frac{4dt}{R^2})$$
$$- \frac{R^4}{d^2 t^2}))$$

The diameter of the finite source heater influences
the validity of the above equation in several ways:-

A. The truncation error introduced by the deletion
of the last term is proportional to the fourth power of
the heater radius. Small values of diameter are desireable
from this standpoint.

B. The effect of possible errors in the value of
the thermal capacity ratio, W , is minimized by small
values of the parameter $\frac{R^2}{2dt}$. Therefore, small diameters
are desireable.

C. Smaller values of diameter result in the term,
$Ln(\frac{4dt}{R^2})$, becoming large with respect to the term,
$$\frac{R^2}{2dt}(C - Ln\frac{4dt}{R^2} \ldots$$

This is advantageous.

D. Inherent in the application of the mathematical model to the physical system is the consistency of boundary conditions. Section 4.3 of this report includes a discussion of these boundary conditions. Condition number 6 therein discussed, and the associated Appendix 4, regarding isothermality across the finite source heater and the systematic error introduced, is proportional to the diameter squared.

Condition number 2 therein discussed, and the associated Appendix 2, regarding axial conduction end effects for the internal heat generation by the finite source heater, illustrates that while the error is a complex relationship between several variables, small values of diameter result in less axial conduction.

In conclusion, the smaller the wire diameter, the more precisely the mathematical model describes the physical system.

2. Effect Of Diameter On The Temperature Rise Of The Source Emitter

If two thermal conductivity cells were operated under identical conditions with the same test media, and both were experiencing identical heat fluxes, but differing in diameter, the following would be observed. The cell with the smaller diameter emitter would exhibit a higher temperature at any instant than the cell with the larger emitter. Additionally, the rate of temperature rise would be greater

with the cell of smaller emitter. It could be stated
that the time constant of the thermal system becomes
shorter as the emitter is made smaller.

The consequences of the above on the experimental
system as a whole are:-

A. The time response of the data acquisition system
and that of the thermal system must be compatible.

B. The magnitude of the heat flux generated within
the emitter must fall within certain limits. The accurate
monitoring of very small or very large electrical currents
requires special development and equipment.

C. The temperature rise which the emitter experiences
during a particular test must be limited to a small range.
This is necessary to render insignificant the effects of
possible temperature dependence of the thermal conductivity
of the test medium.

3. Effect Of Diameter On The Mechanical Design
   Of The Thermal Conductivity Cell

During fabrication and assembly of the cell,
difficulties with manipulation, welding and handling of
the source heater increase as the heater diameter is
decreased. When wire less than .005" diameter is utilized,
special jigs and extra precautions are required to assure
a satisfactory heater. It becomes easy to deform the active
length of the heater itself either by excessively straining

it or by the welding of potential leads to the main heater. This results in non-uniform cross section in the wire and erroneous temperature measurements. Each completed heater and potential tap was inspected under a microscope by the writer and a considerable number of prototypes, of .003" diameter platinum were rejected due to excessive cross section deformation.

4. Selection Of The Optimal Length Of Finite Source Heater

The length of the heater must be sufficient to render insignificant the effects of axial heat conduction at the ends of the heater. This problem is discussed in Section 4.3, Boundary Condition 2. Associated with that section was Appendix 2, which detailed calculations and predictions for the temperature distribution at the ends of the heater wire. These showed that for a platinum heater wire of .003" diameter and under typical test conditions, the end effects were not felt beyond one inch from the terminations.

A second consideration was the requirement that the active length of heater wire be determined with a high degree of accuracy. This was completed by mechanical measurement with vernier calipers. The precision with which this could be measured was consequently related to the absolute length of the wire.

Provided the above conditions were met, the length of

of the heater wire could be adjusted to satisfy mechanical
and thermal design factors of the cell and enclosure.

In conclusion, pure platinum wire of approximately
.003" diameter was considered most feasible. The active
length of wire was 6 inches, being satisfactory with respect
to axial heat loss (Appendix 2), and mechanical arrangements
of the enclosure.

5.3.2 Design And Fabrication Of The Thermal
            Conductivity Cell

The design conditions and requirements of the
thermal conductivity cell were these:-

A. The cell must support and enclose the finite
source heater.

B. The cell must contain the test medium but be
thereto inert.

C. The cell dimensions must be of an extent that
the finite source and medium act as an infinite radial
system during the proposed test time limits. However,
the cell dimensions must be limited to reduce natural
convective heat transfer.

D. The cell must provide suitable charging inlets
for charge and discharge of the test medium in the cell.

E. Mechanical requirements for assembly and manuf-
acture must be considered.

F. The cell must be capable of withstanding the
design temperature range.

With due consideration for these factors, a thermal
conductivity cell was designed and tested in part, by the
writer. The final design, which was reached after con-
siderable testing and subsequent modification is shown on
Illustration 5.

The cell consisted of a length of glass tubing, of

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.50 inch inside diameter, closed at the ends by stainless steel plugs. The latter contained the test fluid in the cell, served as structural members for the support of the finite source heater and as terminations for the charging tubes and electrical wiring to the finite source heater.

The inside diameter of the cell was consistent with system requirements regarding wall effects (See Section 4.3) and regarding natural convective heat transfer (See Appendix 3). The wall thickness of the cell was 1/16 inch.

The length of the cell, 10 inches, was set by the required length of finite source heater, previously discussed in Section 5.3.1.

The material of the cell was glass. This was imposed by the necessity of observing the finite source heater during assembly.

The end caps for the cell, were machined from type 304 stainless steel bar. Provision was made in the caps for an "O" ring type seal. The fill and vent tubes were silver soldered into the lower and upper end plug respectively, the tubing being 1/16 inch diameter stainless steel. Ceramic tubes, with two .010 inch diameter clearance holes through their length were positioned in drilled holes on the axis of each end plug. These ceramic tubes were attached in the end plugs, by ceramic cement. As is shown on Illustration 5, the electrical lead wires, of .010 inch diameter platinum, passed

through the ceramic tubes, thence through a pressure
fitting on the pressure vessel-thermostat. The ceramic
tubes also supported the cell and maintained it centrally
within the pressure vessel-thermostat.

A satisfactory method of suspending and tensioning
the finite source heater was developed after some diffic-
ulty. In all, four different methods were attempted. In
the first two, the finite heater was placed under a constant
pretension during assembly of the cell. These two differed
in the method of affixing the ends of the wire in the end
plugs. Both were unsuccessful, due either to differential
expansion between the cell and wire, or to contraction of
the ceramic cement upon setting. The third method utilized
a coiled platinum spring in place of the stainless steel
weight shown on Illustration 5. With the spring, two
problems were found. The first was the difficulty in
assembling the cell and applying a correct pretension to
the spring. The second was that the spring tended to
untwist, throwing the heater wire off the central axis of
the glass cell.

The fourth method and that illustrated, utilized a
stainless steel weight suspended on the end of the heater
wire. The total weight of the stainless cylinder and glass
envelope was 7 grams, which resulted in a constant tension
of approximately 2200 psi within the heater wire. This
magnitude of axial stress was required to free the wire of

any kinks or bends, and was based upon physical experim-
ents carried out by the writer. The glass envelope
surrounding the weight and attached to it, provided insul-
ation between the potential lead and the stainless weight.

As is obvious from the Illustration 5, the glass
cell could not withstand a significant pressure difference
across the end caps.

This was not necessary, since the pressure vessel-
thermostat exposed the cell to the same operating pressure,
both internally and externally, since the top fill tube
(See Illustration 3) exhausted into the pressure vessel
cavity. The cavity was backcharged with nitrogen gas.

Two "O" rings, see component (3) Illustration 5,
were placed on the outside of the glass enclosure, and
served a function only during assembly of the cell into
the pressure vessel thermostat. The outside diameter of
these "O" rings was less than the diameter of the cavity
in the pressure vessel, and did not offer any resistance
to gas flow between the ends of the pressure vessel cavity.

The "O" ring seals used in the cell could tolerate
an environment of up to 600 Deg. Fah. indefinitely, and
temperatures in excess of that for short periods. Illustrat-
ion 6 shows the arrangement of the finite source heater
used for testing complete with potential leads, and tension-
ing weight.

In total, 7 heaters were fabricated, with the approx-
imate dimensions of the above. Of these, two were rejected

before assembly in the cell, since **visible** deformation
of the active section of the heater wire was observed under
microscopic examination. This occurred in the region of
the potential tap weld. Three heater wires were broken
during cell assembly, due to lack of dexterity on the part
of the writer. One was successfully assembled into the
cell and thermostat, but broke at the potential lead weld
after a one month period. The seventh heater was installed
and successfully withstood 8 months of intermittent testing.

The potential taps and active length of heater wire
consisted of .0025 inch diameter platinum wire, reference
grade, supplied by the Sigmund Cohn Corporation. It was
received and installed in the hard drawn condition.

The welds of the potential leads and .010 inch
diameter current leads were made by spot welding the mat-
erials together, using a capacitive discharge type welder.
The Northern Electric Company, London, Ontario, kindly
completed this work for the writer, gratis.

After a microscopic examination of the heater, the
tensioning weight was attached to the one end of the heater
wire.

The tensioning weight, Illustration 6, consisted of
a small stainless steel cylinder. Through the axis of
this cylinder, a section of hypodermic tubing, .010 inch
inside diameter was inserted and silver soldered. The
current lead was drawn through the hypodermic tubing,
which was thereupon gently crimped.

After assembly of the heater wire within the cell, the active length between the potential taps was measured. This was completed by mounting the cell and heater wire vertically on a surface table. A vernier height gauge, with microscopic eyepiece attached, was adjusted to the level of the topmost potential tap. The vernier height gauge was then lowered to the bottom potential tap, and the difference in the two elevations recorded. This difference represented the active length between the potential taps, while the heater wire was under the operating tension. Independent measurements were completed by the writer and by one of the Engineering Faculty machine shop technicians.

Upon assembly of the completed cell into the pressure vessel-thermostat, the finite source heater was given an annealing heat treatment, by heating the wire by electrical current to a temperature of 800° Fah. It was maintained at this temperature for two hours. The temperature was determined by resistivity measurements of the heater wire.

5.3.3 Design And Fabrication Of The Pressure

Vessel-Thermostat

The design conditions and requirements of this
apparatus were these:-

A. The apparatus must contain the thermal
conductivity cell and further must provide the environ-
ment for the test cell. The range of design temperature
and pressure excursions was 0 to 3000 psi, 70° to 720°
Fah. respectively.

B. The apparatus must provide adequate sealing
penetrations for the charge tubes and electrical wiring.

C. The apparatus and the thermal conductivity cell
must be consistent with respect to assembly.

The writer designed and tested a pressure vessel-
thermostat with due consideration for these factors.

Illustration 7 shows the complete pressure vessel-
thermostat and its' accessories, consisting of the follow-
ing items:-

A. A copper pressure vessel, 3 inches outside
diameter by 14 inches long.

B. A light sheet metal container, cylindrical,
18 inches diameter by 30 inches long. This served to
contain a layer of diatameceous earth around the pressure
vessel.

C. A temperature control system consisting of an

automatic temperature controller, associated thermo-
couple feedback sensors and electrical resistance heaters.

D. A variable pressure supply system to pressurize
the pressure vessel and thermal conductivity cell to the
required test pressure.

The pressure vessel consisted of a length of solid
100% conductivity electolytic tough pitch copper which
was machined to suit. Information regarding the physical
and chemical properties of this material was obtained
from Mr. S. Gawley, metallurgist, Wolverine Tube Ltd.,
London, Ontario. For the material specifications, see
Item 21, P. XI .

The internal dimensions of the cavity were fixed
by the geometry of the thermal conductivity cell, while
the remaining dimensions were determined by stress level
and temperature distribution conditions.

The copper end caps were sized to provide a nominal
working stress of 7000 psi at 3000 psi internal working
pressure. This was determined by assuming that the disc
type end covers were edge supported, and by considering
that the disc experienced the 3000 psi internal pressure
over a central 1.750 inch diameter section. This analysis
was conservative. The stainless steel socket head cap-
screws used to bolt the end caps to the pressure vessel
body operated at a nominal 5000 psi under 3000 psi internal
pressure. The relative thermal expansion of the copper
end caps and the stainless steel capscrews was determined

for the design temperature range. In order to maintain
metal to metal contact between the end caps and the main
body at 720° Fah., it was necessary to tension the cap-
screws to 7000 - 8000 psi tensile stress at ambient
temperature. The end cap and pressure vessel body were
sealed against leakage by an "O" ring joint. Upon assembly
and testing, the pressure vessel and end caps successfully
withstood the design pressure limits without leakage or
excessive deformation.

Pure copper, of which the pressure vessel was
constructed, exhibits surface oxidation at high temper-
atures. To exceed 1000° Fah. for any significant time
with the vessel in the presence of oxygen would result in
a scale formation. At 750° Fah. little surface scaling
would be expected. Scaling at the outside of the pressure
vessel would cause no problems. However, the inner cavity
would be best maintained oxidation-free, since test sample
contamination could occur under some conditions. Two
approaches were used to reduce this scaling. First, the
cavity was charged at all times with nitrogen or argon
gas, reducing the oxygen content of the atmosphere to the
small amount introduced by trace water vapor. Second,
the inner cavity of the pressure vessel and the cavity
side of the end caps were gold plated using an **immersion**
gold plating technique. (See Item 22, P. **XI**). Observat-
ion of the pressure vessel after several months usage

including some operation at 600° Fah. showed no scaling
on the inner cavity, and minor scaling at the outside
surfaces.

The pressure vessel-thermostat was designed to
provide as close to isothermal conditions as practicable
along its length. This condition was essential to the
proper operation of the thermal conductivity cell, and
was, in fact, inherent in the mathematical model boundary
conditions. To produce this approximation to isothermal-
ity, additional input heat flux was required at the ends
of the pressure vessel to compensate for the correspond-
ing heat loss to the insulation from the end caps. An
analysis was completed by the writer to determine the
proper electrical heating input as a function of distance
from the centre of the vessel. Fortunately, Laubitz (70)
at the National Research Council, Ottawa, had also con-
sidered the same problem extensively, and by following
his documented method, the writer determined the required
heat flux distribution which minimized the non-isothermality
of the axial temperature gradient. The reader is referred
to Appendix 7 for the numerical analysis of Laubitz's method
as applied to the writer's system.

In accordance with these numerical conclusions, five
separate heater circuits were incorporated on the pressure
vessel outer surface. As is shown in Illustration 7, the
pressure vessel was machined to provide three spiral grooves

on the outside surface. These were pitched at 1/4 inch. One heater was wrapped into one groove, and extended over the entire length of the vessel, consisting of 16 turns. Two end heaters consisting of 1 turn each were placed in an adjacent groove. Finally the last two heaters were wrapped into the remaining groove, extending 6 turns from each end of the pressure vessel.

By properly proportioning the current flow through the above five heaters, the net heat flux could be adjusted to compensate end losses. This made possible the attaining of a 10 inch long central section of the pressure vessel which was essentially free of an axial temperature gradient.

The selection of a satisfactory heater wire material and the method of attachment of same, necessitated some experimental development. The first method utilized "Fiberglass" insulated resistance wire (Item 23, P.**XI**) embedded in ceramic cement in the machined grooves on the pressure vessel. This method was unsatisfactory due to the difficulty in maintaining good electrical isolation between the heater and the copper pressure vessel. Several heater short circuits were experienced. After further consideration, the problem was successfully solved by using as heater wires, "Inconel" sheathed resistance wire, the sheath being 1/16" diameter (Item 24, P. **XII**). This was held in the machined grooves by peening the copper on the side of the groove over the sheath of the heater wire. This

arrangement presented no further operational difficulties.

In order to verify the thermal performance of the pressure vessel, it was instrumented with thermocouples which permitted the measurement of the temperatures. Appendix 6 shows the arrangement of the thermocouples, consisting of 13 pairs of differential and 4 absolute thermocouples on the body of the pressure vessel itself. All thermocouples consisted of 30 gauge wire, double "Fiberglass" insulation, and met type T T specifications (See Item 6, P. XI). The junctions were formed by twisting and silver soldering. Twenty four differential thermocouple pairs were intercompared by exposing simultaneously all thermocouples to a controlled temperature difference in an ad hoc laboratory furnace.

The average response for the 24 thermocouples was determined, and the 13 which departed least from this average were installed on the pressure vessel.

The pressure vessel-thermostat was prepared for the thermocouples by drilling 3/32 inch diameter holes, 1/4 inch deep in the outside of the pressure vessel. The junctions were embedded in these holes with ceramic cement. All differential thermocouple leads to the recording instruments consisted of copper wire, to minimize spurious voltages due to thermocouple wire inhomogeneity. The position of thermocouples on the pressure vessel is shown on Illustration 8.

A layer of insulation around the pressure vessel formed an important part of the thermostat, being essential in the control of the total heat loss from the pressure vessel. The thickness and thermal coefficient of this insulation layer formed part of the input of data required in determining the optimal heating power distribution for the pressure vessel (See Appendix 7).

A light gauge sheet metal cylinder was procured 18 inch diameter, 30 inches long. This was mounted centrally around the pressure vessel, on a common base. The annular space was filled with insulation material.

Two types of insulation were used. Initially, a granulated expanded asbestos insulation was tried. It was evident that this material did not flow sufficiently freely, leaving voids around and under the pressure vessel. Finally, diatomaceous earth, fine grade, was procured. This was dried in a furnace at 250 °Fah. prior to use, to drive off any moisture, since this powder is delaquescent to some extent. When installing this powder in the thermostat, the material flowed freely, and with gentle rapping of the enclosure, the material filled all interstices.

An automatic temperature controller (Item 25), was provided to control the temperature of the pressure vessel. The details of the temperature control system are shown on Illustration 4.

The system required to pressurize the cavity of the pressure vessel and the test fluid in the thermal

conductivity cell is shown on Illustration 4.

CHAPTER 6

EXPERIMENTAL TECHNIQUE AND INSTRUMENT CALIBRATIONS

6.1 Preliminary Testing Of Insulating Materials

The equipment utilized has been described in Section 5.1 of this report.

Illustration 1 shows the arrangement of the cell and instrumentation.

The heater wire, suspended in its support frame and including the active junction of the absolute thermo-couple pair was installed in the glass container. The granulated insulation material, "Vermiculite" was poured into the vessel until full. No attempt was made to compress the insulation, but the container was lightly tapped by hand to insure that no significant voids existed in the body of insulation.

Before any tests were made, the charged container was left to reach thermal equilibrium with the room. No less than 12 hours elapsed between charging and testing or between successive tests. After this length of time, the voltage from the differential thermocouple was observed, and the resultant temperature compared to the laboratory temperature. Correspondence between the two temperatures indicated that thermal equilibrium had been reached.

Just before a test, the regulated power supply was

adjusted to provide the desired electrical output, it being connected to a resistor at this time. It was then turned off and connected to the heater wire.

To initiate a test, the power supply and electrical timer was simultaneously energized. As quickly as possible, the potentiometer was adjusted to track the rising voltage of the thermocouple. In the initial seconds, this was difficult due to the rapidly rising voltage, but at longer times, it was possible to follow the voltage accurately. The time, and thermocouple voltage were manually simultaneously recorded, as well as the current flow to the heater, the latter at less frequent intervals, since the current flow experienced very small change throughout a test.

A complete test normally occupied 10 to 15 minutes.

The reduction of data and the experimental results are presented in Section 7.1.

6.2 Preliminary Experiments With Fluids

During this phase of the experimental programme, the following tests were made:-

1. Test numbers 67-1 to 67-28 were performed, measurement of the thermal conductivity of air.

2. Test numbers 67-29 to 67-84 were performed, measurement of the thermal conductivity of water.

Thermal conductivity cells A through D were used (see Section 5.2.1) and two electrical readout systems (also described in Section 5.2.1).

The experimental procedure used with any of the above cells A to D, and with the bridge network (refer to Illustration 2) was as follows:-

Initial Calibration -

1. Temporarily increase the value of the dropping resistor by eleven times. This resulted in a 100 fold decrease in the heat input to the finite line source heater.

2. Temporarily connect a galvanometer across the bridge diagonal.

3. Adjust bridge arm 3 for a galvanometer null.

4. Restore dropping resistor to original value.

5. Connect chart recorder in place of galvanometer.

Testing Procedure -

1. Charge cell with test sample.

2. Adjust chart recorder offset to zero.

3. Energize chart feed.

4. Energize power supply to bridge.

5. Record current flow through standard shunt.

6. Readjust dropping resistor and repeat above
steps for test data with different finite source strengths.

A subsequent modification as shown on Illustration 2
included placing the standard shunt in Arm 1 of the bridge.
In this case, the voltage across the shunt was monitored
by channel #2 of the chart recorder.

The procedure used with the potential comparing
network was developed and modified as successive tests were
undertaken. At the termination of the preliminary testing,
the procedure was essentially identical to that used during
the final testing programme. The latter is presented in
detail in Section 6.3.3 under "Test Procedure".

Associated Calibrations And Equipment Checks

Chart Recorder (Item 16, P. XI)

The chart recorder was checked out in accordance
with the manufacturer's instruction manual on several
occasions, and a spot check of the amplifier gain was
made normally each day tests were undertaken. This was
achieved by injecting known microvolt signals from a
microvolt potentiometer (Item 26).

The time base was checked by running the chart paper

through for a time interval, this time interval being
determined by a stopwatch and in some cases by an
electrically powered timer.

At times, a time error approaching 1.25% was
observed. Gain stability of the recorder was improved
by energizing the amplifier power supplies from an
external regulated power supply. Furthermore, the recorder
amplifiers were left continually energized between tests.

Measurement Of The Bridge Resistor Resistances

The bridge resistors resistance in arms 1, 3, and
4 including their associated bridge connections, was
measured using the potential comparator technique,
(Items 1, 2, 3, 4, P. XI). This equipment permitted
resolution to one milliohm or ten milliohm, depending on
range. These measurements were completed with all resistor
components at laboratory ambient temperature.

Check Of Storage Battery Discharge Characteristics

Preliminary testing indicated that the electrical
power supply (Item 9, P. XI), was not sufficiently stable.
Consequently a bank of 6 volt 100 ampere hour (eight in
all) lead-acid storage batteries was procured and used
for all testing. The ampere hour capacity of each battery
was checked on six month intervals. This was determined
by connecting in series with the battery, a 10 ampere
shunt and a load resistance. The current flow and battery
voltage was recorded on a two channel chart recorder.

These results were then integrated numerically with respect to time, from which the ampere hour battery capacity was determined. This test was normally completed with a 10 ampere discharge rate, and served to indicate the gross battery condition and battery deterioration with age. Appendix 8 presents in detail the results of battery testing.

The other test performed on the storage batteries consisted of recording the battery voltage characteristics under a constant small (approximately 100 millampere) discharge. This voltage was recorded over a period of several days, and indicated that after an initial rapid voltage drop, a relatively constant battery voltage could be obtained for long periods.

During thermal conductivity testing, the batteries were operated in this region of constant voltage.

Purity of Test Sample

During tests of water, the resistivity of the sample was determined using a conductivity meter (Item 27, P. **XII**). Triple distilled water was utilized, and the resistivity exceeded two megohm centimeter.

Finite Heater Resistance

Periodic checks were made on the total electrical resistance of the heater wire in the thermal conductivity cell. This would indicate deterioration of the wire

through corrosion.

Check Of The Time Before Wall Reflection Effects

Each test cell and fluid sample was exposed to an initial long time test. This test served to establish the time limits within which subsequent tests could be performed without experiencing the wall effects of the cell. (See Section 4.3, boundary condition 1)

Observation Of The Onset Of Natural Convection

In some cases, when completing tests with large source heat strengths, it was possible to observe in the experimental data, the effects of natural convective heat transfer in the thermal conductivity cell. Subsequent tests were then completed at lower source heat strengths. These results will be discussed in greater detail in Chapter 7.

6.3 Experiments With Fluids To A Higher Accuracy

In this final phase of the experimental programme, the following tests were undertaken (refer to Appendix 10 for a complete listing of experimental tests):-

1. Thermal conductivity of water:- Tests 69-28, 69-30 to 69-36, 70-1 to 70-15, 70-17 to 70-24, 49 tests in all.

2. Thermal conductivity of alcohol:- Tests 70-45 to 70-49, 4 tests in all.

A procedure for assembly of the system was established, and necessary calibration tests for the associated equipment was completed.

6.3.1 Assembly Of System And Components

Finite Source Heater - Illustration 6

1. Using sample pieces of platinum wire of the same diameters as the finite heater, potential taps and power leads, a series (approximately 20) of test welds were made for each type of joint. The power setting and electrode pressure of the spot welder were systematically varied. Each test weld was first observed under a microscope for deformation of the respective wires, and then pulled until the weld ruptured. By this means, it was possible to optimize the weld joint with respect to welder variables. These optimum settings were recorded.

2. A length of platinum wire of the proper diameter was laid out, inspected for surface imperfections and kinks.

3. The .010 inch diameter platinum current leads were spot welded to the ends of the finite source heater. These current leads were left approximately 18 inches long.

4. The heater was taped to a sheet of white paper, and two pencil marks laid down at the points of desired potential lead attachment.

5. A small hole was cut in the paper beside the pencil marks.

6. The fine wire potential leads were laid across

the source heater at the pencil marks, and taped.

7. The potential tap leads, .0025 inch diameter, were welded to the finite source heater, and one end of each potential lead cut off at the heater.

8. The completed heater was subjected to a microscopic examination.

9. The stainless steel tensioning weight was pulled over one current lead, and the stainless hypodermic tubing crimped unto the current lead.

10. The completed heater wire and weight were suspended from a retort stand and the active length of heater wire measured, as described in Section 5.3.2.

Assembly Of Thermal Conductivity Cell - Illustration 5

1. The glass enclosure was cut to length and the ends of this tubing ground square.

2. The source heater wire was drawn through the glass tubing, the latter being positioned horizontally.

3. Both end plugs were prepared for assembly by cementing the porcelain insulators into the end plugs with ceramic cement.

4. Immediately before assembly, the sections of the electrical leads which would fall within the porcelain insulators, were painted with ceramic cement.

5. The platinum lead wires were drawn through the insulators, and simultaneously the end plugs were eased into the glass tube.

6. Additional ceramic cement was worked in beside the lead wires.

7. The cell was positioned vertically, and the heater wire inspected for position and condition within the cell.

8. The cement was permitted to harden.

Assembly Of The Cell Into The Pressure Vessel - Thermostat
Illustrations 4 and 7

1. The pressure vessel was positioned horizontally.

2. The thermal conductivity cell was inserted into the cavity from the end of the pressure vessel which would eventually be the bottom end.

3. The "O" ring seal and fittings were installed on the bottom end plate.

4. The bottom end plate was installed, drawing the electrical leads and fill tubing through their respective fittings in the end plate.

5. The pressure vessel was positioned vertically on the pressure vessel-thermostat support.

6. The top plate was installed, the electrical leads being directed through their respective fittings.

7. The top and bottom plates were secured with cap-screws, and the pressure fittings drawn up.

Preliminary Testing Before Filling With Insulation

Before the pressure vessel was surrounded with insulation, the following tests were completed after elect-

rical and piping connections were made:-

1. All thermocouples were tested for response and lead identification by touching the pressure vessel at or near the active junction with a soldering iron and noting the response on a chart recorder.

2. All heaters and thermocouples were tested for ground isolation.

3. A pressure test was completed on the system.

4. The pressure vessel and support was plumbed with a spirit level.

Installation Of Insulation

1. After the proper operation of all instrumentation was verified, the metal container was placed concentrically around the pressure vessel.

2. Sufficient insulation powder (Item 28, P. **XII**) was placed into a laboratory furnace and brought to 250° Fah. to drive off moisture, and soaked at 250° Fah. for 24 hours.

3. The insulation was then poured into the sheet metal enclosure, tapping the support framework at the same time to ensure the removal of all voids around and under the pressure vessel.

6.3.2 Calibration Of Instruments And Apparatus Test

     Temperature Controller Thermostat

     A series of tests were conducted on the automatic
temperature controller when connected to the pressure
vessel-thermostat, to establish the stability of the system,
and to determine the magnitude of the temperature fluctuat-
ions. These were tests number 70-16A, 16B, and 70-53A.

     For the first test, the temperature controller
(Item 25, P. XII) was isolated from the system. A load
resistor of 100 ohms was connected across the output and
a microvolt potentiometer connected to the input. With
the proportional band control of the controller at the
highest sensitivity setting, it was found that the instru-
ment was sensitive to 1/10 of a microvolt. The maximum
voltage gain obtained under these test conditions was 2.40
x 10^6, where voltage gain was defined in this case as the
change in output voltage across the load resistor/change
in voltage at the input. The manufacturer specified a
sensitivity of 1 microvolt for industrial conditions.

     Under the laboratory conditions for the tests under-
taken by the writer, it would be expected that the temper-
ature controller would meet or exceed the manufacturer's
specifications.

     A later test for the temperature control system

involved the monitoring of temperature of the pressure
vessel-thermostat by one of the absolute thermocouples
installed on the pressure vessel, while the automatic
temperature control system was in operation. A detailed
closed loop control system analysis was not made, but
from the experimental results, the following conclusions
were obtained. At a control temperature of 182° Fah.,
the system reached a steady temperature after 230 minutes
from initiation of power experiencing damped oscillations
up to that time. From 230 to 246 minutes, the temperature
of the thermostat as monitored by the absolute thermocouple
on the pressure vessel, varied by $\pm$ 1/10 of a microvolt,
oscillating by this magnitude with a period of approximately
10 minutes, representing approximately $\pm$ .003 ^{o}Fah.

In order to achieve this order of stability, two
modifications were completed on the temperature controller.
Firstly, the built-in temperature compensating resistor
which was used to provide a thermocouple reference temper-
ature of 32 ^{o} Fah. at the controller was disarmed. An
icebath (Item 5, P. **XI**) and an additional thermocouple
pair was used to provide this function. A second modific-
ation was the energizing of the temperature controller
by a regulated power supply.

Measurement Of Temperature Variations Of The Laboratory

There were indications that the room ambient temper-
ature was varying to a considerable extent, and the writer

undertook a test to verify or show otherwise the adequacy of the laboratory temperature control system. (See Test 69-14).

A differential thermocouple pair, meeting Type TT specifications (Copper-Constantan) was fabricated. One junction was placed in the vicinity of the laboratory temperature control thermostat, the other in a precision icebath (Item 5, P. **XI**). In series with the differential thermocouple pair, in the copper side, was connected a potentiometer (Item 7, P. **XI**) and a chart recorder (Item 16, P. **XI**). The potentiometer was set at 1 milli-volt, the recorder at 200 microvolts full scale. For part of the test, an electric fan was set up to direct a flow of air over the thermocouple junction and the room thermo-stat.

With the fan on, a variation of $\pm$ 70 microvolts was observed, corresponding to $\pm$ 3.08° Fah. The period of oscillation varied from 7 to 8.5 minutes. With the fan off, a variation of $\pm$ 39 microvolts was observed, corresponding to $\pm$ 1.72° Fah. The period of oscillation varied from 17 to 18.2 minutes.

The system guarantee for the subject temperature control system specified $\pm$ 1° Fah., and consequently was not met. The position of the thermostat controller at the side of the room was not optimum, and it could better sense the average room temperature if placed closer to the centre of the room space.

Ground Isolation - Thermocouples And Heaters On

Pressure Vessel

Before charging the system with the test fluid,
a resistance check of all electrical leads to detect
shorting and faulty insulation was made. (See test 69-2A).

The finite source heater showed greater than
20,000,000 ohms to ground. The electrical heaters on
the pressure vessel showed from 200,000 ohms to greater
than 20,000,000 ohms, depending on which of the five
heaters were checked.

All thermocouples to ground showed better than
500,000 ohms.

This measurement was completed using a laboratory
type multimeter.

Pressure Test Of Pressure Vessel

Two pressure tests were made on the apparatus. The
first was conducted on the pressure vessel only, and
consisted of a basic hydrostatic test to check structural
integrity. It was performed by completely filling the
pressure vessel with distilled water, connecting to one
aperture a 3,000 p.s.i. bourdon type gauge, and sealing
all other apertures. The electrical heaters were energized,
and the vessel raised in temperature. Due to the expansion
of the water with temperature, the pressure within the
cell increased. 3,000 p.s.i. was observed on the pressure

gauge with the pressure vessel at approximately 150° Fah. No leaks or deformations were evident.

After all pressure connections had been completed, a pressure test was made on the complete system. The system was charged with nitrogen gas to a pressure of 300 p.s.i. After sealing the charging connections, but leaving a bourdon type 10" dial gauge in the charging line, it was observed that less than 1 p.s.i. pressure drop occurred after 72 hours.

Electrical Noise Of System

Considerable difficulty had been experienced during the preliminary testing with electrical noise on the system instruments. For this final phase of the experimental programme, greater attention was given to the problem.

When the first test run was made with the subject system, electrical noise voltages of $\pm$ 10 microvolts was present at the chart recorder terminals. Following investigation of noise sources, and remedial actions taken, this noise voltage was reduced to approximately $\pm$ 1 microvolt.

The steps taken to effect this reduction were:-

1. A #8 gauge copper wire was connected directly to a laboratory cold water pipe, to be used as a ground wire bus.

2. The pressure vessel, potentiometer ground

connections and chart recorder grounds were connected to the above bus.

3. The interconnecting signal wires were replaced by shielded conductor, the shields being connected to a common signal ground on the chart recorder.

4. All shielded conductor shields and potentiometer shields were connected to the chart recorder shield terminal.

5. One potential lead from the finite source heater was grounded at the chart recorder terminal to the copper ground bus.

6. The chart recorder amplifiers were energized through an isolating transformer, the electrostatic shield of which was grounded to the copper bus.

7. The wet cell storage batteries used as the power supply for the finite source heater, were set on electrical insulators to effectively break any electrical ground loops.

8. All electrical conductors carrying direct current potentials were securely strapped to the tables and associated electrical equipment to prevent their movement in the vicinity of the galvanometer magnetic fields.

Resistance And Power Tests For The Inshop Power Resistors

The two special oil bath resistors described in Section 4.2.2 (Item 17 and 18) were subjected to a resistance test, Test 69-1.

The first resistor, Item 17, consisted of a multitap resistance string, total resistance being 412.2478 ohms at 23.1° Cent. There were 14 steps in all, the minimum being .2187 ohms, the maximum being 200.7809 ohms. All taps ended at copper binding posts.

The resistance between each tap was determined by connecting in series with the total resistor, a 100 ohm standard resistor (Item 29) and a six volt wet cell storage battery. The current flow resulting was approximately 120 milliamperes.

A potentiometer (Item 7) and a voltbox (Item 30), when necessary, were used to monitor the voltage across the standard resistor and the multitap resistor.

Since the battery voltage varied with time, it was necessary to monitor the voltage across the standard resistor between each successive measurement of voltage across the resistor taps. A recording of voltage was made each minute, to 1 microvolt resolution. It was observed that the current decreased by approximately 4 microampere per minute after the circuit had been connected for one hour.

By interpolating time intervals and comparing voltages, it was possible to determine the voltage ratio between the standard resistor and the intertap resistance, and consequently to determine the resistance between taps.

The second resistor (Item 18) consisted of six resistors separated electrically but connected thermally

in one oil bath.

These resistors were temporarily connected in series, and the same approach as above used for resistance determinations.

The resistance values so determined for both resistors are presented in Appendix 9.

Testing Of The Wet Cell Storage Battery Power Supply

The reader is referred to Section 6.2, P. 122 , and Appendix 8 for a discussion of the testing method and the results thereby obtained.

Adjustment And Calibration Of The Associated
System Components

The chart recorder, potentiometers and accessories which formed part of the writer's system were subjected to adjustment and calibration in accordance with the respective manufacturer's instruction manual.

The standard cells used in the potentiometers were intercompared frequently (daily during testing) with the #6 cell of the laboratory bank of standard cells (Item 31).

Two of the portable laboratory cells were found to have deteriorated with age, and two new standard cells were purchased for their replacement (Items 32, 33).

It was observed that a daily variation of up to two microvolts could be expected in the case of the portable standard cells.

Test Sample Purity

The water test samples used in charging the system prior to test was subjected to a resistivity check using a conductivity meter (Item 27). Resistivity at charging normally exceeded 2 megohms cm. After testing in the cell for periods of up to a week, the test sample, when retested, showed a decrease in resistivity, down to 1 megohm cm in the worst case. This indicated the sample was being contaminated to some extent while in the cell.

No checks of chemical purity were made.

Temperature Distribution Along Pressure Vessel-Thermostat

Several tests were conducted to investigate the thermal performance of the pressure vessel-thermostat with respect to axial temperature gradient, tests 70-16B and 68-8 respectively.

While only preliminary investigations were completed, at low enclosure temperatures, indications were that the apparatus was so constructed and instrumented that a region of 80% of the length of the enclosure could be maintained with less than $1/20^{\circ}$ Fah. temperature difference.

Test 70-16B was carried out with the pressure vessel at 200° Fah. Two end heaters and the full length heater was energized. After standing at equilibrium for 24 hours,

the differential thermocouples indicated a maximum

end to centre voltage difference of 1.2 microvolts.

Of the 13 pairs of thermocouples, all but two fell

within .5 microvolt of the straight line joining the

centre and end microvolt value. The remaining two fell

.75 and 1.1 microvolt from the subject line.

At 200° Fah. the value 1.2 microvolt represents

.046° Fah. or 1/22 of one degree.

A graphical presentation of the temperature

distribution along the furnace and further details

are presented in Appendix 6.

Phasing And Adjustment Of The Control Relays

In The Electrical Network

A schematic diagram of the electrical relays

required in the electrical network of the thermal

conductivity system is shown on Illustration 8, P.   .

Upon closing the master switch S1, the chart drive

of the recorder was started, and furthermore Relay 1

circuit was energized. Relay 1 was delayed in closing

by the resistance-capacitance circuit RC1. This permitted

the chart drive motor to accelerate to full speed.

After 50 milliseconds, relay 1 closed. Relay

contacts R1-2 closed, connecting Channel 2 of the chart

recorder in series with potentiometer 2 and the potential

leads of the thermal conductivity cell. When this relay

was de-energized, the input terminals of Channel 2 were

shorted, facilitating zero adjustment of the pen.

As relay Rl was energized, contacts Rl-2 also
closed which energized the circuit of Relay 2. Relay 2
was furthermore delayed in closing by resistance-
capacitance circuit RC2. This delay was necessary to
permit the contacts of Rl-2 to settle.

After approximately 10 milliseconds, relay 2 closed,
which energized the finite source heater from the battery
power supply, simultaneously removing the equivalent load
resistor from the battery circuit. This latter resistance
was variable and was adjusted to the same total resistance
as the finite source heater circuit, and consequently a
uniform current flow from the battery supply existed at
all times.

The above resistance-capacitance delay circuits
were adjusted experimentally to minimize the spurious
responses on the chart recorder channel 2.

The method used by the writer consisted of monitor-
ing the voltages across channel 2 at the time of switching
with an oscilloscope and identifying, if possible, the
source of the transient voltage. Appropriate adjustments
of the delay circuits were then made to minimize the
transients.

6.3.3 Test Procedure

Procedure To Charge The Thermal Conductivity Cell

Before the cell was charged with the test sample, the apparatus was flushed by methyl hydrate, then with argon gas.

The cleaning procedure was as follows:-

(Refer to Illustration 3)

1. The head tank was filled with methyl hydrate to the level of the upper outlet. After waiting 5 minutes, this was refilled again, as the liquid filled the thermal conductivity cell.

2. The liquid was allowed to stand within the cell 1/2 hour.

3. The head tank cover was removed, and both drain valves opened.

4. The process above was repeated three times.

5. The head tank cover was replaced, and the lower drain valve opened.

6. The argon gas supply valve was opened to provide 2 to 3 p.s.i.g. pressure. The valve between the top head tank connection and the argon supply was closed. This resulted in a flow of gas through the cell, it being permitted to flow 10 to 20 minutes.

7. A sample of the test medium was charged and

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discharged twice.

Following the cleaning procedure, the test sample was finally charged and used for testing.

Test Procedure

Before tests were conducted each day, a series of preliminary checks were made. These included:-

1. Check of the chart recorder gain and span on both channels, by introducing known voltages from a microvolt potentiometer.

2. Cross standardize potentiometer 1 and 2 (see Illustration 3) to each other and to laboratory standard cell #6 (Item 31).

3. Adjust and check voltage of relay power supplies (100 VDC and 1.35 VDC - Illustration 8).

4. Monitor the current flow through the finite source heater circuit with the master switch in the off postion (the constant battery current flowing through the equivalent load resistor in this position).

5. The level of test medium in the level tank was observed (see Illustration 3).

6. Rapidly switch the master switch "on" and "off" once to verify the correct adjustment of the equivalent load resistance.

To conduct a test, the following steps were followed:-

1. The paper feed on the chart recorder was manually advanced to a particular position, previously determined,

such that the start of a test would correspond with the accented lines of the chart paper.

2. Energize the potentiometers.

3. Reset chart recorder to zero on both channels.

4. Switch "on" the master switch.

5. Switch "off" the master switch after 10 to 20 seconds.

6. From observation of the test results, potentiometer 1 and 2 were readjusted to maintain a full scale response on the chart recorder.

7. Repeat 1 to 6 for next test.

In all cases, the chart recorder was used at maximum gain of 100 microvolts full scale and maximum speed of 1 inch/second, both channels.

In order to change the source heat flux, it was necessary to readjust the current adjusting resistor (see Illustration 8) to a higher or lower value. Following the adjustment, no further tests were made for 12 hours, in order to permit the battery power supply voltage to stabilize.

The majority of the writer's tests were conducted with the cell at ambient room temperature, without the temperature control system energized. Observation of the temperature of the pressure vessel was completed by monitoring an absolute thermocouple on it, using a potentiometer. For those tests completed above ambient, the temperature control system was energized and the system allowed to

reach equilibrium over a 24 hour period before
tests were initiated.

CHAPTER 7

EXPERIMENTAL RESULTS AND DATA REDUCTION

7.1 Tests With Insulation Material

Under this programme, eight tests in all were conducted, numbers 66-1 to 66-8. For the first four, 66-1 to 66-4, respectively, the analysis of the data was not completed since, for these preliminary tests, the current flow was monitored only by the panel meter associated with the power supply, and current determinations were consequently insufficiently accurate. These tests did serve to establish a range of heating currents which yielded a thermocouple response consistent with the readout apparatus.

Tests 66-5 to 66-8 were then completed but for these determinations a standard shunt was inserted in the heating current circuit, permitting a potentiometric current measurement, this resulting in current determinations to a satisfactory order of accuracy.

The analysis of the data obtained was completed using the line source mathematical model, described and presented in Section 4.2.

Equation 9, Section 4.2 is reproduced below:-

$$K = \frac{q}{4\pi K} \frac{\cdot Ln(t_2/t_1)}{(T(R_0,t_2) - T(R_0,t_1))} \tag{12}$$

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q may be expressed in terms of the electrical power input and the length L of the wire.

$$q = 3.414 \times \frac{R}{L} \times I^2 \qquad (13)$$

Where R = the resistance of wire, length L ohms

L = length of wire, feet

Illustration 9 shows the millivolt-time response of test 66-5, plotted to a semi-log scale. The test data falls on or close to the straight line thereupon drawn. This line was best fitted by the writer by adjusting a straight edge until a maximum of the data points were crossed.

One may obtain at the 100 and 1000 second interval, two millivolt values, which can be converted to temperatures by applying the proper conversion for the thermocouple material.

Since the overall temperature excusion during a test was small, it was reasonable to apply an average millivolt-to-temperature conversion over the range.

Therefore:-

$$T_2 - T_1 = \frac{MV_2 - MV_1}{.023} \qquad (14)$$

Where MV_2, MV_1 =the millivolt values at time t_2, t_1 respectively.

.023 =an average millivolt-degrees Fahrenheit conversion for copper constantan thermocouples in the $70°$ to $150°$ Fah. range.

Substituting equations 14 and 13 into 12, one obtains-

$$K = 3 \cdot 414 \times \frac{R}{L} \times I^2 \times \frac{Ln\ (t_2/t_1)}{(MV_2 - MV_1)} \times \cdot 023 \qquad (15)$$

For test 66-5, the data below is valid:-

L	$= 10 \cdot 16/12$	feet
R	$= 4 \cdot 20179$	ohms, room temperature
I	$= \cdot 4692$	amperes
MV_1	$= 1 \cdot 080$	millivolts
MV_2	$= 1 \cdot 465$	millivolts
t_1	$= 100$	seconds
t_2	$= 1000$	seconds

Substitution into equation 15 yields:-

$$K = \cdot 0409 \qquad \text{BTU/Hr.Ft.Deg.Fah.}$$

For additonal tests using the same apparatus, equation 15 can be shown to yield:-

$$K = \cdot 07161 \frac{I^2}{MV_2 - MV_1}$$

Where MV_1, MV_2 are taken at the 100 and 1000 second intervals.

Analysis of the remainder of the tests showed:-

Test 66-6   K $= \cdot 0519$ BTU/Hr.Ft.Deg.Fah.

Test 66-7   K $= \cdot 0478$ BTU/Hr.Ft.Deg.Fah.

Test 66-8   K $= \cdot 0425$ BTU/Hr.Ft.Deg.Fah.

Gebhart (71) showed a value of thermal conductivity of .045 BTU/Hr.Ft.Deg.Fah. for "Vermiculite" at $150°$ Fah., 9 pounds/cubic foot density.

Additional comments regarding the test results above are contained in Section 8.1.

7.2   Preliminary Results With Fluids

7.2.1   Thermal Conductivity Of Air

In total, 28 tests of the thermal conductivity
of air were completed, tests 67-1 to 67-28 respectively.
These were completed at ambient temperature, atmospheric
pressure.

The test cells utilized were discussed in Section
5.2.1.   These were:-

Test Cell A - .010 inch dia. nickel x 6.00 inch long.

Test Cell B - .010 inch dia. nickel x 6.10 inch long.

Test Cell D - .0025 inch dia. platinum x 11.560 inch long.

The electrical networks used were described in
Section 5.2.2.   These briefly were:-

Network 1 - Bridging Technique

Network 2 - Potential Comparison Technique.

The test data was reduced initially using the
simplified line source idealization.   Subsequently, after
the writer became more aware of the importance of the
effect of the heat capacity of the source heater, five
experiments were again reduced using the finite source
model.   These results were submitted to computer analysis.
Satisfactory agreement between the experimental results
and published data for air was reached with the later five
recalculated experiments.

The analytical models used were:-

1. Line Source Approximation - See Section 4.2.

2. Finite Line Source Approximation with computer reduction, See Section 4.3, and Appendix 11.

Summary Of Tests With Air

Number Of Tests	Test Cell	Network	Data Reduction Model	"K" B.T.U./Hr.Ft.Deg. Fah.		
				Min.	Av.	Max.
67-1 to 67-5	A	1	1	.0069	.0080	.0091
67-6 to 67-14	B	1	1	.0051	.00682	.0086
67-14 to 67-20	D	1	1	.0039	.00516	.0076
67-20 to 67-28	D	2	1	.00515	.00528	.00557
67-18 to 67-21) Recalculated 67-24, 67-25			2	.0151	.01605	.0174

Accepted value for air, 72 Deg. Fah., 14.7 P.S.I.A.

.014 to .015 B.T.U./Hr.Ft.Deg.Fah.

7.2.2 Preliminary Tests Of The Thermal Conductivity
Of Water

Tests 67-29 to 67-84 were completed under this part of the programme, 55 tests in all. The liquid tested was distilled water, under ambient laboratory temperatures and pressures.

The test cell used throughout was Cell D, described in Section 5.2.1, and was furthermore the same cell used for the latter tests of air.

The electrical network used for monitoring the finite source heater was the potential comparison system, as described in Section 5.2.2. During the subject work, several modifications were completed at particular stages of the testing programme, with the following objectives:-

1. Modifications to render the system more convenient in use.

2. Changes to facilitate cross checking of the potentiometers.

3. Modifications to reduce noise interference.

The writer compiled a data reduction programme using the Finite Source Model as a basis, associating with this a least squares technique for data smoothing. The reader is referred to Section 4.3 for a description of the mathematical model, and to Appendix 11 for

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the computer programme.

On the following page is presented a summary of test results obtained. Tests 67-55 to 67-69, 67-71, 67-81, 67-82, 18 in all are presented as representative of the whole, but fall in general near the end of the preliminary testing.

Test 67-55 was the first test analysed completely by computer, incorporating a least squares technique for data smoothing. Earlier tests had been analysed by computer calculation of the finite source coefficients followed by manual plotting and best fitting.

The computer programme removed a possible degree of observer bias from the best fitting, which resulted in a more consistent data analysis.

Tests 67-58, 67-59, 67-60, 67-61 were completed on the same day, with identical finite source strengths, but with varying rest times between tests. These tests were devised to determine if possible, the length of time that fluid currents, induced by convective heat transfer, persisted. Test 67-59 was initiated after a rest time of 10 minutes, test 67-60, after 20 minutes, and test 67-61 after 30 minutes.

As is discussed further in the next chapter, no observable trend was detected. These four tests, on the other hand, could be used as a measurement of the system reproducibility.

Tests 67-62, 67-63, 67-64 were completed for the

Summary Of Preliminary Tests With Water

		"K" B.T.U./Hr.Ft.Deg.Fah.		
Test	Current	3 secs.	5 secs.	10 secs.
67-55	.15205	.3830	.3939	.4258
67-56	.15205	—	.3842	—
67-57	.15198	.3891	.3929	.3982
67-58	.15270	.4119	.4172	.4253
67-59	.15270	.4294	.4272	.4315
67-60	.15270	.4239	.4240	.4343
67-61	.15270	.4413	.4317	.4317
67-62	.15500	.4306	.4338	.4383
67-63	.15500	.4230	.4275	.4348
67-64	.15500	.4217	.4352	.4362
67-65	.15815	.4282	.4242	.4307
67-66	.15295	.4271	.4318	.4370
67-67	.15295	.4378	.4403	.4399
67-68*	.15245	.4256	.4341	.4388
67-69	.15245	.4290	.4315	.4380
67-77	.12190	.3647	.3752	.3784
67-81	.12160	.3797	.3816	.3876
67-82	.10070	.3654	.3744	—
67-68*	.15245	.3669	.3728	.3816

*Recalc.

All testing with water at 72 $\pm$ 1.5° F.

explicit purpose of showing system producibility. Each of these tests was completed with a 10 minute rest period between successive runs. The finite source strengths were identical. No adjustments of accessories were made between the tests, to preserve at a constant value any systematic system error.

The writer was unable for two months to determine the source of a large systematic error which introduced an 18% to 20% discrepancy between his results and accepted data. Error calculations showed that 5% was a probable system error with 10% a maximum possible value. Eventually, a gross wiring error was discovered, this being corrected immediately prior to test 67-77. Recalculation of test 67-68 confirmed that the error had introduced a 15 - 16% offset.

Tests 67-77 to 67-84 yield results within 6% at the 3 second interval, in all cases of the accepted value of thermal conductivity data under the writer's temperature and pressure conditions.

The writer and his advisor thence decided to proceed with the development of a final refined system.

7.3 Tests With The Final Test Apparatus

During this part of the writer's experimentation, one hundred and fourteen tests in all were conducted. Thirty-one of the above tests were required for equipment calibration and checking of characteristics of the experimental system and accessories. The remaining eighty-three tests were determinations of the thermal conductivity of fluids, seventy-eight using distilled water, and five using methyl alcohol. Of these eighty-three determinations, thirty-six were subjected to computer analysis. The other forty-seven were rejected due to a variety of reasons as discussed below.

Those tests involved in calibration and checking of equipment were tests 67-85 to 67-93, 68-1 to 68-8, 69-1 to 69-6, 69-29, 69-37, 69-38, 70-16, 70-16A, and 70-24.

The relevant observations of this testing is reported in Chapter 6.

The initial thermal conductivity determinations made under this part of the programme were numbers 69-7 to 69-28, 69-30 to 69-36 respectively, twenty-eight in all. Of this number, eight were subjected to computer analysis.

The test fluid for these determinations was distilled water, at atmospheric pressure, and at laboratory ambient temperature. The reader is referred to Section 4.3 for a

description of the mathematical model used, and to Appendix 11 for the computer data analysis programme.

The data obtained by the writer are shown on the following page, and it can be observed in all cases, the values of thermal conductivity are excessively low. The thermal conductivity cell in the configuration used during these tests was difficult to purge of residual gas during filling, and the writer suspected that the cell was only partially filled with distilled water. The writer purged and refilled the cell immediately after test 69-28, and again after test 69-32. In each case, the following tests showed a marked difference in the data obtained. Following test 69-36, the cell and enclosure was completely dismantled for replacement of the source heater since it had failed at the completion of test 69-36. Furthermore, the electrical heating wire on the pressure vessel had shorted to the pressure vessel thermostat during test 69-5. The cell was modified at this time to the format as described in Section 4.3.2. This involved changing the charging connections and modifying the purging system by designing new pressure vessel seals which would permit a vacuum to be drawn within the cell. The pressure vessel thermostat heating elements were also replaced by "Inconel" sheathed resistance wire, as has been previously described in Chapter 5.

After the above modifications had been completed, the thermal conductivity determinations 70-1 to 70-15,

Summary Of Tests With Final Apparatus

Test	Current (Milliamperes)	"K" B.T.U./Hr.Ft.Deg.Fah.			
		2 sec.	3 secs.	5 secs.	10 secs.
69-23	27.2	.0236	.0261	.0301	.0370
69-27	27.2	.0207	.0229	.0266	.0330
69-28	27.2	.0247	.0272	.0299	.0385
69-32	52.8	.0638	.0685	.0752	.0849
69-34	52.7	.0926	.1014	.1143	.1327
69-36	52.7	.0736	.0805	.0910	.1088
70-1	85.6	.3445	.3512	.3533	
70-2	85.6	.3203	.3237	.3266	
70-3	85.6	.3424	.3444	.3485	
70-4	85.6	.3491	.3536	.3558	
70-5	85.6	.3524	.3502	.3515	
70-6	85.6	.3422	.3436	.3449	
70-7	84.8	.3307	.3337	.3355	
70-8	84.8	.3539	.3520	.3518	
70-9	84.8	.3053	.3062	.3162	
70-10	111.4	.3657	.3675	.3700	
70-12	111.4	.3512	.3562	.3589	
70-13	109.7	.3580	.3616	.3641	
70-14	109.7	.3615	.3728	.3767	
70-15	109.7	.3505	.3513	.3563	
70-17	85.9	.3344	.3387	.3471	
70-18	85.9	.3302	.3357	.3400	
70-19	85.9	.3481	.3580	.3651	
70-20	85.9	.3732	.3818	.3822	

H_2O
14.7 psia
72° F

Test	Current (Milliamperes)		"K" B.T.U./Hr.Ft.Deg.Fah.			
			2 secs.	3 secs.	5 secs.	10 secs.
70-22	85.7	H₂O 14.7 psia 72°F	.3797	.3830	.3827	
70-23	85.7		.3689	.3712	.3744	
70-25	82.7		.0502	.0769	.1049	
70-26	82.7		.0419	.0605	.0799	
70-34	82.8	Alcohol 14.7 psia 72°F	.0651	.0828	.1161	
70-35	82.8		.0501			
70-36	82.8		.0405	.0593	.0931	
70-38	81.9		.3068	.3286	.3407	
70-40	81.3	H₂O 14.7 psia 72°F	.2987	.3165	.3310	
70-41	81.3		.3182	.3336	.3546	
70-48	79.7	72°F	.1192	–	–	
70-51	79.6		.3678	.3895	.4016	

70-17 to 70-24, 70-21B to 70-42, 70-45 to 70-49, and 70-50 to 70-52 were made. The following tests were subjected to computer analysis, 70-1 to 70-10, 70-12 to 70-15, 70-17 to 70-20, 70-22, 70-23, 70-25, 70-26, 70-34 to 70-36, 70-38, 70-40, 70-41, 70-48 and 70-51. Tests 70-45 to 70-49 were conducted with methyl alcohol within the cell of high but unmeasured purity. The remainder were determinations with distilled water. A resistivity test was conducted before filling the cell, and this showed greater than 2 Meghohms cm. resistivity. No other purity checks were made. All the above tests reported were completed with the test fluid experiencing the laboratory ambient temperature and pressure.

The tests 70-1 to 70-9 were conducted to show if possible overall experimental reproducibility, and were conducted over a period of one week. The finite source current was maintained at a near constant value during the tests, at 85.6 to 85.8 milliampere. A drifting standard cell was replaced following test 70-6. Test 70-9 showed a significant drop in value of thermal conductivity so obtained, but the writer can detect no valid cause for its rejection from the series of tests in determining the overall system reproducibility.

Test 70-11 was rejected due to the chart recorder pen running short of ink.

Tests 70-10 to 70-22 were conducted with three

different heating currents, to make an initial assessment of the effect of this variable.

Test 70-21 was rejected due to excessive drift on one channel of the chart recorder. The cause of the drift was found to be a loose grounding connection.

During test 70-24, the chart paper ran askew.

Tests 70-24C and 70-27 were rejected since one or the other of the chart recorder amplifiers were off scale at the start of the test. Further, this was probably the first indication of a failure of a potential lead on the finite source heater. Test 70-28 showed an unusual response on the chart recorder, which was later shown to be an intermittent open circuit in a potential lead.

Tests 70-25 to the end of the testing showed a large increase in the scatter of results, which the writer feels was caused by the above potential lead failure.

Alcohol was used in the cell for cleaning purposes, and during tests 70-45 to 70-49 this liquid was subjected to a thermal conductivity determination. While the writer suspected a partial failure of the potential lead, he also wished to verify that the potential lead had not become twisted about the source heater. As the source heater was freely suspended, the small movement it would make in response to vibrations of the cell and the laboratory table, would change the contact resistance between the source heater and the potential lead. The writer felt

that alcohol with its higher resistivity could effect the contact resistance. No increase in the stability of the system was noted with alcohol rather than water in the cell. The data as shown for test 70-48, while it is closely representative of accepted values for methyl alcohol, is presented as an aside only.

After testing was terminated at test 70-53, the writer was able to show by resistance measurement that the heater potential lead was indeed open.

By blocking up one particular side of the pressure vessel thermostat to induce a 5 degree angle off vertical, which would result in the source heater being 5 degrees off plumb as well, the lower potential lead circuit exhibited 200,000 ohms or more resistance to the finite source current lead. When the enclosure was again set level, the potential lead circuit showed the nominal resistance of approximately 20 ohms to the finite source current lead. This was considered definite verification of the lower potential lead failure.

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

8.1 Insulating Material

For the four tests completed, 66-5 to 66-8 **(P. 148)**
respectively, a mean value of .046 BTU/Hr.Ft.Deg.Fah.
was obtained, with a maximum spread of 13% between the
mean value and individual test results.

Gebhart (71) showed a value of .045 BTU/Hr.Ft.
Deg.Fah. for the same material ("Vermiculite") at a
density of 9 pounds/Ft.^{3}.

This material consisted of course granular particles,
composed of expanded asbestos chips, some of which exceeded
1/4 inch in length and breadth. When poured into the
thermal conductivity cell, there was evidence that the
material segregated to some extent, and there was small
probability that the mixture could be considered homogen-
eous.

It is the writer's opinion that the assumption of
a homogeneous medium cannot be considered true for this
substance, which would introduce the scatter of experim-
ental results.

Furthermore, the absolute value as quoted by
Gebhart (71) of the thermal conductivity value must be
assigned a considerable tolerance, **due to the variable**

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nature of the material.

The rather good agreement with very basic equip-
ment encouraged the writer and his advisor to proceed
with the next phase, extending the technique to liquids.

8.2   Preliminary Tests With Fluids

8.2.1   Tests With Air

The writer completed tests 67-1 to 67-28
(see P. 152) before he was fully aware of the importance
of the thermal capacity ratio of the finite source material/
test medium, and in fact, was made aware of this factor
after surveying the results obtained using the idealized
line source model.

For this phase of the work, the following observat-
ions were relevant:-

1. The data reduction model 1, the idealized line
source model cannot be used when the thermal capacity
ratio differs greatly from unity, for short time tests
with fluids. For an air-platinum system, the thermal
capacity ratio is approximately 1000.

The same data analysed using the finite source
model differed by 300% in the value of thermal conductiv-
ity so determined.

With reference to Section 7.2.1, tests 67-20 to
67-28, the mean value determined using the line source
model was .00528 BTU/Hr.Ft.Deg.Fah. Tests 67-20, 21, 24,
25 when reduced a second time using the finite source
model, showed .01605 BTU/Hr.Ft.Deg.Fah. as the value of

thermal conductivity. The later value was 6% higher than the accepted value of .014 to .015 BTU/Hr.Ft.Deg.Fah. for air under the test conditions.

2. The reproducibility of the test results was influenced by the electrical network used. When using the bridge system, tests 67-14 to 67-20 showed a scatter of up to 47% from the average value, whereas with the potential comparison technique, tests 67-20 to 67-28 showed a maximum scatter of 5.5%. The same test cell "C" was utilized in both of these series of tests, and cannot be considered to have affected the results. The heating currents through the finite source heater was .086 to .12 amperes in tests 67-14 to 67-20, and from .058 to .151 amperes in tests 67-20 to 67-28.

3. For testing gaseous fluids, a disadvantage of the method was illustrated. The finite source model requires input of the following data:- specific heat of the wire, density of wire, specific heat of the test fluid, density of test fluid, radius squared of the heater wire and thermal diffusivity of the test medium, all having inherent uncertainties. When the thermal capacity ratio differs greatly from unity, the uncertainties associated with the above are reflected in the value of thermal conductivity so determined.

The results obtained at this point were also generally encouraging, and the decision was made to proceed with a system which would contain liquids.

8.2.2 Preliminary Tests With Water

The general objectives of this part of the work were:-

1. Develop a system and technique which would provide thermal conductivity data for water within 5% of the accepted value, the fluid being exposed to ambient laboratory pressures and temperatures.

2. Determine the factors of importance in equipment design which would assist in the design of a refined system capable of greater accuracy.

3. Develop a satisfactory method of data reduction.

4. Increase the overall experience of the writer in the subject method of thermal conductivity measurement.

The following conclusions can be made,

1. The group of tests 67-62, 67-63, and 67-64 was one of several series of runs made to show the reproducibility of the system as a whole. The values of thermal conductivity determined (see Page 156) did not differ by more than $\pm$ 1.25% from the mean value. These particular tests were biased by approximately 18% above the accepted value for water. As was discussed in Chapter 7, Section 7.2.2, the source of the systematic error was discovered and corrected in later tests. The wiring error did not affect the validity of the above tests as indic-

ators of system reproducibility.

2. Tests 67-58, 67-59, 67-60 and 67-61 were completed to show if natural circulation currents persisted after a test run, and if so, to determine the length of time required for these currents to decay. The rest time between runs was extended from 10 minutes to 20 minutes, and finally to 30 minutes between each of the above successive runs. No trend was observed from these tests. If such currents were of significance, their effects were less than the system reproducibility. At the 10 second interval, the maximum deviation from the average thermal conductivity value of the four tests did not exceed 1.26% which was essentially identical with system reproducibility. The purpose of completing this series of tests was based upon a hypothesis that such circulation currents did occur and were, in fact, the source of the systematic error previously discussed.

3. The average thermal conductivity value determined from tests 67-77, 67-81 67-82 and 67-68 (recalculated) was .3691 at the 3 second interval, at the 5 second interval .3760 and at the 10 second interval (neglecting test 67-82 which was run for less than 10 seconds), .3825, all in BTU/Hr.Ft.Deg.Fah. The accepted value of thermal conductivity for water at 72° Fah., 1 atmosphere pressure is .345 BTU/Hr.Ft.Deg.Fah. (Gebhart (71) referring to Schmidt & Sellschopp 1932 (8) data). The above data

exceed by 6.25%, by 9%, and by 10.5%, the Schmidt & Sellschopp data, at the 3 second, 5 second, and 10 second intervals respectively.

4. Each test undertaken and analysed at this time showed a progressive increase in the indicated value of thermal conductivity with time during test. This in all probability indicated that natural convection was occurring during the testing.

8.3 Final Tests With Water

The objectives of the final part of the experimental programme were:-

1. Design, manufacture, and develop a technique and equipment to enable determinations of thermal conductivity of liquids, particularly light and heavy water, using the finite source method.

2. Develop a suitable enclosure providing a close approximation to an isothermal environment for the thermal conductivity cell. The design limits were to be the critical state temperature and pressure for light water.

3. Develop and acquire, where possible, suitable ancillary equipment for the successful operation of the thermal conductivity measurement system.

4. Evaluate the complete system, and show the system reproducibility by testing light water.

It is the writer's opinion that these objectives were met to a considerable degree, but not completely.

The pressure-vessel-thermostat and thermal conductivity cell were constructed and after two major modifications were suitable for testing light and presumably heavy water, at ambient conditions. The pressure vessel-thermostat was pressure tested to 3000 p.s.i. at 150° Fah., without leakage occurring. The writer would point

out that he no longer is confident the pressure-vessel
enclosure would withstand 3000 p.s.i. at 700° Fah.,
since the design stresses of the end caps of the pressure
vessel would be beyond the yield stress of pure copper
at elevated temperatures. He would suggest a stainless
steel backing disc over the present copper end caps
if the above pressure limits at temperature are contem-
plated.

The approach to isothermality of the pressure
vessel-thermostat was verified by test. At 160° Fah., it
was shown possible to obtain less than 1/20° Fah. temper-
ature variation between midpoint of the enclosure to
within one inch of each end. The writer contemplated
further testing of this part of the system, involving
much closer attention to the balancing of the heat inputs
to the end heaters of the thermostat, but late delivery of
a suitable temperature controller and other recurring
equipment problems prevented this further investigation.

The writer developed and tested ancillary equipment
to an extent limited by financial resources. The maximum
resolution and precision using the available equipment
was obtained.

As was presented in Chapter 6 and Chapter 7, a rather
serious problem in the equipment was the manipulation and
endurance of the finite source heater wire. In all,
seven filaments were manufactured, of which only

two were successfully used for thermal conductivity determinations. Both of these failed in service. Yet finite sources of the size utilized by the writer must be used to minimize the thermal capacity correction, and to reduce the test time, which is of importance with respect to natural convective effects.

As discussed in Chapter 7, the results in this part of the programme were terminated by the second finite heater wire failure. Furthermore, this failure of the source heater was progressive, since the first definite indication of failure occurred at test 70-25, with preliminary indications possibly as early as test 70-22. The writer thence considers all succeeding tests as unreliable.

The testing prior to 70-1, namely 69-1 to 69-36, were considered equally as unreliable, since the data indicated incomplete purging of the gases from the cell. This was discussed in further detail in Chapter 7.

The remaining tests, 70-1 to 70-21 were thence considered as the only valid tests.

The first nine tests, 70-1 to 70-9 were completed after the cell modification which permitted proper purging of the gases from within the cell, and were completed under essentially the same source heat strength in all cases. Their purpose was to show system reproducibility.

With reference to the data as presented in Chapter 7,

the following average values were obtained:-

Tests 70-1 to 70-9, nine tests in all, at 72° Fah., atmospheric pressure; heating current 85.6 to 84.8 milliamperes.

Average value of thermal conductivity at 2 second test time-

.3378 BTU/Hr.Ft.Deg.Fah.

Average value of thermal conductivity at 3 second test time-

.3398 BTU/Hr.Ft.Deg.Fah.

Average value of thermal conductivity at 5 second test time-

.3427 BTU/Hr.FT.Deg.Fah.

Tests 70-1 to 70-8, eight tests in all;

Average value of thermal conductivity at 2 second test time-

.3419 BTU/Hr.Ft.Deg.Fah.

Average value of thermal conductivity at 3 second test time-

.3444 BTU/Hr.Ft.Deg.Fah.

Average value of thermal conductivity at 5 second test time-

.3459 BTU/Hr.Ft.Deg.Fah.

The accepted value of thermal conductivity of distilled water at 72° Fah., atmospheric pressure-

.345 BTU/Hr.Ft.Deg.Fah.

The averaged data presented above **vary** by -

-2.08%, -1.5%, and -.67%, considering test 70-1, at the 2, 3, and 5 second test time.

The averaged data for tests 70-1 to 70-8 varies by -1.57%, -.17% and + .262% from the accepted value.

Test 70-9 yielded inexplicably lower results than the preceeding tests 70-1 to 70-8, falling 11.2% lower than the accepted value. The writer was unable to justify this large discrepancy.

The remaining tests 70-1 to 70-8 exhibited a maximum deviation of 4.75% from the average value of the eight tests.

It is the writer's opinion that the system reproductibility could be considered as $\pm$ 5%, until further testing with the equipment is carried out. The accuracy of the results are more difficult to establish, but should better $\pm$ 6%, for finite source heating currents not exceeding 85 milliamperes. These conclusions are furthermore valid for 1 atmosphere pressure and ambient temperatures. In Appendix 13 is presented an error analysis for the complete system, showing a maximum possible error of 4% can be expected, which does not consider the effects of natural convection.

There was evidence in the tests analysed, a progressive increase in the value of the thermal conductivity indicated at 2 seconds after initiation of test. With reference to test 70-3, the value of the thermal conductivity indicated after two seconds into the test, was

.3424 BTU/Hr.Ft.Deg.Fah., at 3 seconds, .3444 BTU/Hr.
Ft.Deg.Fah. and at 5 seconds, .3485 BTU/Hr.Ft.Deg.Fah.,
the latter value being 1.7% greater than the 2 second
test value. This increase indicated the effects of natural
convection within the cell, thereby increasing the effect-
ive measured thermal conductivity. A plot of the test
data for test 70-8 is presented in Appendix 12. It is
possible to observe the region of the onset of natural
convection by reference to this plot.

8.4 Recommendations For Further Study

1. The writer would recommend that further work be pursued using the subject method of thermal conductivity measurement. With suitable electronic readout equipment now available, it would be feasible to attain a ten fold increase in the system precision, since the reproducibility of the present system was essentially dependent on the **resolution** of the readout equipment. The most extensive error source in the present instrumentation was that introduced by the chart recorder, used for data readout. The writer would recommend that high gain signal conditioning amplifiers, showing a frequency response from 0 to 1000 cycles or better, be utilized to replace the chart recorder used. Resolution in the 1/10 microvolt region would be required. The output of these amplifiers could then be recorded by a digital voltmeter and printer, or by computer.

2. The writer would recommend that testing be carried out at lower source heat strength than was possible with the present equipment, limited as it was by sensitivity and frequency response in the readout accessories. The lower the source heat strength, the less significant the natural convective forces become. Sufficiently low source strengths should enable extension

of the subject method to high temperature studies.

3. The writer would recommend that the readout system be interfaced with a small electronic computer. The process by which the writer completed the data reduction was not only laborious but lengthy in feedback. While the test could be completed in under ten seconds, approximately one half hour was required to transcribe the chart recorder data onto computer coding pads. A few hours delay generally occurred before the cards could be typed, after which the data reduction programme was assembled and the computer run completed. This delay made difficult the detection of causes and effects during equipment checkout.

4. The writer would request that the design of the pressure-vessel-thermostat be checked if higher temperatures - high pressure studies are undertaken, considering in particular the temperature-yield stress relations for pure copper.

5. It was the writer's intention to provide a facility to verify the temperature coefficient of resistivity of the finite source heater wire. This was not done due to inherent instability of the small platinum resistance thermometers about which the writer's system was originally designed. An absolute measurement system of high accuracy would require the verification of the temperature coefficient of resistivity of the finite

source when in situ.

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APPENDIX 1

Truncation Errors For The Exponential Integral

The value of the exponential integral has been commonly determined from a series solution of the following form:-

$$Ei\,(x) = C + Ln\,(x) + \sum_{n=1}^{\infty} \frac{x^n}{nn} \qquad (A.1.1)$$

where C is .57721 ..... Euler's Constant

x is the argument, greater than zero

A computer programme was compiled which enabled the calculation of the value of the integral for a wide range of argument covering the values experienced in thermal conductivity testing in this report. The value of the integral was determined for two, three and eleven terms of the series expansion.

For eleven terms, the value of the integral corresponded to the ninth decimal with data compiled by Abramowitz & Stegun (69). The value of the argument extended from .000028 to .007013.

x	Ei (x) (11 terms)	Ei (x) (3 terms)	Ei (x) (2 terms)	Ei (x) (11 terms) / Ei (x) (3 terms)	Ei (x) (11 terms) / Ei (x) (2 terms)
.000280	7.6019258	7.6019268	7.6016464	.9999998	1.0000369
.000701	6.6860580	6.6860590	6.6853580	.9999998	1.0001049
.002524	5.4069452	5.4069471	5.4044227	.9999997	1.0004668
.007013	4.3897724	4.3897848	4.3827715	.9999972	1.0015974

Additional approximations of the integral at interim values of the argument x showed that the truncation error consistently increased with the magnitude of the argument.

In order to relate the argument to the line source solution and thence to a thermal conductivity system, the substitution of operating conditions and cell constants and calculation of the value of the argument for these conditions will be shown below:-

For the line source solution, x is defined as $\dfrac{R^2}{4\,dt}$

where R, d, t is radius, thermal diffusivity of medium, and time respectively.

Radius (Ft)	Time (Hour)	Thermal Diffusivity $(Ft^2/Hour)$	$\dfrac{R^2}{4\,dt}$
$\dfrac{.0005}{12}$	$\dfrac{1}{3600}$	.00557***	.000280
$\dfrac{.0025*}{12}$	$\dfrac{10**}{3600}$	.00557	.000701
$\dfrac{.0015}{12}$	$\dfrac{1}{3600}$	.00557	.002524
$\dfrac{.0025}{12}$	$\dfrac{1}{3600}$	.00557	.007013

* The diameter of platinum wire utilized by the writer in the majority of tests was .0025 inches.

** Testing times fell within a 1 to 10 second time

period.

••• .00557 Ft^2/Hr. is the thermal diffusivity of water at 68° Fah.

APPENDIX 2

Axial Heat Conduction To End Supports Through A Finite Heater

In a thermal conductivity measurement system utilizing a fine wire heater as an emitter, there is the possibility that significant heat conduction may occur in an axial direction through the heater wire itself.

This possibility of axial conduction must be considered in the system design.

Assuming the ends of the heat wire experience a constant temperature throughout a test, then the axial temperature distribution of the wire has been shown by Carslaw & Jaeger (41), Page 151, to be given approximately by:-

$$\frac{T(y)}{T(L)} = \frac{q}{2 R_1 H} \quad \frac{1 - \text{Cosh } M (L-y)}{\text{Cosh } ML} \qquad (A.2.1)$$

where $T(y)$ is the temperature at axial distance y from the end of the heater wire   Deg. Fah.

    q = the source strength   B.T.U./Hr./Ft.

    R_1 = the heater wire radius   Ft.

    L = the half-length of heater wire   Ft.

    y = the axial co-ordinate along

       wire, with y = o end of wire   Ft.

    M is defined as $(2H/K_w R_1)^{\frac{1}{2}}$

    H is defined as $(K_m/R_1 \text{Ln} \frac{Ro}{R_1})$

where Kw = the thermal conductivity of the wire

B.T.U./Hr.Ft.Deg.Fah.

Km = the thermal conductivity of the surrounding

medium   B.T.U./Hr.Ft.Deg.Fah.

Ro = the radius of the containment vessel

Ft.

For values of y close to 0 or to 2L then the hyperbolic function Cosh (x) can be replaced by its' exponential equivalent and upon substitution, yields:-

$$\frac{T\ (y)}{T\ (L)} = \frac{q}{2\ K_m}\ \ln \frac{Ro}{R_1} \left(1 - \exp\left(- \frac{y}{R_1}\ \left(2\ \frac{Km}{Kw}\ /\ \ln\ \frac{Ro}{R_1}\right)^{\frac{1}{2}}\right)\right)$$

(A.2.2)

This equation was used by the writer to investigate the effects of axial conduction. Upon substituting values of cell constants, and thermal data for a typical test, as given below:-

For Rô = .25/12 (cell radius)   Ft.

R_1 = .0015/12 (heater radius) Ft.

Km = .345 (water at 68 Deg. Fah. B.T.U./Hr.Ft.Deg.Fah.

Kw = 40. (platinum)   B.T.U./Hr.Ft.Deg.Fah.

q = .157 (representative heat

flux)   B.T.U./Hr.Ft.

y = .001/12 to 1.00/12   Ft.

the following values were obtained:-

Y Ft.	$\dfrac{T_y}{T_L}$
.001/12	.041
.010/12	.336
.100/12	.984
1.000/12	$1.000 - 1.5 \times 10^{-18}$

It is clear from the above results that the effects of heat conduction to the emitter end supports does not extend more than one inch from the supports.

The potential lead type of cell was utilized by the writer. Potential leads were positioned on the heater wire more than one inch from the ends of wire, consequently the active section of the wire between the potential leads experienced no end effects due to axial conduction.

APPENDIX 3

Determination of Onset Of Natural Convection

The writer has made an extensive review of the literature on the subject of heat transfer by natural convection from fine wires. This is presented in Section 2.3 of this report.

The writer reviewed in detail the approach used by Van der Held et al., (44) by Horrocks & McLaughlin (64), and recently by Tye (6) in the application of the work of Kraussold (43) and Beckmann (42) to the transient finite source method of thermal conductivity measurement.

Upon the review of the experimental work done by the latter two, it was with reluctance that the writer accepted their work as being relevant to the vertical finite source system. However, the criterion reached by this approach was determined for the writer's system, and the numerical values calculated.

The method of determining the time before the onset of convective effects from Kraussold's & Beckmann's work was deduced by Van der Held (44).

Van der Held proposed the following:-

1. The relevant dimensional gap parameter could be considered the thickness of the heated liquid annulus surrounding the finite source. The annular gap was bounded

on the inside by the surface of the finite source. The outer surface was the interface between the heater fluid and the bulk fluid. The latter surface expanded with time as the temperature field extended further from the finite source.

2. The effective temperature difference, that is the thermal potential inducing the convective flow, could be considered as the temperature of the surface of the finite source less the temperature of the fluid outside the interface.

Van der Held, referring to Kraussold's work concluded that for particular values of the Grashof-Prandtl product, natural conductive heat transfer ceased to exist.

For negligible natural convective heat transfer, the criterion became:-

$$\text{Gr. Pr.} = \frac{gB\Delta^3 T}{Vd} \qquad 1000 \qquad\qquad (A.3.1)$$

where g = the gravitational acceleration   Ft./Hr.^{2}

B = the coefficient of expansion of the test medium   1/Deg.Fah.

T = the effective temperature potential difference   Deg.Fah.

Δ = the gap thickness   Ft.

V = the test medium viscosity   Ft.^{2}/Hr.

d = the test medium thermal diffusivity   Ft.^{2}/Hr.

The effective temperature difference between the finite source and the interface of heated and undisturbed

liquid can be determined by using the line source

solution, (see Section 4.1, Equation 2)

$$T(R_1 t) = \frac{-q}{4 \pi Km} \ E \ (i) \left(\frac{R_1^2}{4 \ dt}\right) + T\infty \qquad (A.3.2)$$

$$T(Ro,t) 2 \ T\infty \qquad (A.3.3)$$

where $T(R_1 t)$ = the temperature of the finite source

surface

$T(Ro,t)$ = the temperature of the bulk fluid at the

interface, and equals the bulk fluid temperature $T\infty$.

Therefore $T = T(R_1 t) - T\infty \qquad (A.3.4)$

$$= \frac{-q}{4 \pi Km} \ E \ (i) \left((\frac{R_1^2}{4 \ dt})\right) + T\infty - T\infty \qquad (A.3.5)$$

$$T = \frac{-q}{4 \pi Km} \ E \ (i) \left((\frac{R_1^2}{4 \ dt})\right) \qquad (A.3.6)$$

Considering the conduction equation for the cylindrical

annulus of heated fluid:-

$$T(R_1) - T(Ro) = \frac{q}{2 \pi Km} \ (\ Ln \ \frac{Ro}{R_1}) \qquad (A.3.7)$$

or

$$\frac{Ro}{R_1} = exp \ (\frac{2 \pi K \ T \ (R)}{q}) \quad \text{where } T \ (Ro) - T\infty = o \qquad (A.3.8)$$

$$Ro = R_1 \ exp \ (\frac{2 \pi K}{q}) \ T(R_1) \qquad (A.3.9)$$

The thickness of the annular gap:-

$$= R_o - R_1 \qquad (A.3.10)$$

$$= R_1 \ exp \left(\frac{2 \pi K \ T \ (R_1) \ -1}{q}\right) \qquad (A.3.11)$$

Substituting for $T \ (R_1)$ by the line source solution,

$$= R_1 \quad \exp\left(\tfrac{1}{2} \text{ Ei } (\tfrac{R^2}{4\pi dt}) - 1\right) \qquad \text{(A.3.12)}$$

Upon substitution of equations A.3.6, A.3.12 into equation A.3.1 and equating A.3.1 to 1000, one obtains:-

$$1000 = \frac{gBq - \text{Ei } (\tfrac{R_1^{\frac{1}{2}}}{4 \, dt}) \quad R_1^{\,3}}{4\pi \text{ Km V d}} \quad \exp\left(\tfrac{1}{2} \text{ Ei } (\tfrac{R_1^{\,2}}{4\pi dt}) - 1\right)^{3}$$

$$\text{(A.3.13)}$$

where Ei is the exponential integral.

A computer programme was compiled by the writer which solved the above equation by iteration for the time "t".

For a typical test completed by the writer, the following data was inserted:-

g = 32.2 x 3600 x 3600   Ft.Hr.^{2}

B = .0001 (H_2O at 68 Deg.Fah.)   1/Deg.Fah.

Km= .345 (H_2O at 68 Deg.Fah.)   B.T.U./Hr.Ft.Deg.Fah.

V = .0000108 x 3600 (H_2) at 68 Deg.Fah.   Ft.^{2}Hr.

d = .0054 (H_2O at 68 Deg. Fah.)   Ft.^{2}Hr.

R_1= .00252/24   Ft.

q = .25   B.T.U./Hr.Ft.

Sufficient iterations were completed to bring the right hand side of equation A.3.13 to within .1% of 1000. The value of time calculated, and converted to seconds was:-

t = 16.64 seconds.

This illustrates that for the particular representative test data above, 16.64 seconds would elapse after initiation of the heat input before natural convective heat

transfer would effect the data.

APPENDIX 4

Radial Temperature Distribution Across A Cylinder Under

Constant Internal Heat Generation

Inherent in an experimental thermal conductivity system which utilizes a fine wire as a heat emitter and also as a resistance thermometer is the assumption that the wire be essentially isothermal in a radial direction at all times. Gebhart (71) derived the expression for the temperature distribution across a solid cylinder subjected to a uniformly distributed heat source within the cylinder. Axial conduction was assumed negligible.

This expression, below:-

$$T - T_1 = \frac{q\,R_1^2}{4\pi K}\left(1 - \frac{R^2}{R_1^2}\right) \qquad\qquad (A.4.1)$$

where T is the temperature within the cylinder at radius
R   Deg. Fah.

T_1 = the temperature at the cylinder surface.

Deg. Fah.

q = the constant heat flux generated internally.

B.T.U./Hr.Ft.^{3}

R_1 = the radius of the cylinder surface.

Ft.

R = the radius within the cylinder.

Ft.

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K = the thermal conductivity of the cylinder

material   B.T.U./Hr.Ft.Deg.Fah.

This equation expresses the temperature excess at any point within the cylinder with respect to the surface temperature of the cylinder.

The maximum temperature excess appears for R = o. Therefore

$$T - T_1 = T \max = \frac{q\,R_1^2}{4\pi K} \qquad (A.4.2)$$

From a representative test in this writer's work, the following values were obtained:-

R_1 = .0015/12 = .000125   Ft.

K   = 40.2 (Platinum)   B.T.U./Hr.Ft.Deg.Fah.

q   = 3.3 x 10^6 B.T.U./Hr.Ft.^{3}

Substitution in Equation A.4.2 yields:-

T max = .00031   Deg.Fah.

This indicates that measurement of the surface temperature of the heater wire by resistivity measurements on the heater as a whole would introduce a small systematic error. For the particular data above, the total temperature rise during the test, measured by the resistivity method, was .3 Deg. Fah.

The systematic error introduced in the temperature measurement due to the radial temperature distribution across the emitter, represented somewhat less than 1/2 of .00031 Deg. Fah., and was therefore insignificant.

APPENDIX 5

Properties Of Selected Pure Metals

Reference: Smithells C.J., _Metals Reference Book_,
Interscience Publishers Inc., New York, 1949.

Eckert E.R.G., Drake R.M., _Heat & Mass Transfer_,
McGraw-Hill Book Company, Inc., New York, 1959.

| | Metal | | | | | |
Property	Copper	Silver	Nickel	Gold	Tungsten	Platinum
Temp. Coeff. of Resistance (0 Deg. to 100 Deg. C) ohms/ohm	.0043	.0041	.0064	.0034	.0048	.0039
Stability of Temp. Coeff. of Resistance	-	-	-	-	-	Excellent
Ultimate Strength (P.S.I.)	60,000	18,000	41,000	16,000	150,000	18,000
Percent Elongation (annealed)	45	60	28	25	25	40
Thermo Voltage against copper at 100 Deg. C. -Ref. junction 0 Deg. C. (M.V.)	-	+ 1.50	- .72	+ 1.50	+ 1.88	+ .76
Thermal Capacity Ratio with water	.820	.533	.949	.600	.691	.693
Temp. Coeff. of Expansion 0 to 100 Deg. C in/in x 10^6		19.		14.4	4.45	8.9

APPENDIX 6

Temperature Distributions Along the

Pressure Vessel Thermostat

Differential Thermocouples

Differential — Reference Junction

Furnace

End Cap

Support Leg

1720 °F

Furnace at 160° F

Differential Thermocouple Voltages (microvolts)

Distance along Furnace (inches)

APPENDIX 7

Design Of A Low Gradient Cylindrical Furnace

An objective, and in fact a necessary requirement for the satisfactory performance of the thermal conductivity cell was the creation of a uniform temperature along a significant part of the length of the cell. This required the design and testing of the pressure vessel-thermostat consistent with that criterion.

A cylindrical furnace experiences increased heat loss from the ends of the furnace and must have additional heat input at the ends to compensate for this loss, if gradientless performance is desired. The factors which determine this loss are several, the important parameters being the length/diameter of the furnace core, the thermal resistance of the insulating material with which the core is surrounded, and the thermal properties of the furnace and charge.

During the initial part of the programme, the writer was fortunate in meeting M. T. Laubitz at the National Research Council in Ottawa, who had engaged in serious study regarding the design of low temperature gradient cylindrical furnaces. His 1959 publication, reference 70, "Design of Gradientless Furnaces", represented a compilation of his extensive researches and was an invaluable aid in the design of the subject pressure-vessel thermostat.

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Laubitz presented applicable design data, relevant to the cylindrical furnace, this information being dimensionless in format, which permitted the manufacture of furnaces of variable sizes and geometries, and of differing materials. His approach was both analytical and experimental in scope, with very good agreement.

The general problem of cylindrical furnaces involved the proper compensation of the loss of heat from the ends of the furnace, which required the input of heating power at the ends. For an electrically heated furnace, the heating winding density must be increased, or alternately separate heating circuits must be added at the ends. The magnitude of the additional heat input and its distribution formed the essence of the Laubitz paper.

The pressure-vessel thermostat as described in this report, was designed to meet particular criterion dictated by the thermal conductivity cell. However, those dimensions and parameters which were not limited by the above were adjusted to be consistent with good thermal design in accordance with Laubitz (70).

The parameters of the subject pressure-vessel thermostat of thermal importance, and consistently named in accordance with the nomenclature of Laubitz were these:-

L - Length of furnace and enclosure, inches.

L_h- Length of furnace encompassed by heaters, inches.

R_a- Inner radius of furnace, at the radius of the elect-

rical heaters, inches.

R_b- Outer radius, or that of the sheet metal enclosure, in the writer's system, inches.

K_p- Thermal conductivity of the insulating medium of the furnace, B.T.U./Hr.Ft.Deg.Fah.

H_v- Surface conductance at the shell of the furnace, B.T.U./Hr.Ft.^{2}Deg.Fah.

The value of the parameters of the pressure-vessel thermostat were these:-

L = 30 inches

L_h= 12 inches

R_a= 1½ inches

R_b≅ 9 inches

K_p= 0.04 B.T.U./Hr.Ft.Deg.Fah. (Diatomaceous earth, powder, 200 Deg.Fah.)

H_v= surface conductance, not required.

The following ratios can be formed:-

$$\frac{Rb}{Ra} = \frac{9}{1½} = 6$$

$$\frac{L}{Ra} = \frac{30}{1½} \approx 20$$

$$\frac{K_p}{H_v L} = \frac{.04}{H_v \times 30}$$ (Laubitz required that this parameter have the value zero, or infinity,

only. For the present situation, it was taken as zero).

$$\frac{Lh}{L} = \frac{12}{30} = .4$$

With the above parameters, Laubitz (70) Table 1

shows for the necessary input power factor, q (dimension-less, by interpolation of Table 1),

q = 2.14

The necessary power required to heat the furnace to temperature To, Deg. Fah. equation 1, reference (70) becomes:-

$$Q = q \, K_p \, L \, T_o \qquad \text{(A.7.1)}$$

where Q is the required input power in B.T.U./Hr.

$$Q = 2.14 \times .04 \times \frac{30}{12} \times To$$

$$= .214 \, T_o \text{ B.T.U./Hr.}$$

To determine the respective heat inputs over the length, L, of the furnace, it is necessary to define $1 = L/15$. 1_o refers to the middle of the length of the furnace, whereas 1_1, 1_2, 1_3 etc. are respectively numbered from this middle plane.

With the previously defined parameters L_h/L, R_b/R_a, L/R_a, $K_p/H_v L$, and referring to Table 2, reference (70), the power distribution factors a_n become, through inter-polation as before:-

$a_o = o$

$a_1 = 17$

$a_2 = 82$

$a_3 = 432$

Using the defining equation 2, reference (70),

$$\frac{Q \, Ln}{Q \, Lo} = 1. + A_n \times 10^{-3} \qquad \text{(A.7.2)}$$

Thus $\dfrac{Q\ Lo}{Q\ Lo} = 1.0$, $\dfrac{QL1}{QLo} = 1.017$, $\dfrac{QL2}{QLo} = 1.082$, $\dfrac{QL3}{QLo} = 1.432$

Laubitz pointed out that the above analysis considered only heat loss through the furnace, and neglected the flow of heat through the central tube and charge. This was legitimate when only a small part of the heat flowed through the contents of the furnace. The ratio of the heat passing through the contents of the furnace to that of the heat input required to bring the furnace to temperature T_o, as given by equation A.7.2, unfortunately changes with T_o. This is due to the differing changes of thermal conductivity with temperature of the insulation and of the furnace contents. Laubitz therefore found that the temperature distribution factors varied slightly with temperature, and that the furnace was gradientless at only one particular temperature. To correct for this, he recommended that separate heater circuits be incorporated, to permit independent adjustment of the heater inputs when changing from one furnace temperature to another.

The writer persued this course in the design of the pressure-vessel thermostat, incorporating one full length uniformly wound heater, and in addition, provided two independent end heaters for each end of the vessel. The vessel is further described in Chapter 5. The required wattage of the heaters was based on required inputs as determined by equation A.7.1, solved at 700 Deg. Fah.

Testing of the temperature profile on the completed

furnace showed that at a temperature of 160 Deg. Fah.,
a maximum temperature difference of 1/22 Deg. Fah. was
observed from the centre to 20% from the end of the furnace.
Furthermore the prediction of heating power required, as
given by equation A.7.1 at 160 Deg. Fah., was approximately
7% lower than measured by test.

Appendix 6 illustrates the temperature distribution
along the furnace when at a temperature of 160 Deg. Fah.

APPENDIX 8

Wet Cell Battery Characteristics At Constant

Low Current Drain

During the experimental programme, the regulated
power supply (Item 9) originally used as the power source
for the finite source heater wire was found insufficiently
stable with respect to line voltage fluctuations. A
suitable unit was found to be costly since an output volt-
age regulated to the microvolt level requires elaborate
electronic design.

Thus the writer acquired six wet-cell storage
batteries, automotive type, rated 120 ampere hours, 6 volts,
which he felt to be adequate, considering the low power
requirements during testing.

The particular battery tested as described below,
was two years old at time of test, and a gross discharge
capacity test performed on it indicated a capacity of
88 ampere hours, at a final discharge voltage of 5.50
volts.

After recharge, the battery was discharged 20%, and
then further discharged at a small current flow of 80
milliamperes. The battery was connected in series with
an oil bath power resistor (Item 17) and a 100 ohm
standard resistor and the voltage across the battery was

monitored at frequent occasions by means of a potentio-
meter and volt box. The test was carried out over 6 days,
the time being recorded each time a measurement of voltage
was made. During the first 24 hours the voltage dropped
at 3.84 microvolts/hour, neglecting the first hour after
connection. During the remaining 120 hours on test, the
battery voltage fell at 3.55 microvolts/hour.

Since the majority of the writer's tests involved
time periods of 20 seconds or less, the battery power
supply could be considered close to ideal as a constant
voltage generator.

It was observed that interruption of the current
drain for more than a few milliseconds resulted in a rise
of the battery voltage. Consequently the writer's exper-
imental system included a double throw relay, which connect-
ed an equivalent load across the battery supply when a test
was not in progress.

APPENDIX 9

Measured Resistances Of Inshop Resistors

Item 17 Power Resistor - Test 69-1

Resistance Tap	Resistance (Ohms)	Resistance Tap	Resistance (Ohms)
R1-2	.2187	R8-9	10.3631
R2-3	.4608	R9-10	15.3434
R3-4	.6987	R10-11	25.4010
R4-5	1.0582	R11-12	52.6440
R5-6	1.9630	R12-13	94.6619
R6-7	3.0741	R13-14	200.7809
R7-8	5.5801	R1 -14	412.2478

Temperature 23.2 Deg. Cent.

Item 18 Power Resistor - Test 69-1

Resistor	Resistance
R1	49.0212
R2	24.4996
R3	11.8693
R4	332.2117
R5	25.4853
R6	10.1361

Temperature 23.2 Deg. Cent.

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APPENDIX 10

Chronology Of Tests Completed

The large number of tests and the resultant
data therefrom produced during the testing programme
required a systematic method of collating and of preserv-
ing this information for future reference. The follow-
ing chronology presents the summation of tests completed.
The data, being in the large part on roll charts and
computer data cards, has been deposited in the Heat Transfer
Laboratory Faculty of Engineering Science, for preservation.

To facilitate retrieval of the data, the appropriate
roll charts and computer decks have been identified by
the particular test number assigned to the test run. The
test runs have been numbered consecutively with each number
being prefaced by a two digit number, which represents the
last two digits of the year in which the test was undertaken.

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
66-1	17/10/66	Preliminary tests with "Vermiculite"	1	-	-
66-2	19/10/66	"	1	-	-
66-3	20/10/66	"	1	-	-
66-4	21/10/66	"	1	-	-
66-5	25/10/66	Refined tests with "Vermiculite"	1	-	-
66-6	6/11/66	"	1	-	-
66-7	7/11/66	"	1	-	-
66-8	9/11/66	"	1	-	-

Test Data For Test 66-5, 25/10/66

Time (Seconds)	Thermocouple Voltage (Millivolts)
0	.980
26	.985
43	1.005
60	1.030
77	1.055
94	1.080
114	1.105
136	1.130
160	1.155
188	1.180
200	1.190
214	1.200
227	1.210
242	1.220
256	1.230
274	1.240
290	1.250
308	1.260
328	1.270
347	1.280
369	1.290
390	1.300
414	1.310
440	1.320
466	1.330
493	1.340
521	1.350
554	1.360
587	1.370
620	1.380
830	1.430
930	1.450

Thermocouple:

Copper-Constantan

Reference Junction:

0 Deg. C.

Current Flow

.4692 amperes

Test #	Date	Remarks				Lab. Book #	Data Chart #	Computer Printout
67-1	14/1/67	Thermal	Conductivity	Of	Air	1 - P.26	67-1	-
67-2	14/1/67	"	"	"	"	1 - P.26	67-2	-
67-3	14/1/67	"	"	"	"	1 - P.26	67-3	-
67-4	14/1/67	"	"	"	"	1 - P.27	67-4	-
67-5	14/1/67	"	"	"	"	1 - P.27	67-5	-
67-6	17/1/67	"	"	"	"	1 - P.38	67-6	-
67-7	17/1/67	"	"	"	"	1 - P.38	67-7	-
67-8	17/1/67	"	"	"	"	1 - P.40	67-8	-
67-9	17/1/67	"	"	"	"	1 - P.41	67-9	-
67-10	17/1/67	"	"	"	"	1 - P.41	67-10	-
67-11	17/1/67	"	"	"	"	1 - P.41	67-11	-
67-12	17/1/67	"	"	"	"	1 - P.41	67-12	-
67-13	17/1/67	"	"	"	"	1 - P.42	67-13	-
67-14	29/1/67	"	"	"	"	1 - P.66	67-14	-
67-15	10/2/67	"	"	"	"	1 - P.72	67-15	-
67-16	10/2/67	"	"	"	"	1 - P.72	67-16	-
67-17	13/2/67	"	"	"	"	1 - P.76	67-17	-
67-18	13/2/67	"	"	"	"	1 - P.76	67-18	-
67-19	21/2/67	"	"	"	"	1 - P.78	67-19	1 - P.250
67-20	21/2/67	"	"	"	"	1 - P.78	67-20	1 - P.251
67-21	21/2/67	"	"	"	"	1 - P.78	67-21	1 - P.243
67-22	21/2/67	"	"	"	"	1 - P.78	67-22	-
67-23	21/2/67	"	"	"	"	1 - P.78	67-23	-
67-24	22/2/67	"	"	"	"	1 - P.79	67-24	1 - P.206
67-25	22/2/67	"	"	"	"	1 - P 79	67-25	1 - P.249

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
67-26	22/2/67	Thermal Conductivity Of Air	1 - P.79	67-26	-
67-27	22/2/67	"	1 - P.79	67-27	-
67-28	22/2/67	"	1 - P.79	67-28	-
67-29	2/3/67	Thermal Conductivity Of Water	1 - P.84	67-29	1 - P.223
67-30	2/3/67	"	1 - P.84	67-30	-
67-31	2/3/67	"	1 - P.84	67-31	-
67-32	2/3/67	"	1 - P.84	67-32	-
67-33	2/3/67	"	1 - P.84	67-33	-
67-34	7/3/67	"	1 - P.89	67-34	-
67-35	7/3/67	"	1 - P.89	67-35	-
67-36	7/3/67	"	1 - P.89	67-36	-
67-37	7/3/67	"	1 - P.90	67-37	-
67-38	15/3/67	"	1 - P.94	67-38	1 - P.164
67-39	15/3/67	"	1 - P.94	67-39	1 - P.163
67-40	15/3/67	"	1 - P.94	67-40	1 - P.166
67-41	22/3/67	"	1 - P.96	67-41	1 - P.155
67-42	22/3/67	"	1 - P.96	67-42	-
67-43	23/3/67	"	1 - P.96A	67-43	-
67-44	1/4/67	"	1 - P.97	67-44	1 - P.138
67-45	1/4/67	"	1 - P.97	67-45	-
67-46	1/4/67	"	1 - P.97	67-46	1 - P.135
67-47	1/4/67	"	1 - P.97	67-47	-
67-48	1/4/67	"	1 - P.97	67-48	-
67-49	1/4/67	"	1 - P.97	67-49	-
67-50	1/4/67	"	1 - P.97	67-50	1 - P.151
67-51	1/4/67	"	1 - P.97	67-51	-

Test #	Date	Remarks: Thermal Conductivity Of Water	Lab. Book #	Data Chart #	Computer Printout
67-52	10/4/67	Thermal Conductivity Of Water	1 - P.98	67-52	1 - P.147
67-53	10/4/67	" " " "	1 - P.98	67-53	1 - P.145
67-54	10/3/67	" " " "	1 - P.98	67-54	1 - P.128
67-55	20/4/67	" " " "	1 - P.98A	67-55	1 - P.123
67-56	4/5/67	" " " "	1 - P.98A	67-56	-
67-57	4/5/67	" " " "	1 - P.98A	67-57	1 - P.49
67-58	11/5/67	" " " "	1 - P.99	67-57	1 - P.82
67-59	11/5/67	" " " "	1 - P.99	67-58	1 - P.76
67-60	11/5/67	" " " "	1 - P.99	67-59	1 - P.68
67-61	11/5/67	" " " "	1 - P.99	67-60	1 - P.71
67-62	12/5/67	" " " "	1 - P.99	67-61	1 - P.65
67-63	12/5/67	" " " "	1 - P.99	67-62	1 - P.40
67-64	12/5/67	" " " "	1 - P.99	67-63	1 - P.34
67-65	15/5/67	" " " "	1 - P.101	67-64	1 - P.59
67-66	16/5/67	" " " "	1 - P.102	67-65	1 - P.56
67-67	16/5/67	" " " "	1 - P.102	67-66	1 - P.61
67-68	17/5/67	" " " "	1 - P.102	67-67	1 - P.55
67-69	17/5/67	" " " "	1 - P.102	67-68	1 - P.24
67-70	17/5/67	" " " "	1 - P.102	67-69	-
67-71	17/5/67	" " " "	1 - P.102	67-70	-
67-72	31/5/67	" " " "	1 - P.103	67-71	-
67-73	31/5/67	" " " "	1 - P.103	67-72	-
67-74	31/5/67	" " " "	1 - P.103	67-73	-
67-75	31/5/67	" " " "	1 - P.103	67-74	-
67-76	31/5/67	" " " "	1 - P.103	67-75	-
67-77	31/5/67	" " " "	1 - P.103	67-76	1 - P.22
67-78	1/6/67	" " " "	-	67-77	1 - P.14
				67-78	

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
67-79	1/6/67	Thermal Conductivity Of Water	–	67-79	1 – P.8
67-80	1/6/67	"	–	67-80	1 – P.5
67-81	1/6/67	"	–	67-81	–
67-82	10/7/67	"	–	67-82	–
67-83	10/7/67	"	–	67-83	–
67-84	10/7/67	"	–	67-84	–
67-85	2/9/67	Test Of Temperature Controller	–	67-85	–
67-86	3/9/67	"	–	67-86	–
67-87	3/9/67	Test Of D. C. Amplifier	–	67-87	–
67-88	5/9/67	Test Of Temperature Controller	–	67-88	–
67-89	6/11/67	"	2 – P.65A	67-89	–
67-90	9/11/67	Thermocouple Calibrations	2 – P.65A	67-90	–
67-91	10/11/67	"	2 – P.66	67-91	–
67-92	13/11/67	"	2 – P.67	67-92	–
67-93	17/12/67	Test Of Hipsometer	2 – P.84	67-93	–

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
68-1	17/1/68	Calibration Platinum Resis. Thermometer	2 - P.88	-	-
68-2	24/1/68	Calibration Differential Thermo- couples	2 - P.94	68-1	-
68-3	8/2/68	Check of Hipsometer	2 - P.84	68-2	-
68-4	9/2/68	" " "	2 - P.84	68-3	-
68-5	10/2/68	" " "	2 - P.84	68-4	-
68-6	10/2/68	Discharge Test On Batteries	2 - P.97B	68-5	-
68-7	21/2/68	Test Of Temperature Controller	2 - P.100	68-6	-
68-8	23/2/68	Temperature Checks On Pressure Vessel	2 - P.100- 103	68-7	-

Test #	Date	Remarks	Lab. Book #	Data Chart	Computer Printout
69-1	9/3/69	Check Of Inshop Resistors	3 – P.4-10	–	–
69-1A	11/3/69	Check Of Room Temperature Variations		69-1	–
69-2	20/3/69	Ground Resis. Heaters	3 – P.12	–	–
69-2A	15/3/69	Temperature Controller Checks	3 – P.13	69-2	–
69-3	21/3/69	"	3 – P.16	69-3	–
69-4	22/3/69	"	3 – P.18	69-4	–
69-5	22/3/69	"	3 – P.19	69-5	–
69-5A	23/3/69	Check Electrical Noise	3 – P.19	69-5A	–
69-6	23/3/69	Thermal Conductivity Of Water	3 – P.21	69-6	–
69-7	23/3/69	"	3 – P.22	69-7	2 – P.115
69-8	23/3/69	"	3 – P.22	69-8	–
69-9	23/3/69	"	3 – P.22	69-9	–
69-10	23/3/69	"	3 – P.22	69-10	–
69-11	23/3/69	"	3 – P.22	69-11	–
69-12	23/3/69	"	3 – P.23	–	–
69-13	24/3/69	"	3 – P.23	69-13	–
69-14	24/3/69	"	3 – P.23	69-14	–
69-15	24/3/69	"	3 – P.23	69-15	–
69-16	24/3/69	"	3 – P.23	69-16	–
69-17	24/3/69	"	3 – P.24	69-17	2 – P.111
69-18	24/3/69	"	3 – P.24	69-18	–
69-19	24/3/69	"	3 – P.24	69-19	–
69-20	24/3/69	"	3 – P.24	69-20	–
69-21	24/3/69	"	3 – P.25	69-22	–
69-22	24/3/69	"	3 – P.25	69-22	–

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
69-23	24/3/69	Thermal Conductivity Of Water	3 – P.25	69-23	2 – P.116
69-24	24/3/69	"	3 – P.25	69-24	–
69-25	24/3/69	No Test	–	–	–
69-26	24/3/69	Thermal Conductivity Of Water	3 – P.25	69-26	–
69-27	24/3/69	"	3 – P.26	69-27	2 – P.113
69-28	24/3/69	"			
69-29	2/4/69	Battery Check			
69-30	2/4/69	Thermal Conductivity Of Water			
69-31	2/4/69	"			
69-32	2/4/69	"			
68-33	4/4/69	"	3 – P.31	69-33	
69-34	4/4/69	"	3 – P.31	69-34	
69-35	5/4/69	"	3 – P.32	69-35	2 – P.103
69-36	5/4/69	"	3 – P.32	69-36	2 – P.99
69-37	30/8/69	Battery Check	3 – P.48	–	–
69-38	30/8/69	"	3 – P.48	–	–

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
70-1	10/1/70	Thermal Conductivity Of Water	3 - P.58	70-1	2 - P.70
70-2	10/1/70	"	3 - P.58	70-2	2 - P.24A
70-3	10/1/70	"	3 - P.58	70-3	2 - P.68
70-4	10/1/70	"	3 - P.58	70-4	2 - P.80,85
70-5	10/1/70	"	3 - P.58	70-5	2 - P.27A
70-6	10/1/70	"	3 - P.58	70-6	2 - P.29A
70-7	16/1/70	"	3 - P.60	70-7	2 - P.31A
70-8	16/1/70	"	3 - P.60	70-8	2 - P.1A
70-9	16/1/70	"	3 - P.60	70-9	2 - P.35A
70-10	17/1/70	"	3 - P.62	70-10	2 - P.37A
70-11	17/1/70	"	3 - P.62	70-11	2 - P.39A
70-12	17/1/70	"	3 - P.62	70-12	2 - P.41A
70-13	25/1/70	"	3 - P.63	70-13	2 - P.59,60
70-14	25/1/70	"	3 - P.63	70-14	2 - P.63,65
70-15	25/1/70	"	3 - P.63	70-15	
70-16	1/5/70	Check Of Temperature Controll-er	3 - P.67	—	—
70-16A	2/5/70	Check Of Temperature Differ-ences On Furnace	3 - P.68-70	—	—
70-17	22/5/70	Thermal Conductivity Of Water	3 - P.72	70-17	2 - P.45A
70-18	22/5/70	"	3 - P.72	70-18	2 - P.47A
70-19	22/5/70	"	3 - P.72	70-19	2 - P.33-42
70-20	22/5/70	"	3 - P.72	70-20	2 - P.50,55
70-21	23/5/70	"	3 - P.73	70-21	—
70-22	23/5/70	"	3 - P.73	70-22	2 - P.31,45
70-23	23/5/70	"	3 - P.73	70-23	2 - P.48
70-24	23/5/70	"	3 - P.73	70-24	—

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
70-24A	18/6/70	Chart Recorder Check Out	3 - P.74	-	--
70-24B	10/7/70	Thermal Conductivity Of Water	3 - P.76	70-24B	--
70-24C	10/7/70	"	3 - P.76	70-24C	--
70-25	10/7/70	" "	3 - P.76	70-25	2 - P.28
70-26	10/7/70	" "	3 - P.76	70-26	2 - P.49A
70-27	10/7/70	" "	3 - P.76	70-27	--
70-28	10/7/70	" "	3 - P.76	70-28	--
70-29	10/7/70	" "	3 - P.76	70-29	--
70-30	10/7/70	" "	3 - P.76	70-30	--
70-31	10/7/70	" "	3 - P.77	70-31	--
70-32	10/7/70	" "	3 - P.77	70-32	--
70-33	10/7/70	" "	3 - P.77	70-33	--
70-34	10/7/70	" "	3 - P.77	70-34	2 - P.51A
70-35	10/7/70	" "	3 - P.77	70-35	2 - P.25,26
70-36	10/7/70	" "	3 - P.77	70-36	2 - P.53A
70-37	18/7/70	" "	3 - P.78	70-37	--
70-38	18/7/70	" "	3 - P.78	70-38	2 - P.22
70-39	18/7/70	" "	3 - P.78	70-39	--
70-40	24/7/70	" "	3 - P.78	70-40	2 - P.55A
70-41	24/7/70	" "	3 - P.78	70-41	2 - P.17
70-42	24/7/70	Time Base Of Chart Recorder	3 - P.78	70-42	--
70-43	24/7/70	Frequency Response Of Recorder	3 - P.78	70-43	--
70-44	24/7/70	Thermal Conductivity Of Alcohol	3 - P.78	70-44	--
70-45	1/8/70	" "	3 - P.79	70-45	--
70-46	1/8/70	" "	3 - P.79	70-46	--

Test #	Date	Remarks	Lab. Book #	Data Chart #	Computer Printout
70-47	1/8/70	Thermal Conductivity Of Alcohol	3 - P.79	70-47	-
70-48	1/8/70	"	3 - P.79	70-48	2 - P.6,8,16
70-49	1/8/70	"	3 - P.79	70-49	-
70-49A	2/8/70	Gain Check Of Chart Recorder	3 - P.80	-	-
70-50	2/8/70	Thermal Conductivity Of Water	3 - P.80	70-50	-
70-51	2/8/70	"	3 - P.80	70-51	2 - P.1
70-52	2/8/70	"	3 - P.80	70-52	
70-53A	8/8/70	Temperature Controller Check	3 - P.82,94	70-53A	
70-53	21/8/70	Cell Checks	3 - P.95	70-53	

APPENDIX 11

Data Reduction Programme

The data from the majority of test runs conducted
in this report were reduced by a computer programme
compiled for the IBM 1130 Digital Computer. The programme
was written in Fortran IV language.

A copy of the programme is presented below, amply
supplied with comment statements which explain the programme
in detail. The printout for test 70-8 has been included, as
a typical example. For convenience in binding, the writer
has truncated the printout to three decimal places, whereas
five were used throughout at all other times.

The explanation of the printout is as follows:-
First line of numerical printout, five numbers in all,

First number - potentiometer setting, volts, across
potential lead circuit.

Second number- potentiometer setting, volts, across
standard resistor circuit.

Third number - chart recorder gain correction, potential
lead channel.

Fourth number- chart recorder gain correction, standard
resistor channel.

Fifth number - standard resistor value, in ohms.

The series of twelve columns represent:-

First column   - time, in seconds, from initiation of
                    test.

Second column   - voltage values in microvolts, correspond-
                    ing to the times, transcribed from
                    potential lead chart recorder channel.

Third column   - voltage values in microvolts, correspond-
                    ing to the times, transcribed from the
                    standard resistor chart recorder channel.

Fourth column   - temperature of the finite source in Deg.
                    Fah. above the temperature at the start
                    of the test.

Fifth column   - the reciprocal of the rate of temperature
                    rise, in seconds/Deg.Fah.

Sixth column   - value of the statistical correlation co-
                    efficient for (J-1) data points. (J is
                    the counter, shown in column twelve).

Seventh column   - value of the intercept of the least
                    squares line with the ordinate, after
                    (J-1) data points.

Eighth column   - value of the slope of the least squares
                    line, which is also the value of the
                    experimentally determined thermal conduct-
                    ivity for the inputed test data. Units
                    are B.T.U./Hr.Ft.Deg.Fah.

Ninth column   - value of the finite source heat strength,

B.T.U./Hr.Ft., (J) data point.

Tenth column   - value of the abscissa of the Jth data point.

Eleventh column - value of the ordinate of the Jth data point.

Twelfth column - counter, which also corresponds to the number of data points.

```
PAGE  2        TEST      70-8      COMP JAN 16/70 TRICK

      DIMENSION T(75),V(75),C(75)
C     T( )   IS THE VALUE OF TIME IN SECONDS AT WHICH THE VOLTAGE ACROSS
C            THE POTENTIAL LEAD CIRCUIT AND ACROSS THE STANDARD RESISTOR
C            CIRCUIT ARE TRANSCRIBED FROM THE CHART RECORDER RECORD.
C     V( )   IS THE VALUE OF THE CHANGE IN VOLTAGE ACROSS THE
C            POTENTIAL LEAD CIRCUIT, IN MICROVOLTS.
C     C( )   IS THE VALUE OF THE CHANGE IN VOLTAGE ACROSS THE
C            STANDARD RESISTOR CIRCUIT, IN MICROVOLTS.
      L=1
C     L      IS A COUNTER WHICH DICTATES THE POINT AT WHICH THE LEAST
C            SQUARES ANALYSIS INITIATES.
      M=36
C     M      IS A COUNTER WHICH DICTATES THE POINT OF TERMINATION
C            OF THE LEAST SQUARES ANALYSIS.
      TC=.345
C     TC     IS AN APPROXIMATE VALUE OF THE THERMAL CONDUCTIVITY
C            OF THE TEST MEDIUM, USED TO CALCULATE THE TEMPERATURE RISE
C            OF THE FINITE SOURCE AT THE FIRST DATA POINT.
      RAD=.0025/24.
C     RAD    IS THE RADIUS OF THE FINITE SOURCE, FEET
      CPW =1.
      DENW =62.4
      CPP= .0324
      DENP=1334.1
C     CPW, DENW, CPP, DENP    ARE THE VALUES OF THE SPECIFIC HEAT, AND
C            THE DENSITY OF THE TEST MEDIUM, AND OF THE FINITE SOURCE
C            MATERIAL, RESPECTIVELY, IN B.T.U./POUND DEG. FAH. ,AND
C            POUNDS/ FOOT CUBED.
      W=CPP*DENP/(CPW*DENW)
C     W      IS THE THERMAL CAPACITY RATIO.
      TD=.00557
C     TD     IS THE VALUE OF THE THERMAL DIFFUSIVITY OF THE TEST
C            MEDIUM, FEET SQUARED/ DEG. FAH.
      WL=5.887/12.
C     WL     IS THE LENGTH OF THE FINITE SOURCE HEATER, FEET
```

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```
C
C      TCR=0.00392
C      TCR         IS THE TEMPERATURE COEFFICIENT OF RESISTIVITY OF THE
C                  MATERIAL OF THE SOURCE HEATER.
C      READ(2,3)VA,CA,CFV,CFC,RSH
C      VA          IS THE POTENTIAL LEAD POTENTIOMETER SETTING, VOLTS.
C      CA          IS THE STANDARD RESISTOR POTENTIOMETER SETTING, VOLTS.
C      CFV         IS THE GAIN CORRECTION FACTOR
C      CFC         IS THE GAIN CORRECTION FACTOR
C      RSH         IS THE VALUE OF THE STANDARD RESISTOR, OHMS.
     3 FORMAT(5F10.6)
       WRITE(5,4) VA,CA,CFV,CFC,RSH
       CA=CA/RSH
       READ(2,1)T
     1 FORMAT(16F5.1)
     4 FORMAT(///5F15.6 //)
     5 FORMAT(16F5.1)
    55 FORMAT(16F5.1)
       READ(2,5)V
       READ(2,55)C
       DT1=0.
       DT2=0.
       YY=0.
       XX=0.
       SX=0.
       SX2=0.
       SY2=0.
       SY=0.
       SXY=0.
       DO6J=L,M
       N=J-L+1
       DV=V(J)*CFV/1000000.
       DC=C(J)*CFC/RSH/1000000.
       VTOT=VA+DV
       CTOT=CA+DC
       HF=VTOT*CTOT*3.413/WL
C      HF          IS THE HEAT FLUX STRENGTH IN THE FINITE SOURCE,
C                  B.T.U./HR. DEG. FAH.
       RTOT=VTOT/CTOT
       IF(J-L)50,50,51
    50 RO=RTOT
```

```
      F=RAD*RAD*3600./(2.*TD*T(J))
      CABS=ALOG(2./(F*1.7811))*(1.+F*(1.-W))+F
      DTI=VTOT*CTOT*3.413*CABS/(3.1416*4.*WL)/TC
      GO TO 51
   51 DT=(RTDT-RO)/RO/TCR*1.8
      DT2=DT
  500 DT=DT+DTI
  501 DELT=DT-DT1
      DT1=DT
      IF(J-L)60,60,61
   60 DBYT=DTI/T(J)
      DBYT=1./DBYT
      GO TO 62
   61 DBYT=DELT/(T(J-1)-T(J))
      DBYT=-1./DBYT
   62 TIME=ALOG(T(J))
      F=RAD*RAD*3600./(2.*TD*T(J))
      CABS=ALOG(2./(F*1.7811))*(1.+F*(1.-W))+F
      CORD=(DT*3.1416*4.*WL/(VTOT*CTOT*3.413))
C     CABS     IS THE VALUE OF THE ABCISSA OF THE DATA POINT (J).
C     CORD     IS THE VALUE OF THE ORDINATE OF THE DATA POINT (J).
      YY=CORD
      XX=CABS
      SX=SX+XX
      SX2=SX2+XX*XX
      SY=SY+YY
      SY2=SY2+YY*YY
      SXY=SXY+YY*XX
      B=(N*SXY-SX*SY)/(N*SX2-(SX*SX))
C  B           IS THE VALUE OF THE SLOPE OF THE BEST FIT LINE THROUGH
C              (J-1) DATA POINTS, ALSO THE EXPERIMENTAL THERMAL COND-
C              UCTIVITY IN B.T.U./HR. FT. DEG. FAH.
      AA=(SY-B*SX)/N
C  AA           IS THE INTERCEPT OF THE LEAST SQUARES LINE
      B=1./B
      RC=(N*SXY-SX*SY)/((N*SX2-SX*SX)*(N*SY2-SY*SY))**.5
C  RC           IS THE LEAST SQUARES CORRELATION COEFFICIENT
      WRITE(5,7)T(J),V(J),C(J), DT,DBYT,RC,AA,B,HF,CABS,CORD,J
    7 FORMAT(3(F6.2,1X),F6.3,F9.3,1X),4(F6.3,1X),2(F7.3,1X),I4)
    6 CONTINUE
      STOP
      END
```
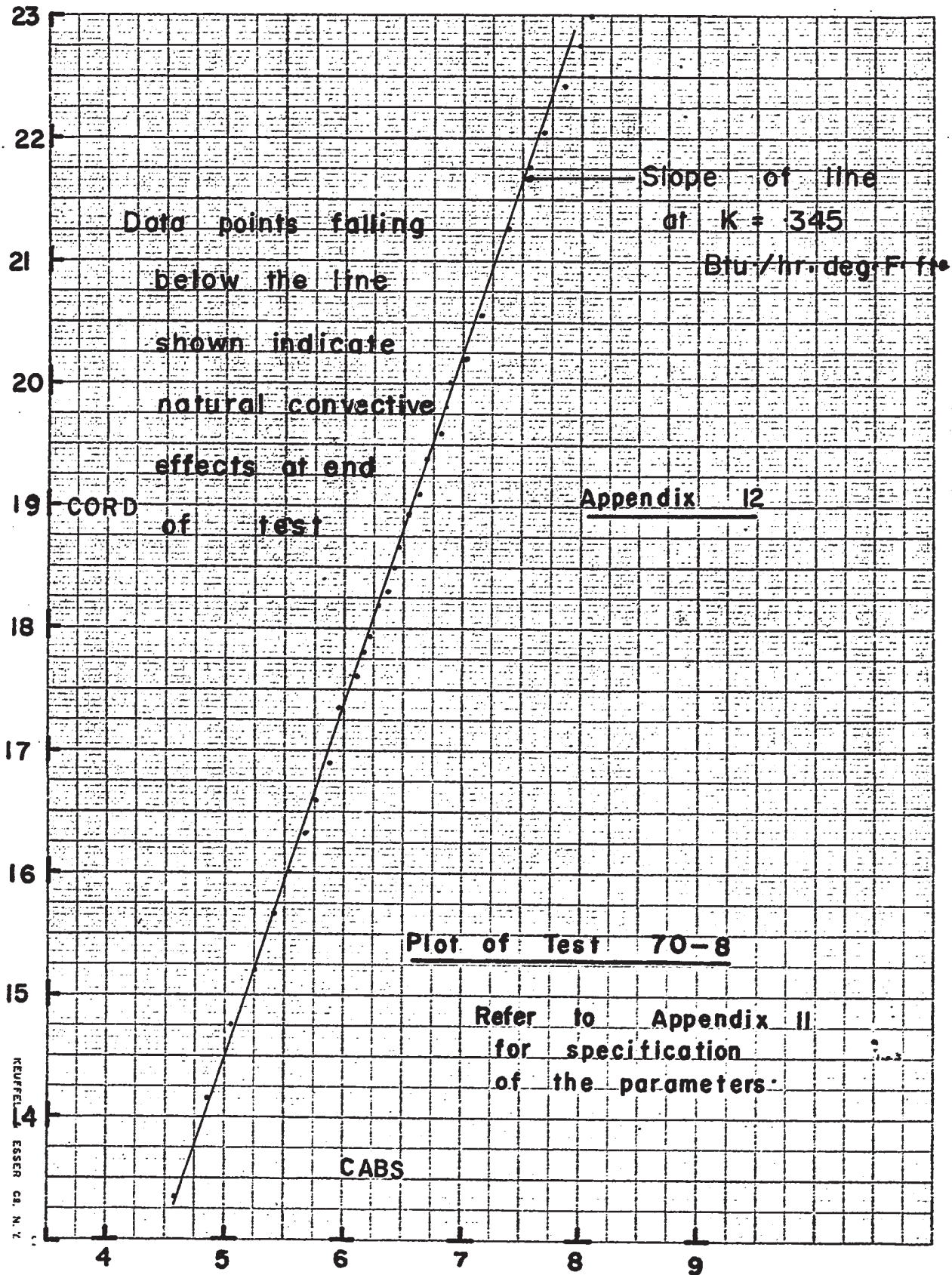
	0.302980	0.084792		1.030000		1.015000		1.000000		
1	0.30	33.00	30.00	0.189	1.583	−0.637	4.610	0.527	0.178	4.593	13.313
2	0.40	40.50	30.00	0.201	8.587	1.000	−0.038	0.344	0.178	4.874	14.131
3	0.50	46.00	30.00	0.209	11.653	0.999	0.270	0.352	0.178	5.093	14.734
4	0.60	50.00	30.00	0.215	15.840	0.999	0.700	0.363	0.178	5.273	15.177
5	0.70	54.00	29.90	0.222	14.698	0.999	0.565	0.359	0.178	5.425	15.655
6	0.80	57.50	29.90	0.228	18.540	0.999	0.479	0.357	0.178	5.557	16.034
7	0.90	60.10	29.90	0.232	24.720	0.999	0.547	0.359	0.178	5.674	16.316
8	1.00	62.50	29.90	0.236	26.314	0.999	0.632	0.361	0.178	5.778	16.585
9	1.10	65.10	29.80	0.240	22.047	0.999	0.576	0.360	0.178	5.873	16.904
10	1.20	67.50	29.80	0.244	26.314	0.999	0.502	0.358	0.178	5.959	17.171
11	1.30	69.00	29.80	0.246	41.834	0.999	0.526	0.358	0.178	6.039	17.338
12	1.40	71.00	29.70	0.250	28.130	0.999	0.489	0.357	0.178	6.112	17.588
13	1.50	72.30	29.70	0.253	34.713	0.999	0.453	0.357	0.178	6.181	17.790
14	1.60	74.00	29.70	0.254	56.259	0.999	0.479	0.357	0.178	6.245	17.915
15	1.70	76.00	29.60	0.258	26.746	0.999	0.415	0.356	0.178	6.305	18.178
16	1.80	77.00	29.60	0.260	65.260	0.999	0.409	0.356	0.178	6.362	18.285
17	1.90	78.40	29.50	0.262	36.256	0.999	0.372	0.355	0.178	6.416	18.479
18	2.00	80.00	29.50	0.265	39.794	0.999	0.321	0.353	0.178	6.467	18.656
19	2.20	82.50	29.50	0.269	51.794	0.999	0.275	0.352	0.178	6.562	18.927
20	2.43	84.00	29.50	0.271	83.668	0.999	0.289	0.353	0.178	6.648	19.095
21	2.60	86.50	29.50	0.275	51.794	0.999	0.271	0.352	0.178	6.728	19.366
22	2.80	88.00	29.40	0.278	67.980	0.999	0.259	0.352	0.178	6.802	19.573
23	3.00	90.00	29.40	0.281	63.981	0.999	0.235	0.352	0.178	6.871	19.792
24	3.50	93.50	29.40	0.287	91.659	0.999	0.244	0.352	0.178	7.024	20.175
25	4.00	96.50	29.30	0.292	95.971	0.999	0.259	0.352	0.178	7.158	20.541
26	5.00	103.00	29.30	0.302	98.880	0.999	0.227	0.351	0.178	7.380	21.251
27	6.00	107.20	29.20	0.309	139.446	0.999	0.208	0.351	0.178	7.562	21.755
28	7.00	110.00	29.20	0.313	229.793	0.999	0.262	0.352	0.178	7.716	22.060
29	8.00	113.00	29.10	0.319	194.230	0.999	0.311	0.353	0.178	7.849	22.422
30	9.00	116.00	29.10	0.323	211.887	0.999	0.349	0.354	0.178	7.967	22.753
31	10.00	118.00	29.10	0.327	313.757	0.999	0.408	0.355	0.178	8.072	22.977
32	11.00	120.00	29.00	0.330	276.532	0.999	0.459	0.356	0.178	8.168	23.231
33	12.00	122.00	29.00	0.333	313.751	0.999	0.507	0.357	0.178	8.254	23.455
34	13.00	124.60	28.90	0.338	217.538	0.999	0.514	0.357	0.178	8.334	23.777
35	14.00	126.20	28.90	0.340	407.887	0.999	0.530	0.358	0.178	8.409	23.949
36	15.00	128.00	28.90	0.343	354.675	0.999	0.542	0.358	0.178	8.477	24.147

APPENDIX 12

Plot Of Test 70-8

Plot showing a graph on grid paper with the following annotations:

Slope of line at K = 345 Btu/hr·deg·F·ft

Data points falling below the line shown indicate natural convective effects at end of test

Appendix 12

Plot of Test 70-8

Refer to Appendix 11 for specification of the parameters

CORD

CABS

APPENDIX 13

Error Analysis

As assessment of the overall system error for the source technique required the consideration of the individual error contributions of a number of variables. Unfortunately, a few of these variables were not mathematically resolvable, and an estimate of their error required resorting to the judgment and experience of the writer.

Test 70-8 has been selected as a particular example for error investigation. The sources of error which could contribute to the overall error were these:-

1. Experimental equipment errors in measured values.

These variables were -

(a) Time errors due to

    1. Chart recorder chart drive motor variations and ultimately, line frequency deviations.

    2. Chart recorder paper length changes due to humidity and inaccuracy in printing.

    3. Possible interpolation error for the starting point of a test on the chart paper.

(b) Errors in measurement of voltage across potential taps of finite source heater, due to:-

1. Potentiometer standardization and dial linearity errors.

2. Standard voltage cell error.

3. Chart recorder gain instability and linearity error.

4. Spurious voltage due to noise pickup or thermo voltages at circuit joints.

(c) Errors in measurement of current through finite source heater, due to:-

1. Potentiometer standardization and dial linearity errors.

2. Standard voltage cell error.

3. Chart recorder gain instability and linearity error.

4. Spurious voltages due to noise pickup or thermo voltages at circuit joints.

5. Standard resistor error.

(d) Errors in the dimensions of the finite source heater wire -

1. Error in the effective position of the potential lead attachment.

2. Error in the measurement of distance between potential leads.

3. Error in the measurement of diameter of wire (a secondary variable.

(e) Possible error in the assumed temperature coefficient of resistivity of the platinum source heater.

(f) Effect of a constant stress level on the temperature coefficient of resistivity of the platinum source heater.

2. Inherent errors in the thermal property values used in the finite source model, including:-

       (a) specific heat of water.

       (b) density of water.

       (c) specific heat of platinum.

       (d) density of platinum.

       (e) thermal diffusivity of water.

3. Errors introduced by the thermal conductivity cell - water system due to:-

       (a) Shunting of current past the finite source heater due to the finite conductivity of distilled water.

       (b) Presence of natural convective fluid movement.

       (c) Presence of non-isothermality along the length of the cell.

       (d) Presence of impurities within the test medium.

4. Mathematical errors of analysis due to:-

       (a) application of mathematical model at short times, for which the model's accuracy was degraded.

       (b) possible error due to truncation of third order terms in the model. This error is associated with

4 (a) above.

The writer completed a partial differential analysis of the mathematical model used in his computer programme, to determine the effect of an error in each variable. The effect on the value of the thermal conductivity so determined when a 1% change in a variable was introduced, is shown below, computed for test 70-8.

Experimental Variable	Resultant Change In Thermal Conductivity Value Determined	
	3 seconds	5 seconds
1. Time + 1% linear time error	+ .010%	+ .011%
2. Voltage + 1% in the potentiometer voltage (potential leads)	≠ 2.00%	+ 1.97%
3. Voltage + 1% in the chart recorder record (potential leads)	− 1.05%	− 1.01%
4. Voltage + 1% in the potentiometer used to measure source heating current	+ 1.03%	+ 1.02%
5. Voltage + 1% in the chart recorder record used to monitor the change in source heating current	+ .032%	+ .031%

	3 seconds	5 seconds
6. Resistance error + 1% in standard resistor	− 1.20%	− 1.02%
7. Active length of finite source heater + 1%	− 2.00%	− 2.00%
8. Diameter of finite source heater + 1%	− .017%	− .014%
9. Specific heat of water + 1%	− .017%	− .014%
10.Density of water + 1%	− .017%	− .014%
11.Specific heat of platinum + 1%	+ .017%	+ .014%
12.Density of platinum + 1%	+ .017%	+ .014%
13.Thermal diffusivity of water + 1%	+ .014%	+ .014%
14.Temperature coefficient of resistivity of platinum + 1%	+ 1.00%	+ 1.00%

The above can be combined into equation form to determine a combined effect for an estimated maximum possible error, when considered at the 3 second test time:-

Estimated maximum possible error, %

= (linear time error, %) x .01

+ (potential lead potentiometer error, %) x 2.00

+ (potential lead chart record error, %) x 1.05

+ (current monitoring potentiometer error, %) x 1.03

+ (current monitoring chart record error, %) x .032

+ (standard resistor error, %) x 1.10

+ (active length of source heater error, %) x 1.00

+ (diameter of source heater error, %) x .017

+ (specific heat, water, data error, %) x .017

+ (specific heat, platinum data error, %) x .017

+ (density, water, error, %) x .017

+ (density, platinum error, %) x .017

+ (thermal diffusivity, water error, %) x .014 (A.13.1)

+ (temperature coefficient of resistivity of platinum,
  %) x 1.00

At the 5 second test interval, the equation becomes:-
Estimated maximum possible error, %

= ( ) x .011 + ( ) x 1.97 + ( ) x 1.01 + ( ) x 1.02 + ( ) x .031 + ( ) x 1.02 + ( ) x 2.00 + ( ) x .014 + ( ) x .014 + ( ) x .014 + ( ) x .014 + ( ) x .014 ( ) x .014 + ( ) x 2.00

(A.13.2)

where the brackets represent the same variables as shown in equation A.13.1.

The writer points out that the above equation properly accounts for the effects of individual experimental errors which are mathematically resolvable. In addition, the effects of natural convective heat transfer, impurity of water sample, effect of straining on the finite source heater and non-isothermality of the cell introduce addit-

ional uncertainty which must be considered. For these variables, only special testing and experience with the test apparatus can give the experimenter suffucient judgment to make a valid estimate of their contribution to the overall system accuracy.

In order to provide a prediction of the resolvable errors, an assessment of the magnitude of the maximum possible errors of each variable will be made below.

1. Time Scale – This variable was determined by division count on the chart recorder record. The chart recorder drive was energized by a synchronous drive motor and gear train, and as such was inherently linked to the frequency of the mains. Short term variations (over a few minutes) can reach 1%, while the 24 hour frequency stability is extremely good. Necessarily, the chart drive motor could experience this speed variation due to frequency variation. The chart paper itself can under some circumstances due to humidity and temperature conditions, change in length. The writer felt that this effect was minimized in the temperature controlled laboratory, wherein all testing was undertaken. There existed a source of error due to the identification of the point of the initiation of a test. The least count on the chart paper represented .1 second, and interpolation to .05 second was possible, thus defining the test initiation time within .05 seconds.

Considering the above factors and the results of three calibrations of the chart recorder time scale, using a stopwatch once, and an electronic counter twice, a maximum possible linear error estimate was 1.5%.

2. Potentiometer Error - Voltage Across Potential Lead Circuit

The potentiometer used in this circuit was the Guildline Model 9144, featuring resolution to .1 microvolt. The power supply for the instrument was a 60 ampere hour wet storage cell, and after standardization of the potentiometer, very small adjustment was required, at infrequent intervals. This potentiometer and, in fact, the current measurement-standard resistor circuit potentiometer as well, were standardized from the number 6 cell of the Honeywell #2778 standard cell bank. This was done normally before each test.

The maximum possible error of .1% is conservative.

3. Chart Recorder - Potential Lead Circuit

The Honeywell Electronik 16 two channel chart recorder as used in this work introduced the largest error. It was used in almost all cases at the highest gain, of 100 microvolts full scale. As previously presented in Chapters 5 and 6, the instrument was provided with an additional regulated power supply, and the utmost care taken in shielding and grounding to prevent amplifier drift. Furthermore, the instrument was limited in frequency response particularly at the initial part of a test

wherein the voltage changes were rapid.

The instrument was daily checked during testing for span and linearity, and an appropriate correction added in the data reduction programme. The span on the 100 microvolt range exhibited drift up to 3% of full scale over a week.

The maximum possible error of 1.5% was predicted provided daily gain corrections were made.

4. Potentiometer - Current Monitoring Circuit

A Prediction of .2% maximum possible error was conservative.

5. Chart Recorder - Current Monitoring Circuit

As in (3) above, a prediction of 1.5% maximum possible error was reasonable.

6. Standard Resistor Error

The standard resistor used was the Julie 1 ohm standard. The specified tolerance of the manufacturer was .1%, but cross- calibration to laboratory standards showed .05% or better.

The maximum possible error of .075% was justified.

7. Active Length Of Source Heater Wire

The measurement of the length of wire between the potential taps has been discussed previously. The measured length of 5.887 inches was determined by three tests with two observers, and the writer presents a tolerance of $\pm$ .005 inches as reasonable. In addition to this, the

junction of approximately .005 inch diameter would introduce an additional ambiguity with respect to the active length of the finite source heater equal to $\pm$ .005 inches, resulting in a total uncertainty of $\pm$ .010 inches.

The maximum possible error was therefore $\pm$ .2%.

8. Diameter of Source Heater

The diameter was determined by using a vernier micrometer at several points along its' diameter by two machinists in the Engineering Machine Shop and by the writer. Furthermore, resistivity checks after install-ation verified the mechanical measurements.

The least count of the micrometer was .0001 inch. For the wire diameter of .0025, the measurement could not have exceeded a maximum possible error of $\pm$ 4%.

9. Specific Heat Of Water At Room Temperature And Pressure

Nominal $\pm$ 1% maximum possible error.

10. Specific Heat Of Platinum

Nominal $\pm$ 1.5% maximum possible error.

11. Density Of Platinum

Nominal $\pm$ .75% maximum possible.

12. Thermal Diffusivity Of Water

Nominal $\pm$ 1% maximum possible.

13. Temperature Coefficient Of Resistivity Of Platinum

It was the writer's intention to verify the magnitude of the above variable by comparison of the in-situ finite source heater to a secondary platinum resistance thermometer which was designed to be installed in the pressure-vessel-thermostat wall. This unfortunately was not completed due to malfunction of the secondary platinum resistance thermometer. As a result, the platinum finite source heater could not be checked properly.

The platinum utilized for the source heater was purchased as reference grade platinum and carried a certification indicating that the temperature coefficient of resistivity of that particular spool reached or exceeded a value of .00392 over the 0 to 100 Deg. Cent. range. This coefficient is used to verify the purity of platinum and to check its quality with respect to use in platinum resistance thermometers. It can not exceed .00393 in the highest purity form. It was therefore possible to expect the temperature coefficient of the wire purchased for this work to fall between .00392 and .00393, representing an uncertainty of .33%.

However, in the present system, the platinum wire experienced a constant tension of approximately 2000 p.s.i. This constant tension superimposed on the temperature fluctuations could effect the temperature coefficient. The writer was unable to locate documented researches

regarding the above phenomena and the additional un-
certainty associated due to the constant stress level
must be an estimate on the writer's part.

Estimated maximum possible error in the temperat-
ure coefficient of resistivity, $\pm$ 1.5%.

By insertion of the above maximum possible error
estimates into equation A.13.1 –
Estimated maximum possible error

$$= (1.5) \times .01 + (.1) \times 2.00 + (1.5) \times 1.05 + (.2) \times 1.03$$
$$+ (1.5) \times .032 + (.075) \times 1.10 + (.2) \times 1 + (4) \times .017$$
$$+ (1) \times .017 + (1.5) \times .017 + (.5) \times .017 + (.75) \times .017$$
$$+ (1) \times .014 + (1.5) \times 1.00 = \pm 3.95\%$$

The preceeding error estimate represented the
resolvable error sources. The error introduced by natural
convective forces in the fluid, can only be determined
by repeating tests with lower source strengths until
no evidence of such occurring exists in the experimental
results. In this work, it was not possible to operate
at sufficiently low heating currents due to limitations
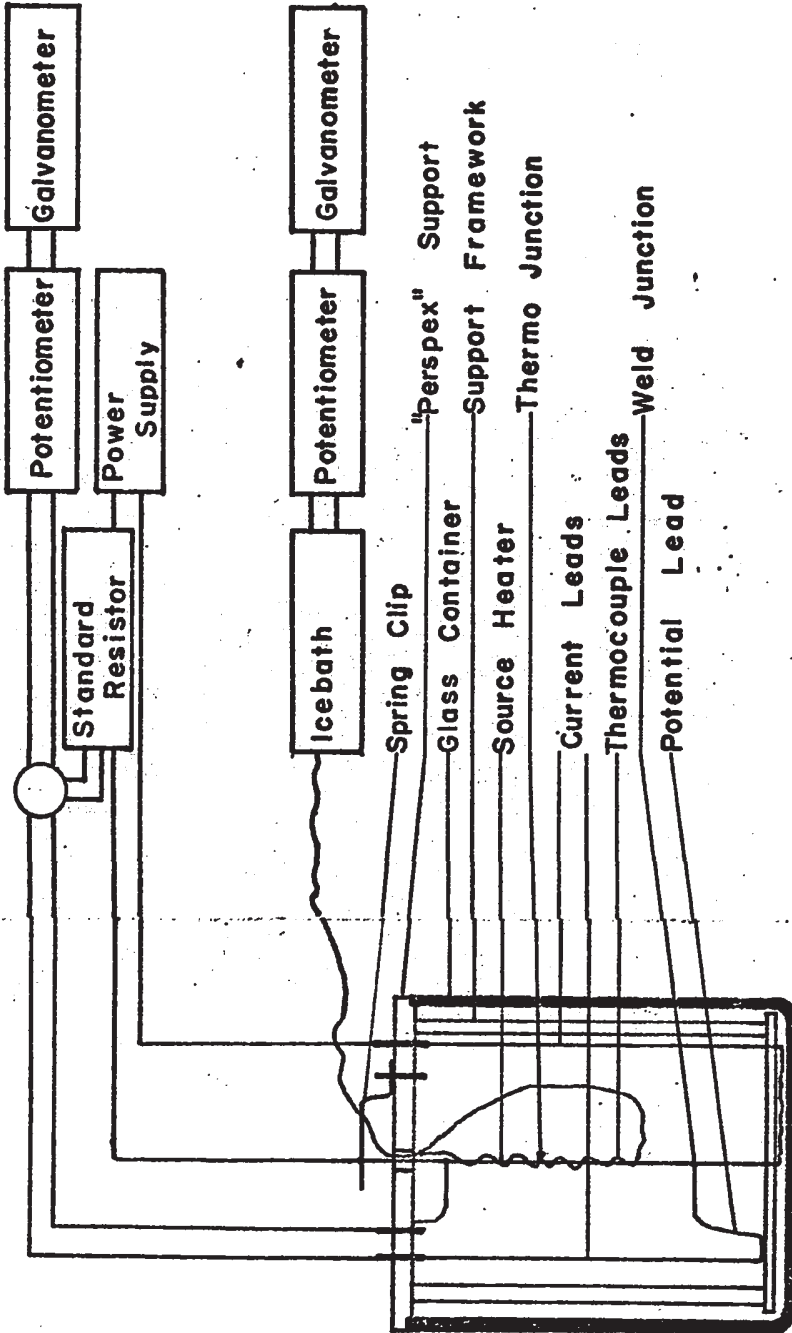in readout apparatus.

The mathematical limitations in the heat transfer
model introduced no significant error as has been shown
in Chapter 4.

The electrical shunting effect of the distilled
water which was in intimate contact with the platinum
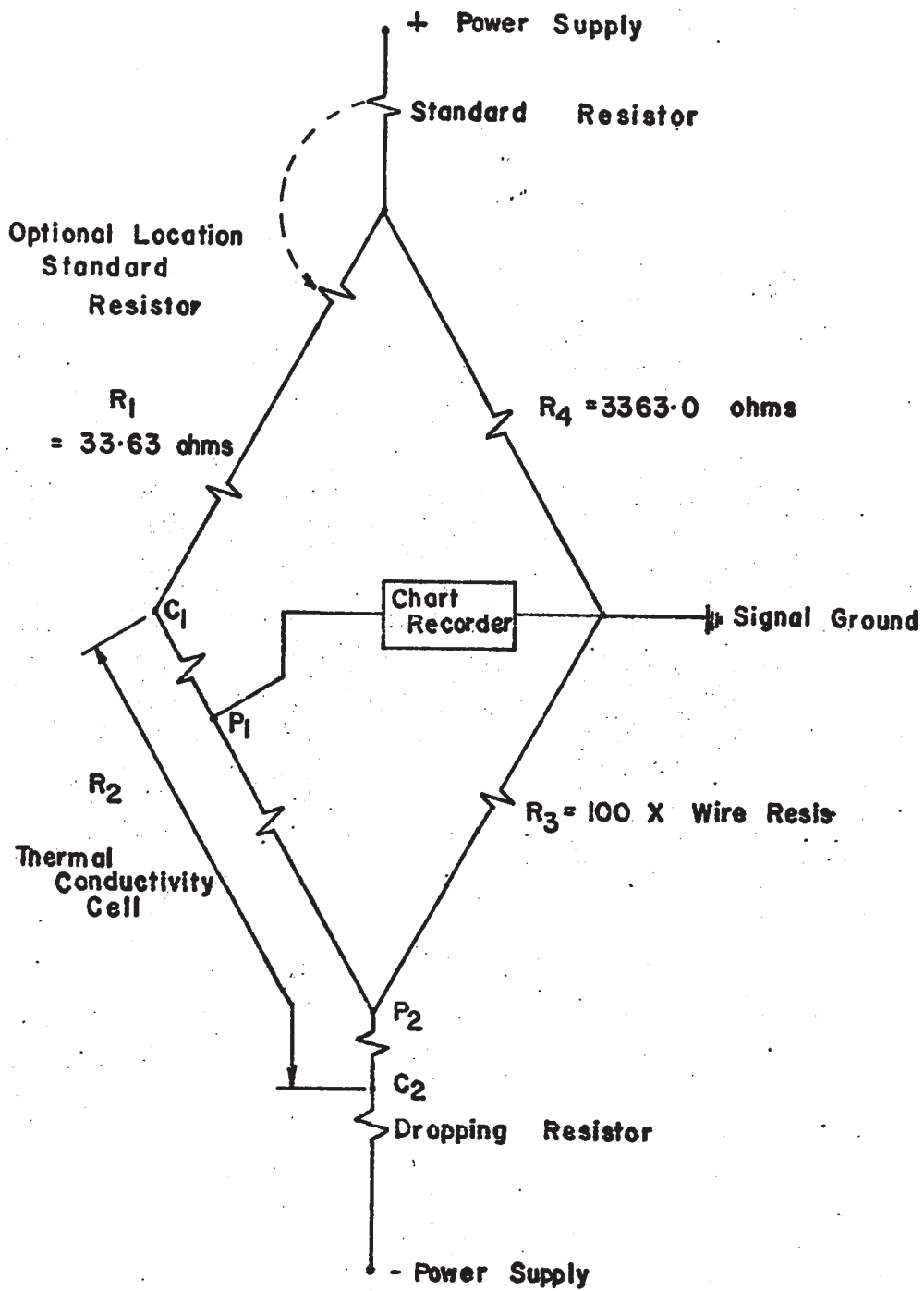source heat can be shown to introduce less than .01% in

the data, if water of 2 megohms cm resistivity is utilized.

During the final series of tests, the thermal conductivity cell and enclosure were at laboratory ambient temperature. The enclosure wall temperature, as monitored by differential thermocouples thereupon attached, showed that isothermal conditions were nearly attained, to the limits of reproducibility of the differential thermocouples.

In conclusion, the maximum possible error of the system appeared to be $\pm$ 4%. Statistically, the probable error should reach some value considerably less than this value. However, based on personal discussions with two senior researchers involved in work of this type, the probable error in most cases becomes equal to the maximum possible error due to unrecognized additional variables with their respective error contributions.
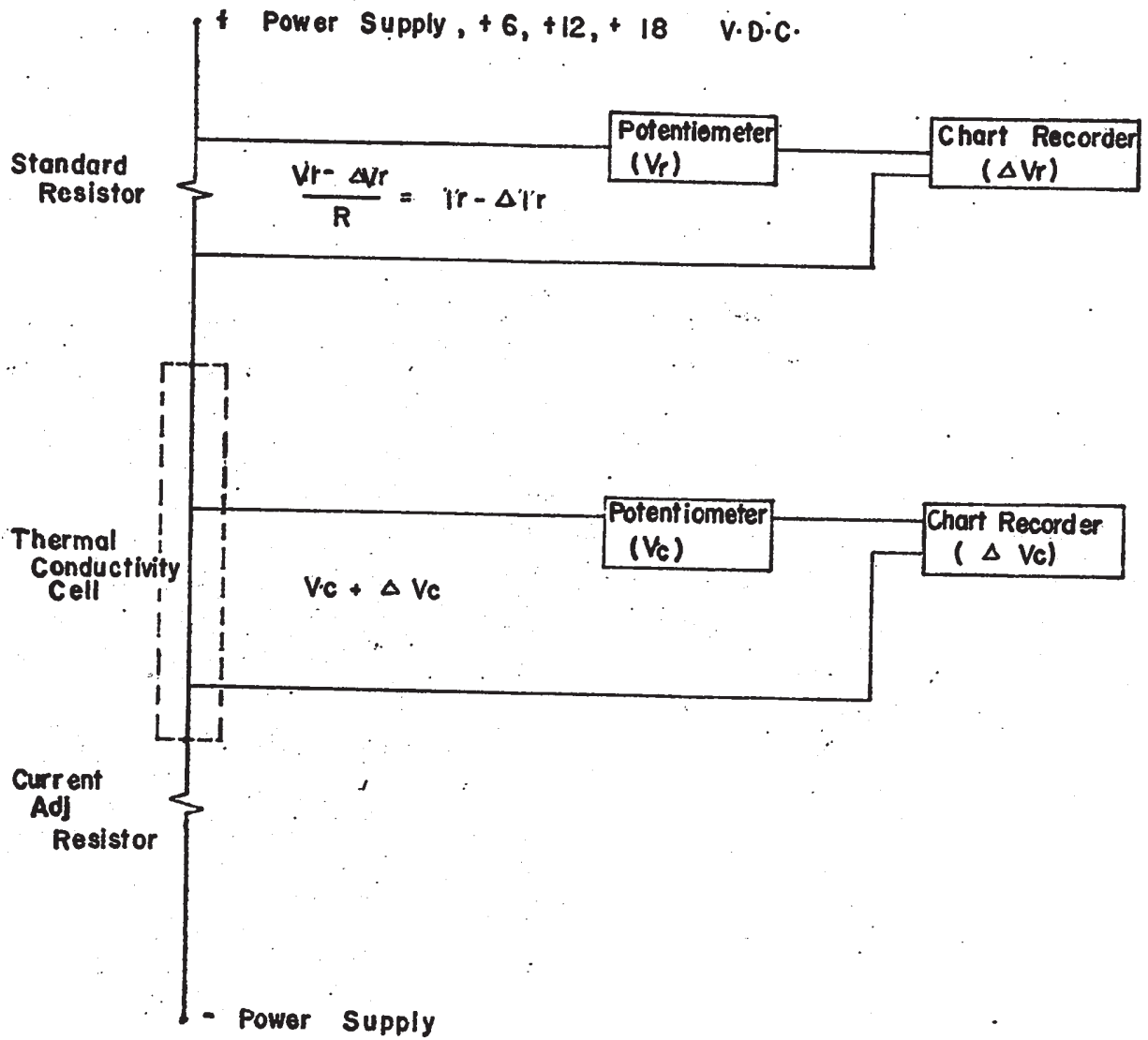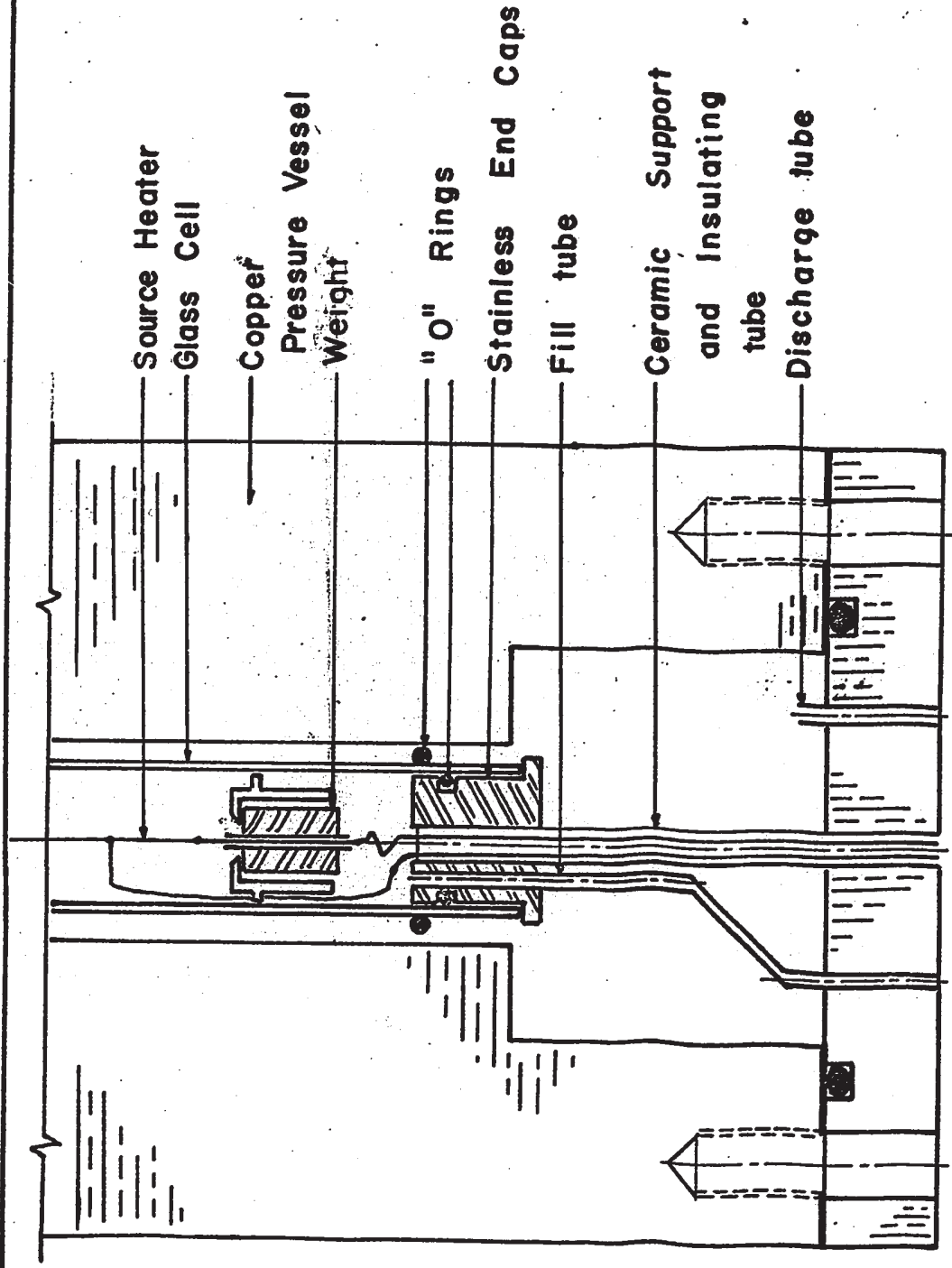
APPARATUS FOR TESTING INSULATION MATERIALS

ILL. 1

+ Power Supply

Standard Resistor

Optional Location
Standard
Resistor

R_1
= 33.63 ohms

R_4 = 3363.0 ohms

C_1

Chart
Recorder

⊩ Signal Ground

P_1

R_2

Thermal
Conductivity
Cell

R_3 = 100 X Wire Resis·

P_2

C_2

Dropping Resistor

- Power Supply

ILL· 2   ELECTRICAL BRIDGE SCHEMATIC

‡ Power Supply , + 6 , + 12 , + 18   V·D·C·

Standard
Resistor

$$\frac{Vr - \Delta Vr}{R} = Ir - \Delta Ir$$

| Potentiometer (V_r) |

| Chart Recorder (ΔV_r) |

Thermal
Conductivity
Cell

$Vc + \Delta Vc$

| Potentiometer (V_c) |

| Chart Recorder (ΔVc) |

Current
Adj
Resistor

- Power Supply

ILL· 3   POTENTIAL   COMPARISON   SCHEMATIC

ILL. 4   THERMAL CONDUCTIVITY SYSTEM

Source Heater
Glass Cell
Copper
Pressure Vessel
Weight
"O" Rings
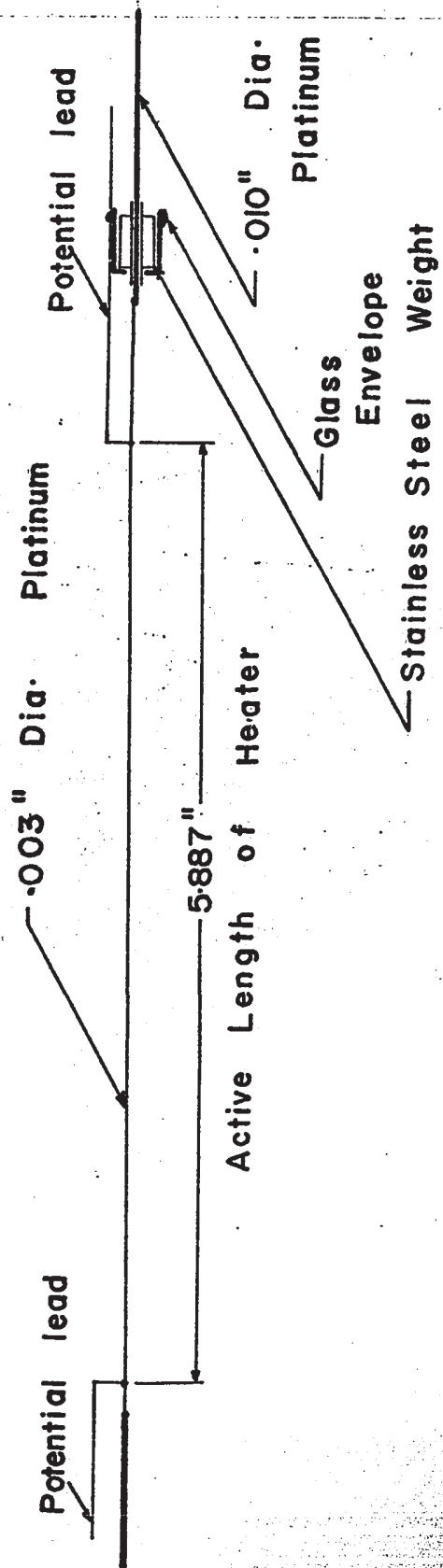Stainless End Caps
Fill tube
Ceramic Support
and Insulating tube
Discharge tube

ILL. 5   THERMAL CONDUCTIVITY CELL DETAIL

Potential lead

Potential lead

.003" Dia. Platinum

5.887"

Active Length of Heater

Potential lead

.010" Dia. Platinum

Glass Envelope

Stainless Steel Weight

ILL. 6   FINITE SOURCE HEATER

3 start thread

60°

·25"

·063."

Detail of
Heating Wire
Grooves

Mat'l:
copper

6·00
Half length

1·25"

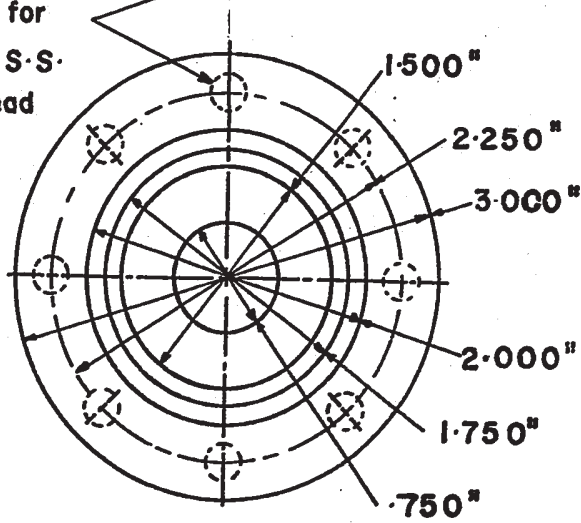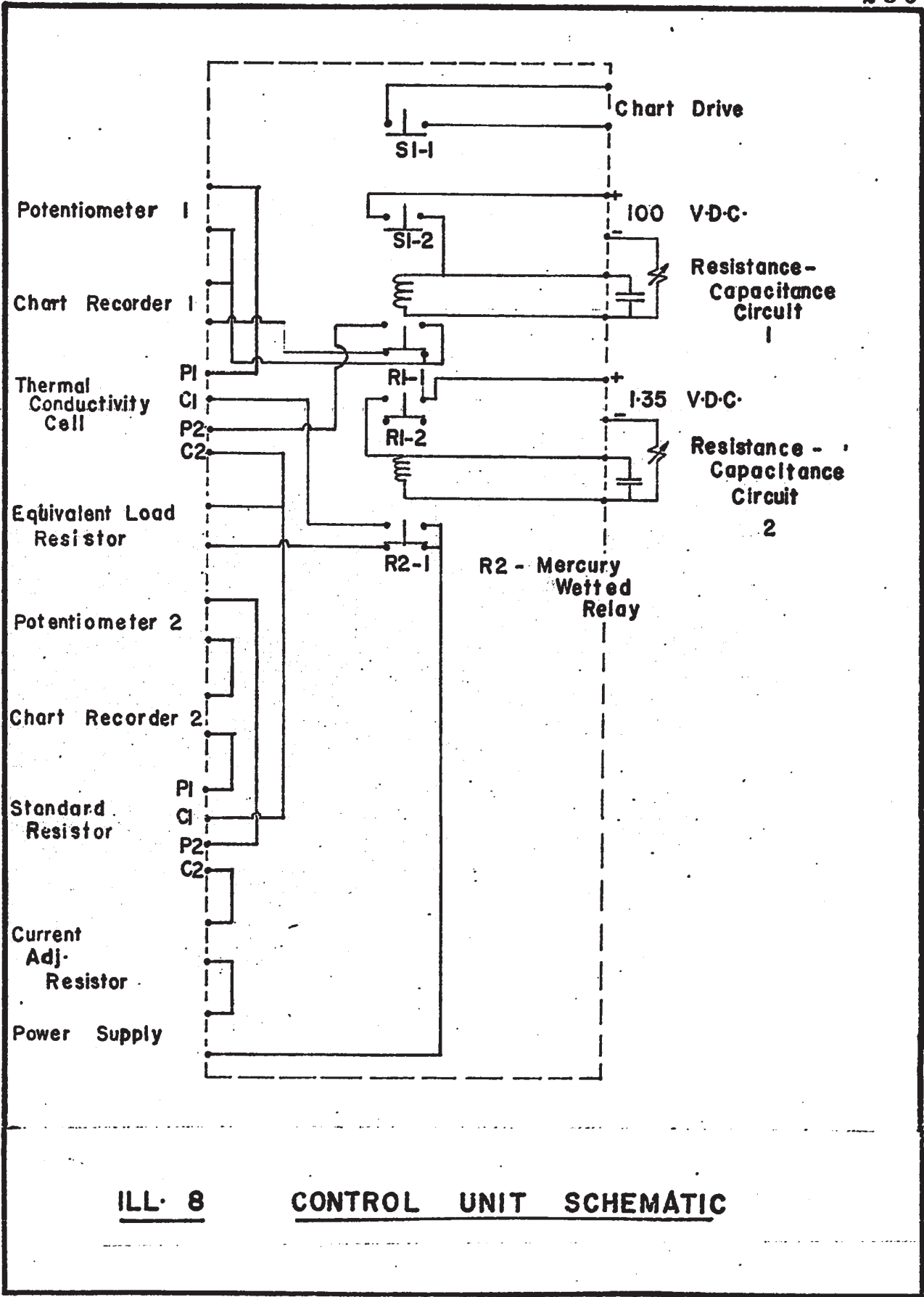·050"

Drill and tap 1/4–20
8 places for
1" long S·S·
socket head
cap's·

1·500"

2·250"

3·000"

2·000"

1·750"

·750"

2250"

3·000"

·50"
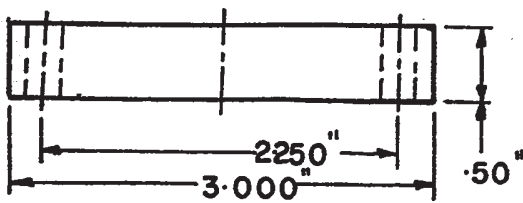
End Cap — 2 Req'd
Mat'l — copper

ILL · 7 —   PRESSURE   VESSEL   THERMOSTAT

ILL· 8   CONTROL UNIT SCHEMATIC

ILL. 9 — TEST DATA — TEST 66.-5., "VERMICULITE "

Thermocouple Response with Time.

Current Flow = 4692 amperes.

Copper Constantan Thermocouple
Ref. Jch. — 32 Deg. Fah.

Time (sec.)

Millivolts