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THE THERMAL CONDUCTIVITY OF POTASSIUM CHLORIDE AT ELEVATED TEMPERATURES

BY

WENDELL D. MILLER

A

THESIS

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Approved by William H. Bessey
Professor of Physics

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INTRODUCTION

The transport of heat in solids is attributed to the combined effect of the quantized vibrations of lattice points, or phonons, and the flow of valence electrons. In dielectric solids, the thermal conductivity is due almost entirely to phonons, while in metals the latter method preponderates. Scattering processes in dielectric solids, giving rise to thermal resistance, also serve to limit the thermal conductivity. Scattering results from vacancies, dislocations, interstitial atoms and phonon interaction. Crystal boundaries also function as scattering points and in crystals of small dimensions, this process is the most promounced conductivity-limiting factor. At low temperatures the thermal resistance of an ideal crystal is due almost entirely to boundary scattering while at high temperatures anharmonic coupling is the primary limiting factor. As the temperature increases, boundary scattering becomes less pronounced but anharmonic coupling increases. At some median temperature the conductivity is a maximum.

From theoretical considerations, Debye[1] was able to show the conductivity was approximately proportional to 1/T for temperatures above the point of maximum

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(1) Debye, P., Vortrage uber die Kineticbe Theorie, M. Planck et al, Teubner, Leipzig, p. 46, (1914).

conductivity. Analysis by Peierls[2], using quantum mechanics, resulted in the same reciprocal temperature dependence. Pomeranchuk[3] has proposed a theoretical treatment based on a more general law of sound absorption than previously used. These calculations show a mixed conductivity, proportional to between $1/T$ and $1/T^{3/2}$. Using considerations presented by Pomeranchuk, Peierls[4] has shown a conductivity proportional to between $1/T$ and $1/T^{2}$.

Considerable experimental work has been done in the past to determine the thermal conductivity of various alkali halides below the boiling point of water. Values determined by early experimenters differed widely, but these discrepencies are attributed to various major defects in the samples used. Measurements at the Missouri School of Mines and Metallurgy of the conductivity of sodium chloride above this temperature and to approximately 600°K indicate a relationship proportional to $1/T^{1.3}$. Therefore, it appears that if theory is to be substantiated by experimental

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(2) Peierls, R., Zur Kinetischen Theorie der Warmeleitung in Kristallen, Ann. Phys. (Leipzig), Vol. 3, pp. 1055-1101 (1929).

(3) Pomeranchuk, On the Thermal Conductivity of Dielectrics at the Temperature Higher than the Debye Temperature, Journal of Physics, Moscow, Vol. 4, pp. 259-268, (1941).

(4) Peierls, R., Quantum Theory of Solids, Oxford, (1955), pp. 50-52.

determinations, the conductivity of a single specimen must be examined to the maximum attainable temperature, i.e. the melting point of the substance.

Equipment built by Weisbrod[5] and improved by Brown[6] and Ohlsen[7] was available at the beginning of the investigation. Its design utilizes an absolute steady state method of measurement. Two nearly perfect cylindrical crystals of potassium chloride were used in the experiment, one approximately half the thickness of the other. The purpose of this investigation was to improve the operation of existing equipment and to extend the experimental data available on the conductivity of potassium chloride.

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(5) Weisbrod, Harold, Determination of the Thermal Conductivity of Sodium Chloride at Elevated Temperatures, Missouri School of Mines unpub. Masters Thesis (1954).

(6) Brown, Howard, The Thermal Conductivity of Sodium Chloride at Elevated Temperatures, Missouri School of Mines unpub. Masters Thesis (1955).

(7) Ohlsen, Paul, The Thermal Conductivity of Sodium Chloride Within the Temperature Range 375°K to 637°K, Missouri School of Mines unpub. Masters Thesis (1956).

REVIEW OF LITERATURE

The initial study of the thermal conductivity of insulating solids is attributed to Tuckschmidt[8], who obtained experimental values for several alkali halides. E cken[9], in 1911, reported several values for sylvite above and below room temperature. In 1950, Ballard[10] found two values slightly above room temperature. Perhaps the most significant work done so far with respect to experimental technique, temperature range and variety of specimens, was accomplished by Birch and Clark[11]. Values were reported for several alkali halides, but no work was done with potassium chloride. More recently, calculations from theoretical considerations have been made by Dugsdale and MacDonald[12] on the conductivity of various alkali halides. They are in fair agreement with observed values. These calcu-

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(8) Tuckschmidt, Ann. Phys. Beibl., Vol. 7, p. 490, (1884).

(9) Eucken, A., Ann. Phys. (leipzig), Vol. 32, p. 185, (1911).

(10) Ballard, S. S., Mc Carthy, K. A., and Davis, W., Review of Scientific Instruments, Vol. 21, No. 11, pp. 905-907, (1950).

(11) Birch, Francis, and Clark, Harry, The Thermal Conductivity of Rocks and Its Dependence Upon Temperature and Composition, Am. Journal of Science, Vol. 38, pp. 529-558, (1940).

(12) Dugsdale, J. S., and MacDonald, D., Lattice Thermal Conductivity, Physical Review, Vol. 98, No. 6, pp. 1751-1752, (1955).

lations were performed with the idea of verifying observations made concerning the departure of the lattice from harmonic behavior.

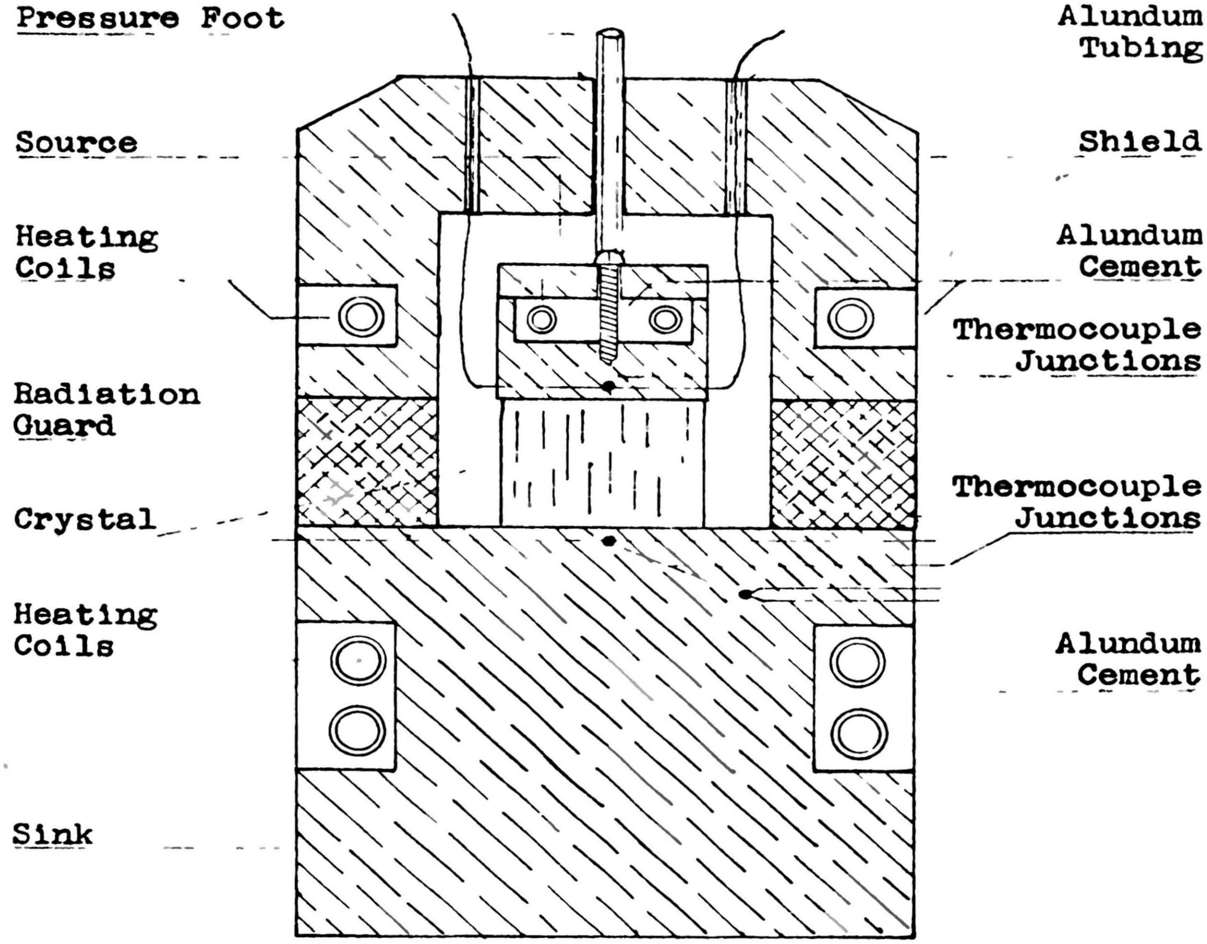
THE CENTRAL ASSEMBLY

The heat flow portion of the apparatus consists of a sink, radiation shield, source and source shield. The potassium chloride crystal is centrally located between the sink and the source and source shield. It is surrounded by the radiation guard, a hollow ceramic disc whose thickness is comparable to that of the crystal. The sink is a large cylindrical copper block, electrically heated to maintain any desired predetermined temperature by a thermoelectrically-controlled thyratron control circuit. The source shield, a cup-shaped copper affair, is symmetrically located over and around the source. It is separated from the sink by the radiation shield. Heating is accomplished electrically and is controlled so that the shield can be maintained at the same temperature as the source.

It is essential that good thermal contact be maintained between all touching components of the assembly. To insure this, lead foil, brought to its fusion point during an initial outgassing run, was placed between all contacting surfaces. This serves to eliminate air spaces caused by surface roughness. The pressure necessary to hold the source and crystal on the sink is supplied by a spring-fed section of alundum tubing extending through a small hole in the center of the source shield. Contact between the source shield, radiation shield and sink is maintained with a mica insulated coil spring. A cross-sectional diagram of the assembly is shown in Figure 1.

When thermal equilibrium has been established, all copper components are at the same temperature. As the source is heated and the temperature rises above the basic sink temperature, the source shield temperature, controlled by an opposing thermocouple circuit, rises a like amount. Hence the total radiation loss by the source to the immediate surroundings is essentially zero. The temperature gradient along the inner surface of the radiation shield, a refractory material of low thermal conductivity, is approximately the same as that along the exposed crystal surface. Thus, radiation losses from the crystal in a radial direction are minimized. Consequently, all heat developed in the source passes through the crystal and in a direction paralell to its axis. This permits calculation of the thermal conductivity to be made on the basis of one dimensional steady state heat flow.

The aforementioned portion of the apparatus is enclosed in a bell jar mounted on a sixteen inch pump plate. All necessary electrical and monitoring connections are made through seals on the bottom of the plate. The system is evacuated by a forepump to reduce heat losses by convection currents. Chromel-alumel thermocouples are used to measure and regulate temperatures.



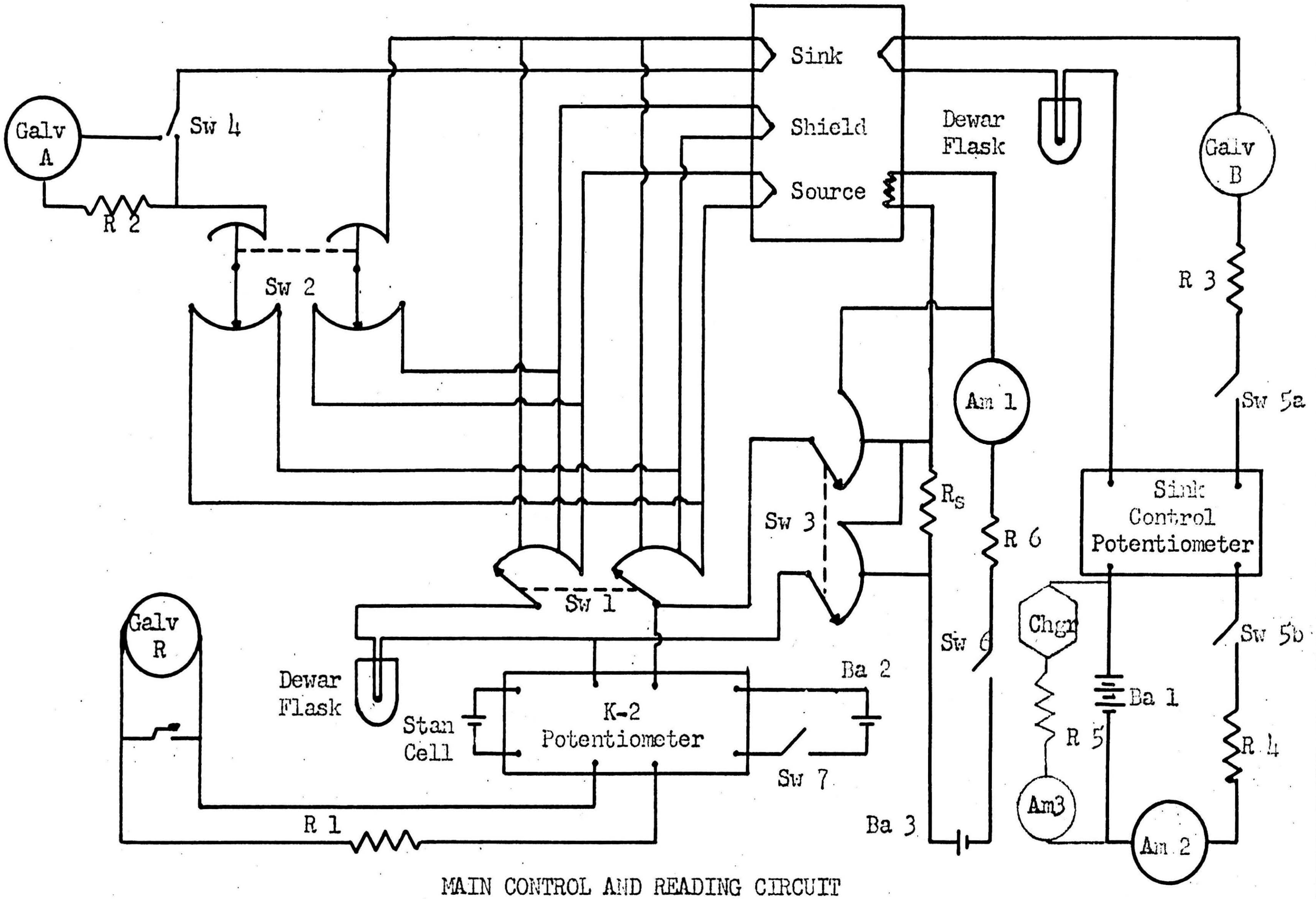
CROSS SECTION OF HEAT FLOW APPARATUS

Figure 1

THE CONTROL CIRCUITS

The initial phase of this work was concerned with placing the available equipment in a more usable form. All slidewire resistors, used to control sink and shield power supplies, were wall mounted. All switches were incorporated into a single panel. Silver contact rotary switches were used throughout the thermocouple circuits to minimize contact resistance and facilitate reading. To improve existing accuracy, a straight potentiometer method of determining the source power input was utilized. The reading galvanometer was wallmounted to reduce vibrational hazards. Since some AC and thermocouple circuits were close togeather, it was necessary to determing if there was any interaction between the two. Temperature measurements were made with the AC circuits on and then rechecked immediately afterward with the circuits off. No evidence of interaction could be detected in this manner. The revised circuit diagram is shown in Figure 2.

Sink and shield heating is accomplished almost entirely by AC current. Corrections necessary to maintain a constant temperature are supplied by small pulsating DC components supplied through a thyratron. Opposing thermocouples, one imbedded in each of the components to be regulated, are used to control the shield corrective input. A temperature difference be-



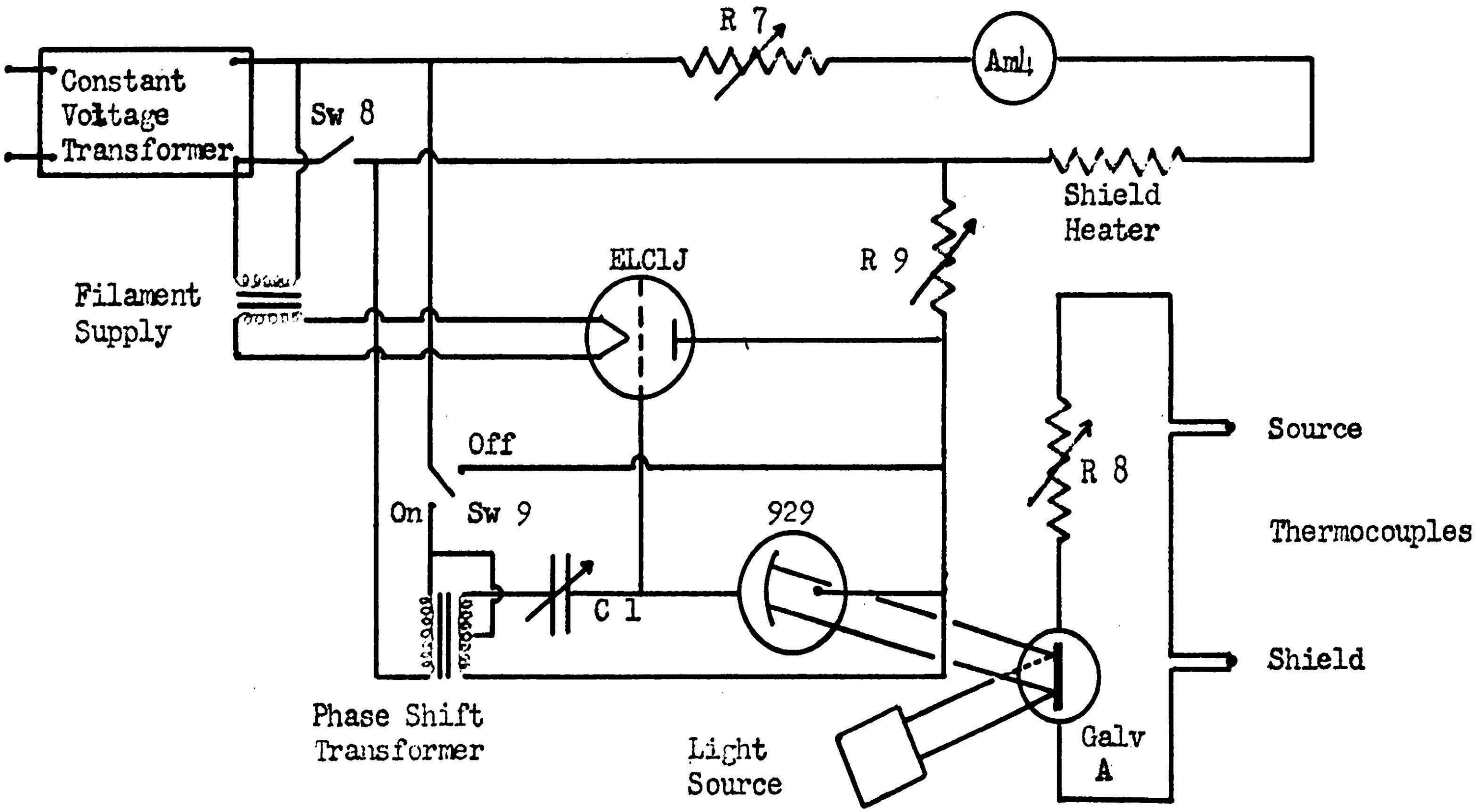
MAIN CONTROL AND READING CIRCUIT

Figure 2

tween the source and shield results in a current through a control galvanometer. A converging beam of light from a stationary source is reflected from the mirror and forms a wedge shaped image on a photocell grid. As the galvanometer deflects the image plays across the photocell grid, decreasing its resistance as the width of the wedge increases. The tube then fires, supplying the corrective current necessary for reduction of the temperature difference. As the correction is made, the above process is reversed, and when temperatures are matched the tube quenches. This control circuit is shown in Figure 3.

The sink temperature is maintained by a control circuit similar to the one previously discussed. In this case the output of a single thermocouple, imbedded in the sink, is bucked against the output from a potentiometer, used as a divider. This circuit is shown in Figure 2. The thyratron circuit is identical to the one used in the shield control.

To improve existing accuracy, a straight potentiometer method of determining the source power input was incorporated. The switching arrangement was such that the same potentiometer used to determine temperature equivalents could be used. The circuit current was evaluated by measuring the drop across a standard 10.00 ohm resistor.



SHIELD TEMPERATURE CONTROL CIRCUIT

Figure 3

OPERATING PROCEEDURE

Operation of the apparatus will be explained with the aid of Figures 2 and 3. First, all switches are placed in the off position and all battery connections secured. The forepump, constant voltage transformers and light sources are then turned on. Ba 1 and Ba 2 should be turned on as soon as possible so that the terminal voltage drift of these batteries will be negligible when data is to be taken. With Sw 5b on, R 4 is adjusted so that the current thru Am 2 is approximately 20 milliamperes. The battery charger is then turned on and R 5 is varied so that the current indicated on Am 3 is the same as through Am 2. When the system pressure has been reduced to approximately 10 microns, Sw 8 and Sw 9 are turned on and assembly heating begins. With the operating temperature chosen, R 7 is varied until the estimated current necessary to maintain this level is indicated on Am 4. The control circuit is then zeroed in by the following process. The photocell is moved on its track so that only the point of the wedge-shaped image falls on the grid. C 1, a variable condenser, is adjusted so that the thyratron flickers. The position is noted and then changed so that a maximum amount of light falls on the photocell grid. This should produce an increase of approximately .03 amperes in the heater current, indicated by Am 4.

The current increase can be adjusted by varying R 9. The photocell slider is then returned to its original position and control sensitivity is checked. If, when the image oscillates slowly past the photocell, the thyratron fires and quenches at the same position, the control circuit is ready to operate. If not, R 9 and C 1 must be changed until a more suitable combination is found. Sw 9 is then turned off, placing R 7 and R 9 in paralèll. This increases the basic heater current and provides faster initial heating. At the same time and in the same manner the sink is brought to the operating temperature.

The reference junction of the thermocouple circuit is prepared by filling the Dewar flask with crushed ice and distilled water. The K-2 potentiometer is then balanced and the temperature rise of the various components noted. Individual thermocouple circuits can be routed to the potentiometer by Sw 1. When the operating temperature is reached, Sw 9 is moved to the on position and the temperature checked for a short period. At this time it may be necessary to make minor corrections to the base sink or shield current to stay at the selected operating temperature. When it is apparent the base currents are correct, Sw 5a is closed and the sink control potentiometer output adjusted until the wedge of light deflecting across the photocell is at the preset zero position. At this point the thyratron will be on the verge of firing. Decreasing the basic sink current .01 to .02 amperes completes the operation of placing the thyratron circuit in control of the sink temperature.

To effect control of the shield temperature, Sw 1 is turned off and Sw 2 moved to the Shield-Source position. With R 2 set at the maximum value (10^4 ohms), Sw 4 is closed and R 2 reduced slowly to a position where control sensitivity is sufficiently fine. The shield base current is then reduced .01 to .02 amperes.

The ability of the apparatus to effect and maintain steady state conditions depends a great deal on the selection and adjustment of the base currents. If the thyratron is overloaded by continued firing in the maximum range, the temperature will oscillate excessively about the intended point. This can be corrected by increasing, very slightly, the base current. On the other hand, when the base current is too high, control sensitivity is reduced and fluctuations in the line frequency and voltage may increase the base power to a point where control is lost.

After control has been attained, steady state conditions will be reached within an hour or two and data can be taken. Power is supplied to the source by closing Sw 6. The power input is measured by utilizing

Sw 3, first measuring the drop across Rs to determine the circuit current and then the drop across the source heater coil to determine the voltage. Am 1 is included only as a monitoring instrument, to eliminate excessive potentiometer manipulation. In approximately one-half hour steady state conditions will again prevail and data can be taken. To establish linearity (see calculations), data is then taken for another power setting for several runs.

The source and shield thermocouples have two functions, temperature determinations and control of the shield temperature. Therefore, when either temperature is being read the control circuit is temporarily out of action. If measurements are made quickly, this fault is not serious and control is not lost.

BACKGROUND

When steady state conditions have been established and with no power input to the source, all components of the central assembly should be at the same temperature. Such is not the case and the existing temperature difference between source and sink is termed background. As suggested by Ohlsen[13], this difference might be due to the method used in making thermocouple junctions, in the method of installation and in the placement of thermocouples in the various components. He also noted that it was very difficult, if not impossible, to prepare two thermocouples that would develop the same emf at the same temperature.

In an effort to gain some insight into the problem of thermocouple placement, the following experiment was performed. An electrically heated copper cylinder, similar to the sink, was constructed. No control circuit was used, final temperature being regulated by a series resistance. Five holes, 4 millimeters deep and in multiples of 5 millimeters from the center, were drilled in the upper surface of the cylinder. A spiral placement pattern was used so that radial lines would be broken in only one place. Using one thermocouple, the temperature at these five points was measured at the surface and at a depth of 4 millimeters. One

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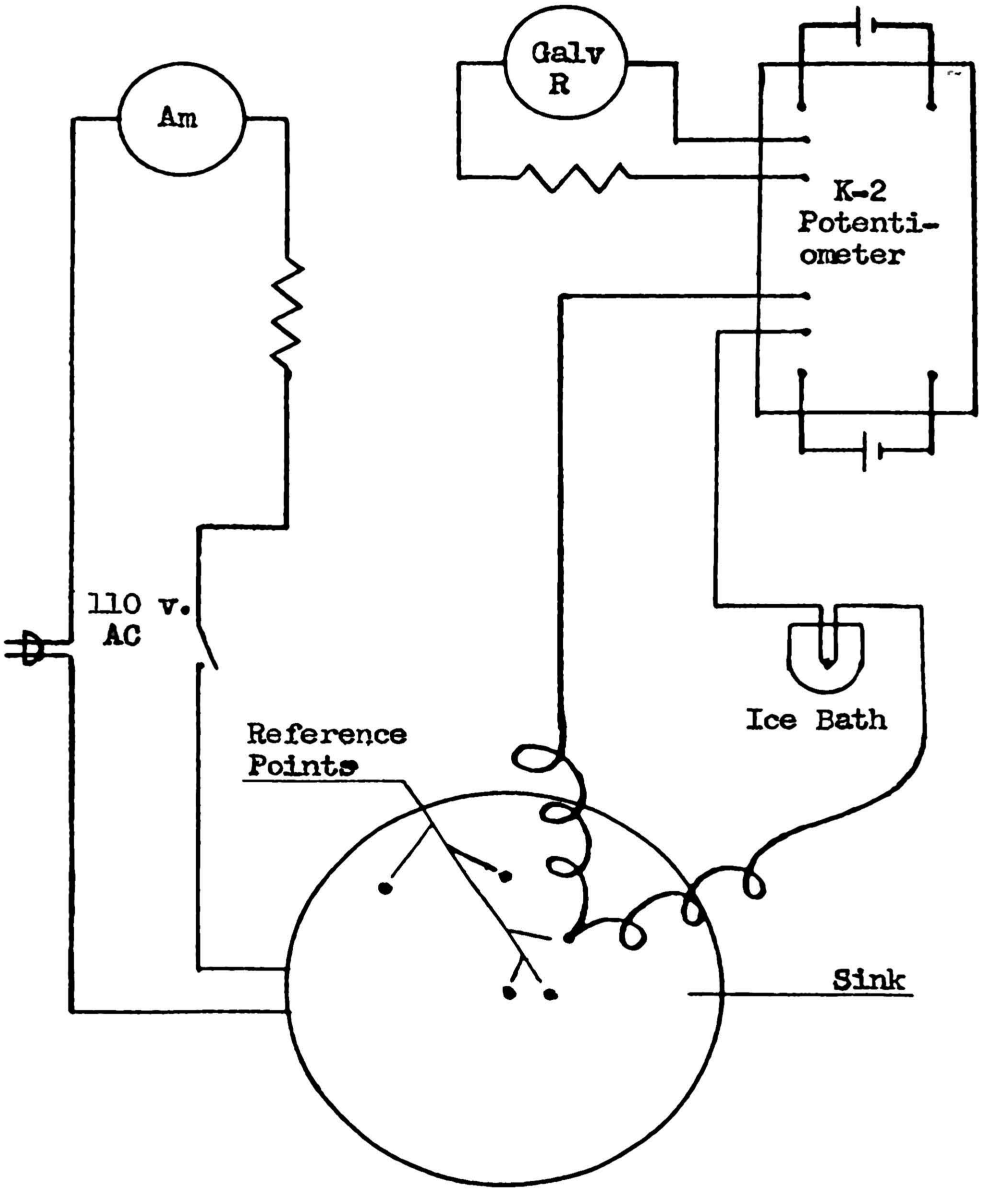
(13) Ohlsen, op. cit., p. 19.

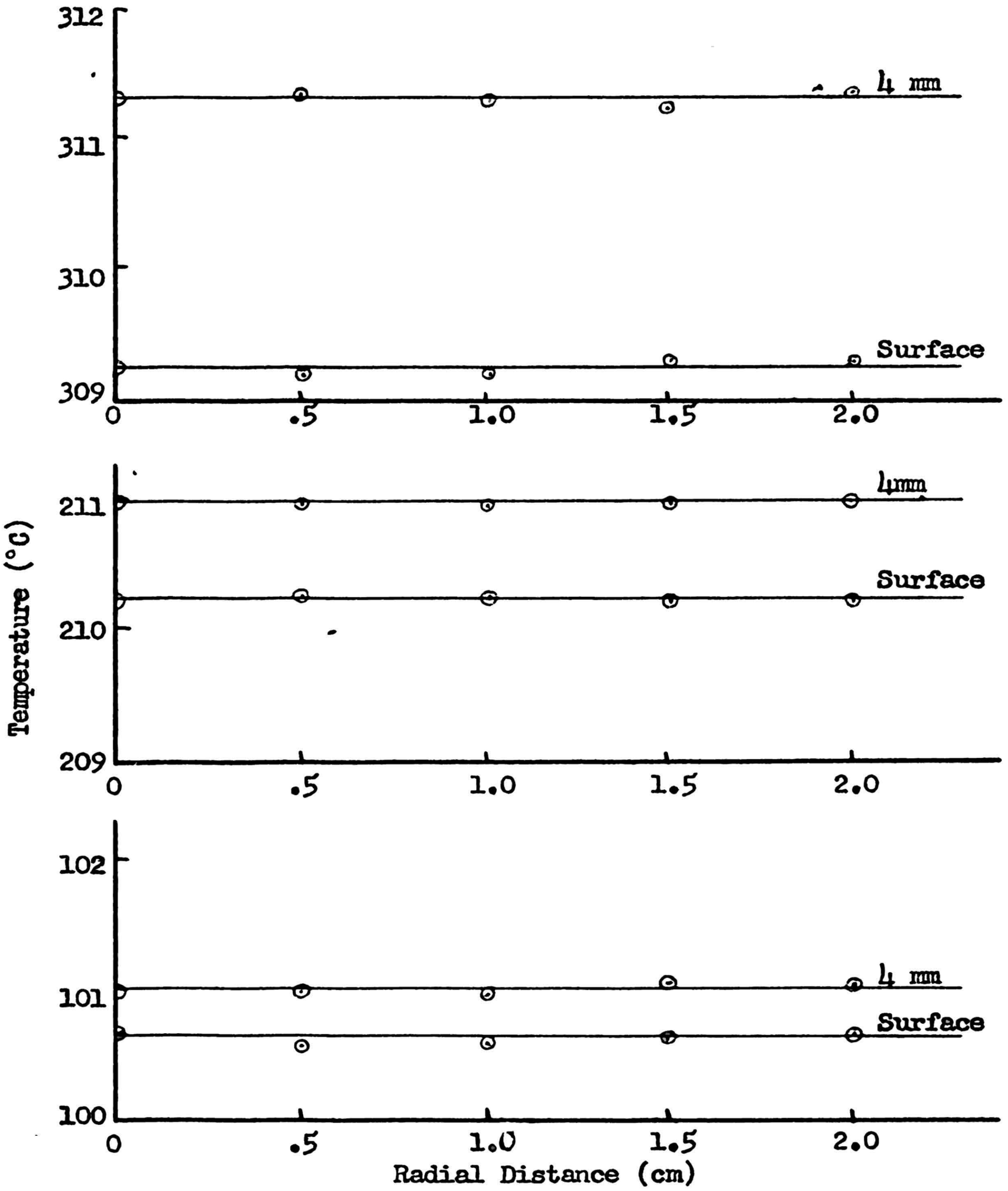
thermocouple was used so that individual inconsistencies could be eliminated. The diameter of the holes was such that the thermocouple junction would slide in and out easily. This necessitated the use of an interface medium to promote good thermal contact. Silicone vacuum grease, silicone oil, and napthalene were tried but were not effective. Finally, a ternary alloy, composed of one-third bismuth and two-thirds lead-tin solder, was prepared and proved satisfactory. The melting point of this alloy (approximately 98°C) was low enough to allow readings to be taken at the first conductivity data point. A diagram of the apparatus is shown as Figure 4.

A graphical representation of the results is shown in Figure 5. This would indicate that radial distance is not too critical but vertical displacement might be an important factor. The experiment was performed in air and without any surface shielding, so convection and radiation losses would be at a maximum. In actual practice, the source and sink thermocouples are approximately the same distance from their respective component surfaces. However, the source thermocouple is installed by silver soldering it in place while the sink contact is made by tightening a copper screw on the thermocouple junction. Neither method is entirely feasible for both installations.



RADIAL TEMPERATURE DETERMINATION APPARATUS

Figure 4



TEMPERATURE VERSUS RADIAL DISTANCE

Figure 5

Hence it appears that as long as some semblance of symmetry is maintained, the exact point of installation is not too critical. The method of preparation and installation appears to be the greatest factor contributing to background. So far, no particularly elegant solution to this problem has been devised.

CALCULATIONS

Calculations are made on the basis of the one-dimensional steady state heat flow equation,

$$Q = kA(T_2 - T_1)t/L.$$

In the CGS system, Q is expressed in calories; A, the cross-sectional area, in centimeters squared; $(T_2 - T_1)/L$, the thermal gradient, in °C/cm; and t, time, in seconds. The use of electrical heating suggests replacing the ratio Q/t with electrical units. The thermal conductivity, k, is then expressed as

$$k = LP/(T_2 - T_1)A,$$

where P is the source power input in watts.

Determination of the quantity $(T_2 - T_1)$ cannot be made in one data stand due to the presence of background. The thermal conductivity is not a linear function of temperature and, from an overall viewpoint, the ratio $P/(T_2 - T_1)$ is not a constant. However, for limited temperature increases (approximately .5°C) this ratio is essentially constant. Thus if thermal equilibrium is established twice, once with no power input, and again with a power input sufficient to cause an increase in temperature of approximately .5°C, the ratio $P/(T_2 - T_1)$ can be replaced by $\Delta P/\Delta T$. In this case, ΔT is the temperature difference resulting from a change in power input, ΔP. Information as to the reliability of data and the exchange of radiant energy between assembly components can be obtained by establishing thermal equilibrium a third time, with a different power input to the source. Graphically, temperature difference versus power input for a particular base temperature should be essentially a linear function.

Data collected to determine the source power input must be corrected for lead losses. The leads, that part of the resistance wire not enclosed by the source and extending through the shield, are joined to copper supply wires by presses just outside the shield. Their total length is approximately 5.55 inches. Using the resistance specified by the manufacturer corrected for temperature, the drop across the leads is determined and subtracted from the total drop across the resistance element. The product of this corrected factor and the circuit current gives the true power input.

RESULTS

Data and results for two different potassium chloride crystals are shown in table 1. All values have been corrected for deficiencies noted elsewhere. A logarithmic plot of thermal conductivity versus temperature for the full crystal is nearly linear, with a slope of -1.23. A like plot for the half crystal indicates a slope of -1.29, but a conductivity differing from the full crystal by nearly a factor of two. These plots are shown in Figure 7. It was expected that the conductivity of the thinner crystal would be less than that of the full crystal, but not to the degree exhibited. Inspection of the half crystal upon disassembly showed a rather large line defect running almost diametrically across the lower face. This defect was not noted before assembly, but was distinctly visable later due to an accumulation of the lead interface medium in the groove. It is believed that this defect, possible aggravated by heating and pressure, was the cause for a portion of the difference in the reported values.

If radiant exchange between the crystal and the radiation guard was appreciable, then the loss by the full crystal would be greater than by the half crystal. This would cause the reported conductivity to be higher than it should be. Thus the lower conductivity value

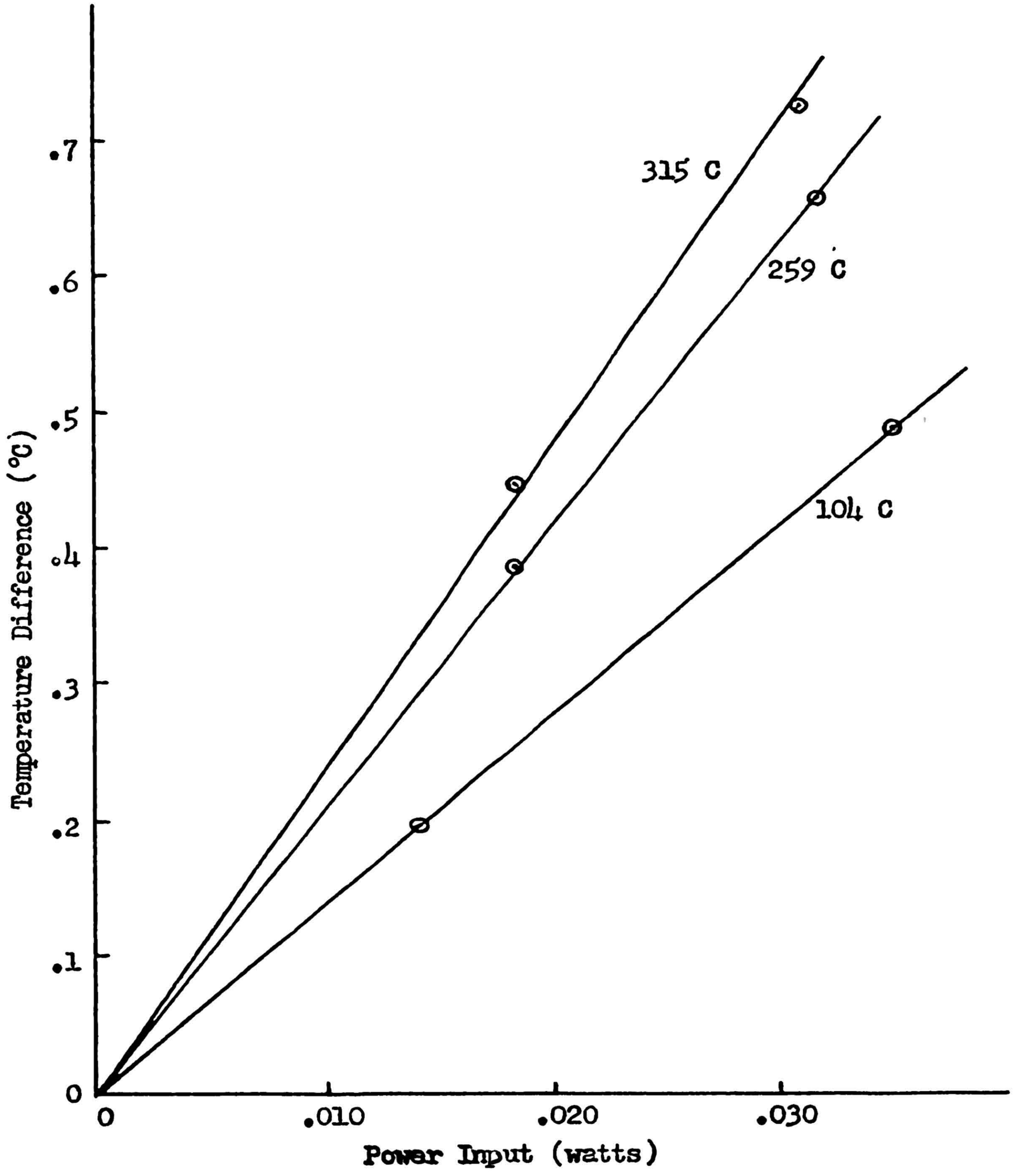
	Power Input (watts)	Temp. Dif. (°C)	$\frac{\text{Power}}{\text{Temp. Dif.}}$ (watts/°C)	Temp. (°K)	k $\left(\frac{\text{watts}}{\text{cm }^\circ\text{C}}\right)$
Full Crystal	.0143	.195	.0716	377	.0532
	.0352	.487			
	.0190	.293	.0650	418	.0482
	.0261	.478	.0546	479	.0405
	.0188	.385	.0471	532	.0349
	.0319	.663			
	.0186	.446	.0449	588	.0309
	.0310	.722			
	.0206	.512	.0402	626	.0298
Half Crystal	.0285	.342	.0834	382	.0300
	.0310	.529	.0586	480	.0211
	.0307	.605	.0507	589	.0183
	.0199	.495	.0402	659	.0145

THERMAL CONDUCTIVITY OF POTASSIUM CHLORIDE

Table 1

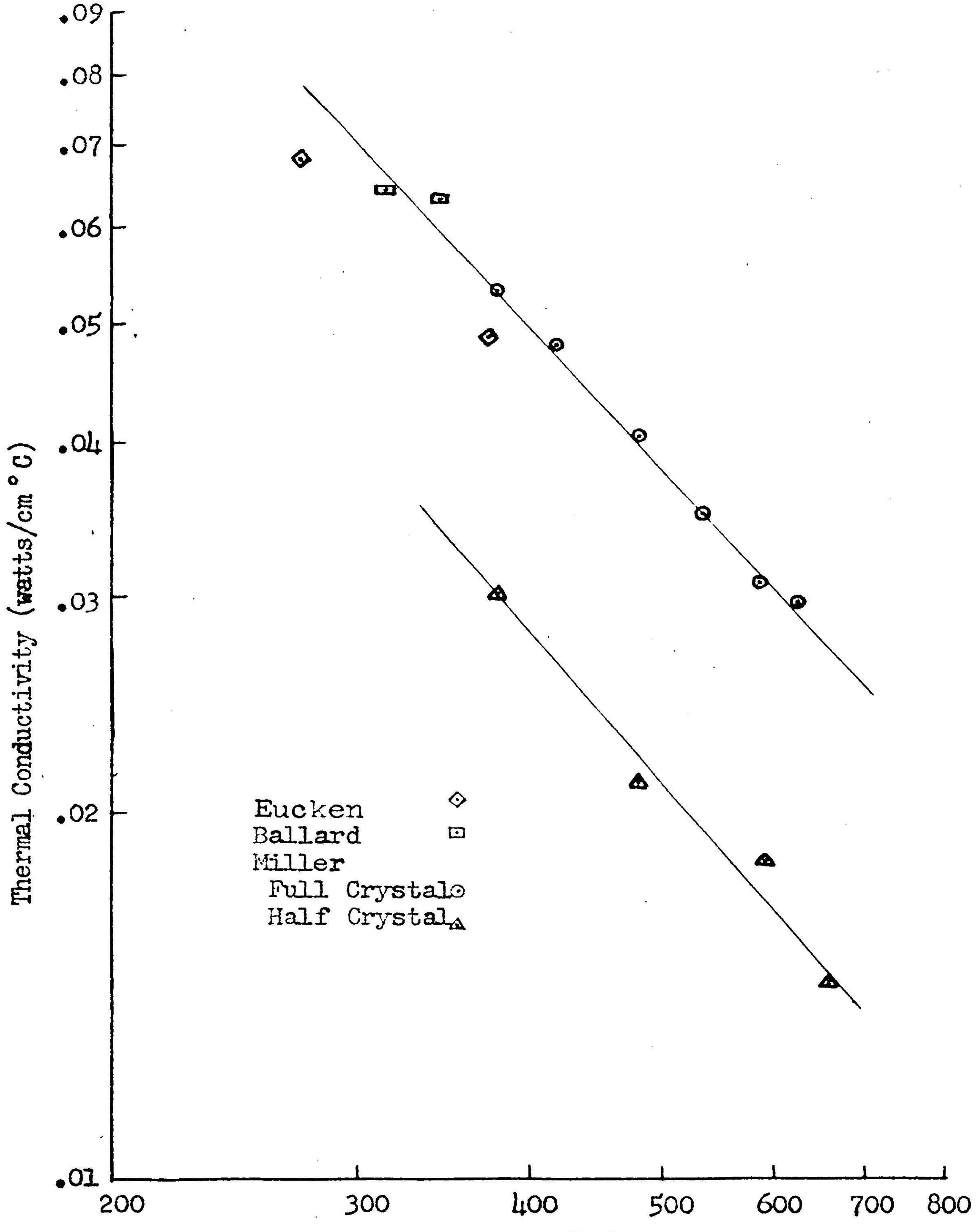
of the half crystal may be an indication axial flow conditions do not exist.

Data reliability can be checked by inspection of Figure 6. The deviation from linearity at higher temperatures is mainly due to the inability to establish steady state conditions to the same degree as at lower temperatures.



TEMPERATURE DIFFERENCE VERSUS POWER INPUT

Figure 6



THERMAL CONDUCTIVITY VERSUS TEMPERATURE

Figure 7

ERRORS AND SUGGESTED IMPROVEMENTS

Energy exchange between the source and shield due to a temperature difference will introduce an error as to the actual strength of the source. Calculations of the radiant losses by the Stefan-Boltzman equation are questionable since the exact degree of mismatching is not known. However, the same temperature difference will exist for several source power values at one operating temperature. By using multiple data and treating background as an additive constant, the effective source power and actual temperature difference can be accurately determined. Background corrections were made on all data and the error introduced by this fault is therefore slight. The temperature difference is also affected by observable drifts in the measured temperature. These cyclic fluctuations, .01 to .02°C in amplitude, are largely compensatory in nature since numerous data is taken over a period of time. The sensitivity of the reading circuit is such that temperature changes of .005°C can be detected. Error contributed by the above factors has been estimated at a maximum of 3%.

Heat loss through source lead wires has been estimated at less than 1%. Normal crystal imperfections and poor thermal contacts contribute an estimated maximum error of 5%. Error in wattage has been reduced to less than .25%. Correction for thermal expansion eliminated an estimated error of .5% per 100°C temperature rise.

It has been noted that improper thermocouple installation is probably the main factor contributing to background. Since no remedy to this problem is apparent, it seems advisable to make correction for the fault after installation is made. To date, all temperature readings have been determined using thermocouple calibrations supplied by the wire manufacturer. Calibration of the individual component thermocouples at several temperatures would supply information which might explain the apparent temperature differences now observed. These corrected calibration curves could then be employed in the actual experiment, eliminating the necessity of taking multiple data.

For operation at higher temperatures (above 400°C) adequate thermal shielding must be provided for the central assembly. Rapid dissipation of energy from external surfaces requires that corrective factors be more extreme. This, in turn, greatly increases the cyclic temperature drift. An electrically heated enclosure maintained at a temperature slightly lower than the operating temperature would appear to be the most obvious solution to this problem.

SUMMARY

An apparatus for measuring the thermal conductivity of a poorly conducting substance, utilizing an absolute steady state method, has been described. The general performance and reliability has been improved by redesigning and rebuilding a major portion of the original equipment. A side investigation was performed to determine the radial temperature distribution within a heated cylinder and to see what bearing this has on thermocouple placement. Conductivity measurements were made on two crystal specimens of potassium chloride. Results indicate a conductivity proportional to approximately $T^{-1.25}$ for both specimens, but differing numerically by nearly a factor of two. Suggestions were made that would lend further refinement to the apparatus and extend its operating range appreciably.

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VITA

Wendell D. Miller, son of Mrs. Arthur Bethke, was born in Cherokee, Iowa on September 13, 1928. Following graduation from Sutherland High School in 1946, he enlisted in the United States Army for two years. After military service, he entered Buena Vista College in September 1949. Military service interupted his formal schooling in October 1950, when he was recalled to active duty with the enlisted reserve. After 14 months he was separated from service and continued his education at Buena Vista College. Majoring in Physics and Mathematics, he recieved his Bachelor of Science degree in February 1955. He then entered the Missouri School of Mines and Metallurgy as a Graduate Assistant in the Department of Physics.