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NAVORD REPORT 6094

AN ADIABATIC CALORIMETER FOR THE DETERMINATION OF THE SPECIFIC HEATS OF CASTING RESINS

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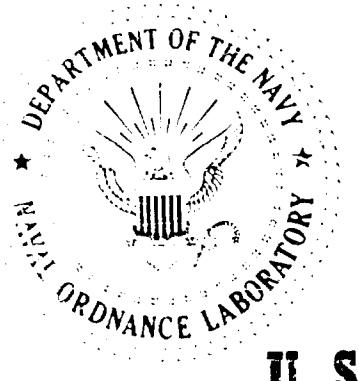

U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

NAVORD Report 6094

AN ADIABATIC CALORIMETER FOR THE DETERMINATION OF THE SPECIFIC HEATS OF CASTING RESINS

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ABSTRACT: This report describes an adiabatic calorimeter, with temperature controlling and measuring instrumentation, which has been developed for the determination of the specific heat of casting resins as a function of temperature over the range 30°C to 160°C. Results are presented for a number of typical casting resins. In some resins changes in specific heat values are observed which are indicative of second order transitions.

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This report describes the design, calibration and use of a adiabatic calorimeter used for obtaining the specific heats of casting resins as a function of temperature. Sample data are presented for a number of resins. The values of specific heat obtained by this instrument are to be used in calculating the heats of polymerization of casting resins. This study was done under project NIO_{g}-1-56 as part of a general resin investigation program being carried out by the Non-Metallic Materials Division of the Naval Ordnance Laboratory.

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Captain, USN
Commander

Albert Lightbody

A. LIGHTBODY
By direction

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ILLUSTRATIONS

AN ADIABATIC CALORIMETER FOR THE DETERMINATION OF THE SPECIFIC HEATS OF CASTING RESINS

INTRODUCTION

1. A knowledge of the specific heats of casting resins is necessary in predicting temperature rises during polymerisation in applications involving the use of a casting resin in the encapsulation of temperature sensitive electronic components. Specific heat data as a function of temperature are important in calculating both the heat of polymerization and thermal diffusivity of these materials. In addition, such data are indicative of changes in the thermal, electrical and mechanical properties at elevated temperatures and appear to be related to the second order transitions of these resins.

2. Very little work has been done on determining the thermal properties of synthetic resins, and a systematic study of the thermal properties of casting resins has not been previously undertaken. Dole (reference (1)) and co-workers have determined the specific heats of a few synthetic resins including polyethylene, and styrene has been studied by Ueberreiter and Otto-Laupenmuhlen (reference (2)). The need for similar data on casting resins has become highly important as the use of these resins rapidly increases in ordnance applications, often under high temperature conditions.

3. The heat capacity of a material may be defined as the quantity of heat necessary to produce a unit change of temperature in a unit mass of the material. The specific heat, Cp, of a material is the ratio of its heat capacity to that of water at 15°C. If a quantity of heat in calories, Q, is required to raise the temperature of M grams of material, maintained adiabatically in the calorimeter, from T_1 to T_2, the average specific heat of the material between T_1 and T_2 may be calculated by means of Equation (1):

$$Cp = \frac{Q}{M(T_2 - T_1)} \qquad (1)$$

4. A calorimeter has been constructed for the purpose of determining the specific heats and heats of polymerization of casting resins. The design of this instrument is based on the maintaining of adiabaticity between the resin sample in the calorimeter vessel and the calorimeter wall during the determination of the specific heat of the resin. The temperature of the resin sample is increased continuously by means of an embedded resistance coil which is connected to a source of power. Adiabatic conditions are maintained by electrically heating the calorimeter wall slowly so that the wall temperature is within 1°C of the temperature of the resin sample at all times during the determination.

5. The calorimeter described here was designed to obtain engineering data. More emphasis has been placed on obtaining the data with a minimum expenditure of time than on the highest obtainable accuracy. The calorimeter has been so constructed as to be versatile and simple in operation and can be operated by a single technician. The data obtained are accurate to within 5%. The use of this instrument in obtaining heats of polymerization will be described in a subsequent report. This work is part of a long term study of casting resins being undertaken in an effort to obtain a basic understanding of the properties and polymerization characteristics of these compounds. Other reports have already been prepared on the electrical properties of these resins (references (3) and (4)) as well as on test methods (reference (5)) for studying the properties of casting resins.

DESCRIPTION OF CALORIMETER AND CALORIMETER VESSEL

6. The calorimeter used in this investigation consists of two concentric cylinders separated by a 1.125" dead air space. The larger outer cylinder 6.0" in diameter, is constructed of 0.125" thick brass and closed off at the bottom with a 0.125" thick brass plate which is brazed to the cylinder. The smaller inner cylinder, 3.5" in diameter, is built of 0.062" thick copper and is closed at the bottom with copper sheet 0.062" thick brazed to the bottom of the inner cylinder. Both inner and outer cylinders are nickel plated to minimize radiation losses. The inner cylinder rests on a cylindrical piece of Teflon 1.375" thick and 2" in diameter. This block serves both to support the inner cylinder and to separate it from the outer cylinder. The overall dimensions of the calorimeter are 6.0" in diameter and 8.0" high.

7. Wrapped around the outside of the inner cylinder is a thin layer of asbestos. This serves to insulate the cylinder from the heating coils which are then placed around the cylinder. This heating coil, which serves to maintain adiabaticity during both the specific heat and heat of polymerization runs, consists of #16 nichrome resistance wire. The leads to this coil are passed through a small hole 0.25" in diameter in the outer cylinder to the instrumentation which controls the adiabatic jacket.

8. The top of the calorimeter consists of two plastic covers. The first cover, of linen base phenolic plastic, is screwed down tightly into the outer cylinder and fits very closely against the inner cylinder. Thus the inner cylinder is held in place by means of this cover, which also serves to close off the 1.125" air space between the two cylinders. The second cover is 0.75" thick and is constructed of phenolic plastic. It is fitted very tightly into the first cover and serves to close the inner cylinder of the calorimeter. Two holes 0.125" in diameter are drilled through this cover and serve to admit the thermocouples and the leads of the resistance coil. During a specific heat determination these holes are closed by small pieces of rubber which fit very closely against the leads. Figure 1 shows the calorimeter setup for a specific heat determination.

9. For specific heat determinations, the calorimeter vessel (sample holder) consists of a standard #1 can, 2.75" in diameter and 4" high. This is shown in Figure 2. The can, resistance coil, and thermocouples are carefully weighed to the nearest gram before the resin is mixed and poured into the cell. A thermocouple (iron-constantan) is taped to the inside of the can, halfway between the top and bottom. A previously prepared resistance coil of #16 nichrome wire, consisting of 22 turns and having a resistance of about 2.5 ohms, is so suspended in the can as to be in the approximate center of the can, raised from the bottom of the can 0.75". This coil is made on a spring winding machine and is 3.0" high and 1.5" in diameter. It is shown in Figures 3 and 4 being inserted into the can. Before the coil is encapsulated by the resin under study, it must be firmly held in place in the can. A small jig consisting of a ring stand and three clamps has been found satisfactory.

10. Immediately before the liquid resin is poured into the can containing the suspended resistance coil, a thermocouple is placed in the geometric center of the can and held securely in place by means of one of the clamps on the jig. Care must be taken that this thermocouple is at the exact center of the can and does not move before or during the polymerization of the resin. If the coil is placed correctly in the can, the thermocouple will be about 0.5" from the resistance coil. It is this thermocouple which will be used to measure the rise in temperature of the sample during the course of a specific heat determination.

11. In the specific heat determinations, heat is developed in the sample so slowly that the entire mass of resin rises in temperature at a uniform rate. Thus, the possibility of thermal gradients existing within the casting are minimized. This has been checked many times by embedded thermocouples at different places in the resin and on the heating coil. In addition, no overheating has been noted in the immediate area around the coil.

DESCRIPTION OF INSTRUMENTATION

Introduction

Temperature Measurements

12. In an adiabatic process, which by definition is one in which no heat exchange takes place between an object and its surroundings, the accurate measurement and control of the temperature is very important. Many different methods have been employed in calorimetry for determining the temperatures of the sample, the calorimeter wall and jackets, and other necessary measurements. Among the more important temperature measuring methods are electrical resistance and mercury thermometers, thermopiles and thermocouples. These methods have been discussed by Swietoslawski (reference (6)) and their use in adiabatic calorimetry described in detail. In this work it was decided to use iron-constantan thermocouples for all the temperature readings. The range of the measurements and the fact that suitable calibrated potentiometers were available reinforced this decision.

13. The temperature measurements can be divided into two types. In the first are the measurements of the rise in temperature of the resin sample during the specific heat determinations due to the heat imparted to the resin by the resistance coil. This rise in temperature must be determined very accurately. The second case consists of the measurements of the calorimeter wall, the calorimeter jacket and vessel, and the outer part of the resin mass. These measurements need only be accurate to within 1°C and, hence, can be measured by an ordinary recording potentiometer.

The Brown Recording Potentiometer

14. The measurements of the calorimeter walls, vessel and jacket, and outer part of the resin during the specific heat determinations are made by a Six Point Brown Electronik Recording Potentiometer Model #153X64A6-X-42. The details of its operation will be found in reference (7). This instrument, which is calibrated for iron-constantan thermocouples, prints the temperature of six thermocouples on chart paper as a function of time. In Figure 5 this instrument is shown at the right. This recording potentiometer is used to measure the temperature at various places within the sample and calorimeter and will rapidly show any thermal gradients if present.

The Leeds and Northrup Potentiometer

15. The measurements of the rise in temperature of the resin sample during the specific heat determinations are made on a Leeds and Northrup Model #8662 Portable Precision Potentiometer. A complete description of the operation of this instrument will be found in reference (8). With this instrument an EMF as small as 0.001 millivolt may be measured. In a specific heat determination the EMF varies from 1.200 millivolt to about 8.500 millivolts. These are converted to degrees Centigrade by standard tables (reference (9)). Before use, this instrument is carefully checked, the standard cell is checked, and the galvanometer balanced. To avoid error, a reference junction is maintained in an ice-water mixture in a vacuum flask kept beside the potentiometer during the determination. A picture of the potentiometer and vacuum flask is shown in Figure 6.

Control of Adiabaticity

Introduction and Principle

16. In calorimetry the term "adiabatic" indicates that the temperature of the calorimeter wall (jacket) is changed so that the temperature of the wall will be equal to that of the calorimeter vessel throughout the experiment. Thus, the sample in the calorimeter vessel will neither gain nor lose heat from the surroundings. In the calorimeter described in this report adiabaticity is accomplished by the technique of heating the jacket by means of the nichrome resistance wire wound around the inner wall of the calorimeter, thereby keeping it at the same temperature as the sample within 1°C.

Instruments

17. The maintenance of adiabaticity during a specific heat determination is accomplished by means of a Brown Circular Recorder Model 151321. This recorder monitors a thermocouple taped to the wall of the calorimeter vessel and differentially connected to a thermocouple taped to the inner wall of the calorimeter. These bucking thermocouples are shown in Figure 7. If both of the thermocouples are at the same temperature the Brown Circular Recorder will show a zero setting. However, if the calorimeter vessel increases very slightly in temperature (less than 1°C) over that attained at the same time on the calorimeter wall, an EMF is set up in the differentially connected thermocouple circuit. This EMF on being received by the recorder activates a micro switch by means of a cam gear on the center shaft of the recorder. This completes a circuit which then heats the calorimeter wall. This circuit remains closed as long as a difference in temperature exists between the calorimeter vessel and calorimeter wall. When the wall temperature has increased to the point that no EMF is set up by the differentially connected thermocouples, i.e., both vessel and wall are at the same temperature, the microswitch automatically opens and remains open as long as there are no temperature differences. A Variac has been incorporated into the circuit to give a fine control to the heating circuit, thus compensating for any temperature lag which may occur. The Brown Circular Recorder may be described as a null balancing instrument. Several minutes after the start of an experiment, adiabatic conditions can be attained by careful use and control of this instrumentation. This can be shown by comparing the temperatures of the center of the sample with that of the calorimeter wall, being careful that both temperatures are measured at exactly the same time. The calorimeter heating circuit and control circuit is shown in Figure 8.

CALIBRATION OF THE CALORIMETER

18. Because the instrument is not absolutely adiabatic, it was necessary first to determine the calorimeter constant or, more correctly, the energy equivalent of the calorimeter. This constant can be determined by carefully measuring the apparent specific heat of a material of known specific heat as a function of temperature. The difference between the actual specific heat and the apparent specific heat is the calorimeter constant. The calorimeter constant as used in this report is a function of the temperature and represents a summation of the heat capacity of the calorimeter and any heat losses occurring during the experiment.

19. At the time this work was conducted, there was no resin whose specific heat as a function of temperature was reported in the literature. Since it was advisable to use a material of similar thermal properties when compared to casting resins in order to have a meaningful determination, a survey of the literature for specific heat data on various organic compounds was made. The number of possible compounds was not large. Benzoic acid has long been used as a calorimetric standard in both heat of combustion and specific heat standardizations. However,

its relatively low melting point (122°C) limited its use, since it was desired to make measurements to 160°C. The search of the literature revealed that this data was available only on succinic acid. Its specific heat had been measured over the temperature range 0°C to 160°C (reference (10)). This material is a white crystalline solid and has a melting point of 185°C. Therefore, it could be melted and cast in the calorimeter vessel containing the heating coil and thermocouples.

20. A study of the data shown in Figure 9 indicates that the apparent specific heat is greater than the actual value. However, the deviation from the actual value is a linear function of the temperature. The value of the specific heat of succinic acid as given in reference (10) is $0.248 + 0.00154T$ in the range 0°C to 160°C. The apparent specific heat of succinic acid as determined by this calorimeter was found to be $0.253 + 0.00174T$. The difference between the real and apparent specific heats is then $0.005 + 0.0002T$. Since the apparent specific heat is greater than the actual value, the term $(0.005 + 0.0002T)$ must be subtracted from the measured value. The temperature, T, must be the average temperature in the temperature interval considered. For a 600 second interval the rise in temperature may be 4°C. This value of the calorimeter constant has been established as the result of many experiments with succinic acid. Figure 9 shows the apparent and actual specific heats of succinic acid as a function of temperature.

OPERATING PROCEDURE

Preparation of the Resin Casting and Final Setup of the Calorimeter

21. The casting resin under study is carefully weighed out, mixed, and slowly poured into the calorimeter vessel which is held firmly in the jig. The catalyzed resin is slowly poured into the can and allowed to fill the can to within about 0.125" of the top, care being taken to insure that none is allowed to flow over the sides. It is important to observe carefully that the resin completely covers the resistance coil, leaving only the two leads sticking up through the resin, and that the thermocouple in the center of the resin does not move during the pouring operation and subsequent polymerization. In practice, no trouble has been experienced, provided that all the operations are conducted slowly and carefully. Figure 10 shows the calorimeter vessel containing the encapsulated resistance coil and thermocouples.

22. The casting should be cured at a slow rate for a long period of time. This long cure reduces the possibility of cracking, volatilization of the curing agent (bubbles), or changing the position of the center thermocouple. After gelation occurs, it is advisable to post-cure the casting for 24 hours at 160°C. This post-cure will insure that polymerization is complete. This is an important point. If polymerization were not complete and some of the heat of polymerization were evolved during a specific heat determination, it would serve to raise the temperature of the mass of resin. This additional heat would cause a serious error in the calculation of the specific heat.

23. After polymerization the casting is removed from the jig. The complete calorimeter vessel is now weighed to the nearest gram. The previously determined weights of the can, coil and thermocouples are subtracted from the total weight and the weight of the resin determined. Two additional thermocouples are then taped halfway up on the sides of the calorimeter vessel, 180° apart. One of these thermocouples is used to measure the wall temperature of the vessel, the other is used to control the adiabaticity of the system. The resistance of the now encapsulated heating coil is now accurately determined on a bridge (Leeds and Northrup Test Set #5430-A). The measured resistance at 25°C will increase slightly with increasing temperature. To determine this change the resistance is measured periodically as the casting cools down from 160°C to 25°C in the calorimeter. It has been found that a coil having a resistance of 2.40 ohms at 25°C will have a resistance of 2.45 ohms at 160°C. Two leads of insulated copper wire are carefully soldered to the two short (about 0.25" - 0.50") ends of the resistance coil.

24. The calorimeter vessel is then carefully inserted into the calorimeter. In practice, the vessel has been allowed to rest on a small piece of cork 0.5" thick and 1.5" in diameter, which was placed on the bottom of the inner cylinder. The leads from the two thermocouples in the resin, the two taped to the calorimeter vessel, and the three taped to the inside wall of the inner cylinder are all passed through the plastic cover of the calorimeter. In a similar manner the two insulated copper leads from the resistance coil are also passed through the cover, and small bits of rubber are used to seal the holes. The top of the calorimeter is then pushed into place. This completes the sealing of the calorimeter, and insofar as the actual instrument is concerned, it is ready for use. However, before a determination can be made, the thermocouples must be connected to the temperature controlling and temperature measuring sections of the instrumentation and the resistance coil must be connected through a precision ammeter (Westinghouse Type PY-5) to a constant source of EMF.

DETERMINATION OF THE SPECIFIC HEAT

25. After the calorimeter vessel has been placed and sealed in the calorimeter and the thermocouples connected to the proper instruments (see Section III) a determination of the specific heat of the resin as a function of temperature may be made. The source of power for the heating of the sample is a 6-volt wet storage battery (Navy Model, Lyons Storage Battery Co.). In practice, only two of the three cells of this battery are used, the average delivered voltage being about 3.5 to 3.7 volts. The battery, heating element and a Westinghouse PY-5 precision ammeter are connected in series to heat the sample as shown in Figure 8. During an experiment the EMF and the heating coil resistance will change slightly; however, these are linear relationships which can be accurately determined. The change in EMF is a function of time and the change in resistance is a function of temperature. The current is about 1.5 amps. and decreases slightly during the run. The ammeter is read at 300-second intervals.

26. While the run is in progress, adiabatic conditions are maintained by means of the technique described in detail in Section III, 2. By careful control of the adiabatic system (Section III, 2) the temperature difference (thermal head) between the resin sample and the calorimeter wall can be maintained within 1°C. The average initial rate of heating of the sample is approximately 1°C per 100 seconds. The rate slows down considerably with increasing temperature. A precision timer is used to measure the time intervals.

27. Readings of the rise in temperature of the sample are made every 300 seconds. During the run the six-point Brown Recording Potentiometer which records the readings of the wall temperatures of both the sample and calorimeter is constantly observed to detect any change in the adiabaticity of the system. If any change should occur, it can be quickly noted and corrected. In practice, a large number of runs has been made, and if care is taken, no difficulty will be experienced in maintaining adiabaticity.

28. Readings of change in temperature of the resin sample are made every 300 seconds as are current readings. However, the time interval used in the calculation is 600 seconds. This is to average out any error over a longer time interval. For example, both change in temperature (ΔT) and current (I) readings are made at 0, 300, 600, 900, 1200, 1500 etc. second intervals, but the differences in temperature used in the calculation are 0 to 600 seconds, 300 to 900 seconds, 600 to 1200 seconds, etc. The calculated values of specific heat are, therefore, average values over the temperature interval.

29. The specific heat of the resin may be calculated by means of Equation (2):

$$Cp = \frac{I^2 R t}{4.185 M \Delta T} \qquad (2)$$

where I = the current in amperes.

R = the resistance of the heating coil in ohms.

t = the time in seconds.

M = the mass of the resin in grams.

ΔT = the rise in temperature of the resin in °C.

The constant 4.185 is the mechanical equivalent of heat in joules per calorie. The calculated values of specific heat are then corrected by the calorimeter constant. This method has the advantage over other methods in that the measurements are made continuously.

DETERMINATION OF THE SPECIFIC HEAT OF LOW PRESSURE POLYETHYLENE

30. After determining the calorimeter constant, it was desirable to check the calorimeter and instrumentation and the calorimeter constant against a casting resin of known specific heat. Unfortunately, such information was not available for any of the thermosetting casting resins. However, during the course of this work, Wunderlich and Dole (reference (1)) presented data on the specific heat of low pressure polyethylene (Marlex 50, Phillips Petroleum Co.) in the temperature range 50°C - 150°C. This thermoplastic resin melts at about 134°C. The increase in the apparent specific heat which occurs during melting is due to the heat of fusion of this material which has been estimated at 66 cal/gm at 134°C. by Wunderlich and Dole. About 90% of the melting takes place over a 15° temperature range.

31. Under certain conditions this type of polyethylene can be used as a casting resin; for example, vacuum castings of this material can be prepared by heating of the commercially available pellets in a suitable mold under vacuum at 160°C. Since this resin is very similar in its thermal properties to many of the common casting resins, it was decided to use it as a check on the accuracy of the calorimeter. It must be noted here that the Marlex 50 as supplied by the manufacturer is completely polymerized; no additional polymerization takes place during the vacuum casting operation.

32. A casting of low pressure polyethylene containing the resistance coil and thermocouples was prepared as described in Section V, 1, except that the calorimeter vessel, containing the resistance coil and thermocouples, was filled with the polymerized pellets and then heated overnight at 160°C in a vacuum oven. Several refillings were necessary to completely fill the vessel. When completely filled, the casting was slowly cooled to 25°C.

33. The calorimeter vessel was then placed in the calorimeter and connected to both the temperature measuring and controlling instrumentation. The actual determination was made in exactly the same manner as described in Section V, 2, except that the run was stopped at 141°C. This was done as a precaution, since the now liquid (but very viscous) polyethylene might allow the resistance coil or thermocouple to move in the calorimeter vessel and cause short circuits in the system. Later examination showed that this had not occurred. During the experiment, it was noted that in the region near the melting point the rate of rise in temperature was very slow. During the 600 second interval in which melting was completed, the change in temperature was about 0.3°C. This necessitated keeping the Variac so adjusted that the wall temperature was almost constant for about 600 seconds. No difficulty was experienced during this experiment and adiabaticity was maintained within less than 1°C.

34. The results of the specific heat determinations are shown in Figure 11. Figure 12 shows the same values of specific heat vs. temperature plotted on a different scale, which shows the scatter in both sets of data points. These graphs show that the results obtained in this Laboratory with the calibrated adiabatic calorimeter are in excellent agreement with the results of Wunderlich and Dole in the temperature range 50°C - 141°C. The most important point of agreement is the fact that the results also agree during the melting range. It will be noted that after melting, the specific heat rapidly decreased to less than one cal./gm. °C.

35. These results indicate that this adiabatic calorimeter compares favorably with that of Wunderlich and Dole. These investigators used a calorimeter which is considerably more complex both in design and in operation, and is used exclusively for specific heat determinations. Their instrument is described in detail in reference (11). Their calorimeter appears to be slightly more accurate than the instrument described in this report. However, the values obtained by the adiabatic calorimeter are excellent for engineering purposes.

DISCUSSION OF THE RESULTS

36. Plots of specific heat vs. temperature are shown in Figures 13, 14 and 15. These plots are typical of those obtained by this method. From a study of these plots several interesting facts are evident. First, an increase in specific heat is noted with increasing temperature. This is to be expected, having been previously noted by other investigators (references (1), (2) and (13)). Secondly, in Figure 13 a discontinuity is noted at about 60-70°C. This is the second order transition point of the resin. This transition, on heating a polymer from a hard, glass-like state to a rubbery state, manifests itself as a change in the temperature dependency of a large number of physical properties. At this point a discontinuity occurs in the secondary thermodynamical quantities, such as specific heat, thermal conductivity, thermal expansion, etc. As the temperature is raised through this point, a change occurs, without latent heat, which results in larger values of the secondary thermodynamical quantities and a change in the general physical properties of the material. In general, the polymer becomes softer and somewhat rubbery.

37. This discontinuity in the specific heat vs. temperature plot has been noticed by Smith and Dole (reference (12)) and Gast (reference (13)) and interpreted in terms of a second order transition. Spencer and Boyer (reference (14)) have considered the second order transition in high polymers and give an excellent discussion of this topic. It will be noted in Figure 14 that no second order transition is noted for Epon 828 when cured with 12.6% of m-phenylene diamine. This is not surprising when one considers the high heat distortion point, 150°C, (reference (15)) of this resin system. The heat distortion point and the second order transition point are different, although related. The former is characteristic of the macro properties of the system;

the latter, of the micro properties. Another point which must be noted is that these thermosetting resin systems are all amorphous in structure, not crystalline. This means that the observed temperature dependence of the secondary thermodynamical quantities will occur over a temperature range. This is shown in Figure 13 where the observed second order transition occurs over a 8°C range.

38. The accuracy of a typical value of specific heat, as calculated by the procedure of paragraph 29 is estimated from Equation (2). The estimated errors in the various quantities appearing in Equation (2) are tabulated below.

Quantity	Error
I^2	1.5%
R	0.4%
m	0.2%
t	0.2%
T	0.5%
Cp	2.8%

The data obtained by this method are reproducible and represent average values obtained by at least two determinations.

39. Comparison of the values for the specific heat of Epon 828 cured with different amine curing agents is interesting. Epon 828 when cured with diethylaminopropylamine tends to reach an approximately constant value of specific heat at about 113°C, while the same resin cured with m-phenylene diamine continues to show an increase in specific heat at an approximately constant rate throughout the experiment. This latter system is generally considered to be the most highly crosslinked of the three systems. Epon 828 with Tris(dimethylaminomethyl)-phenol tri (2-ethyl hexoate) shows a rather sharp second order transition at about 68°C and then a slow but uniform increase in specific heat till 160°C. Figure 16 shows the specific heat of DuPont 820-001 epoxide resin. This resin contains about 15% of SiO_2 filler.

CONCLUSION

40. An adiabatic calorimeter has been constructed and successfully used to determine the specific heats as a function of temperature of a number of casting resins. This instrument is used for obtaining engineering data and is accurate to within 5%. It was found that the calorimeter could be calibrated against succinic acid, and low pressure polyethylene was used to check the accuracy of the instrument. The data obtained on some of the epoxide resins were indicative of second order transitions in the cured resin.

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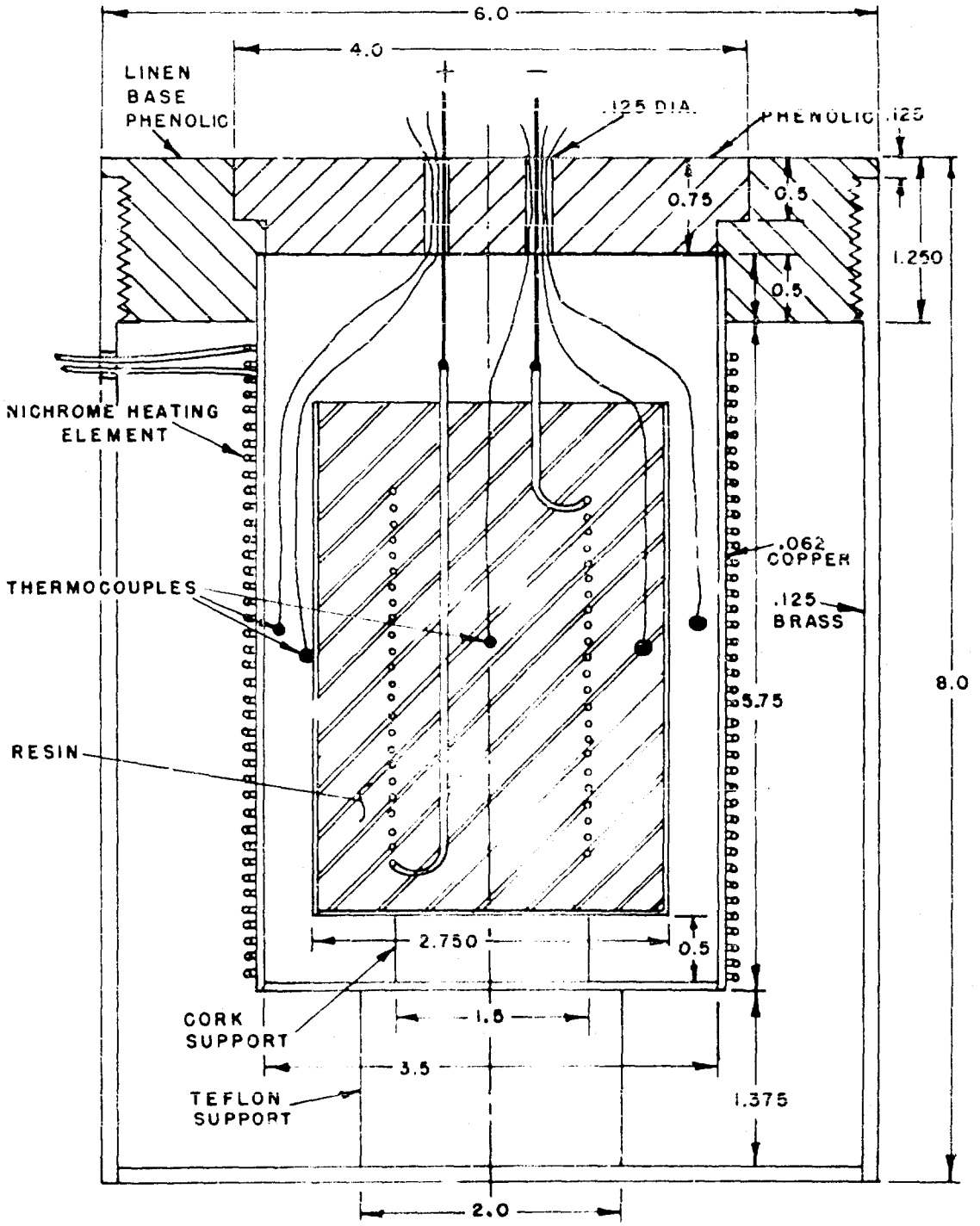
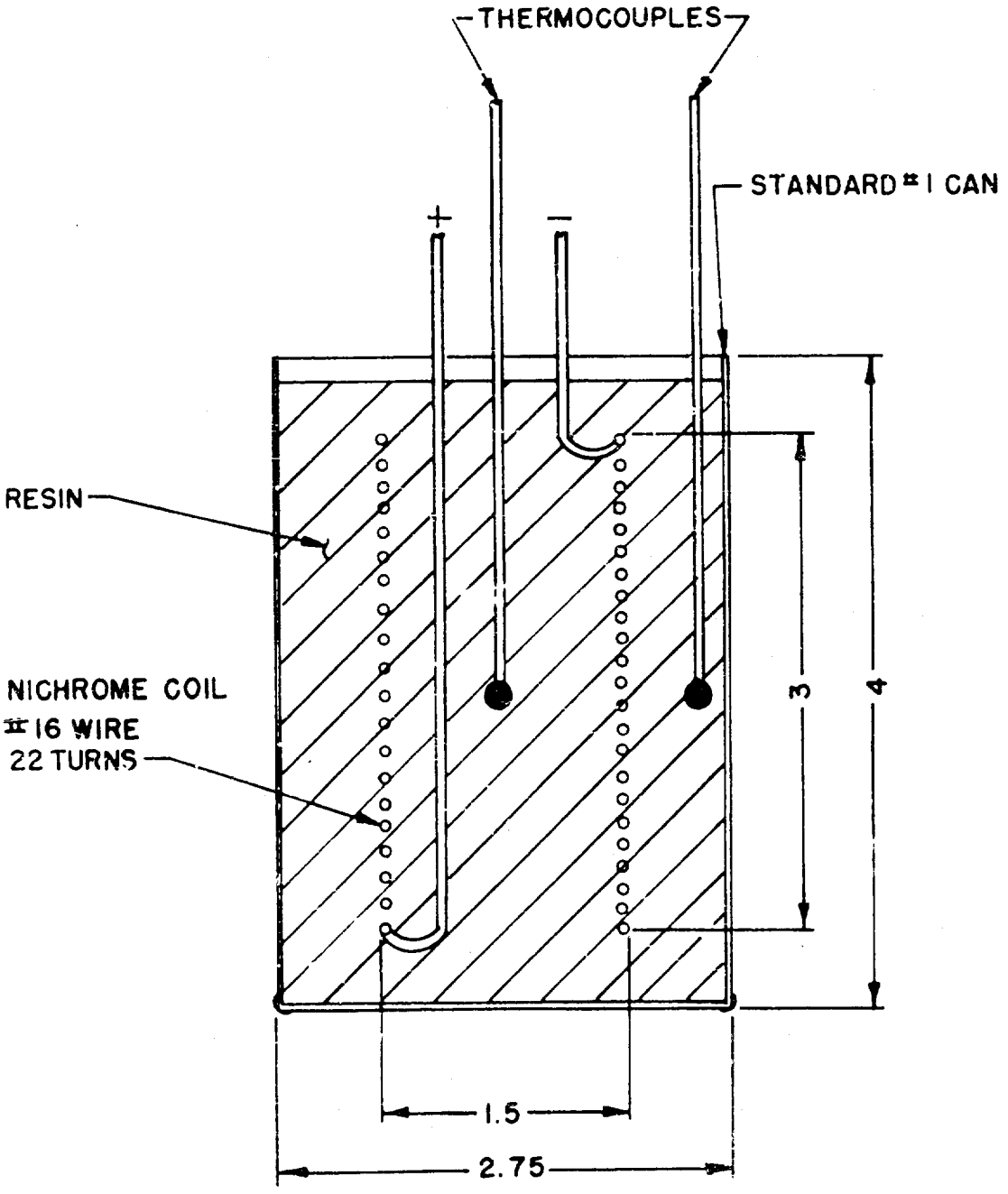


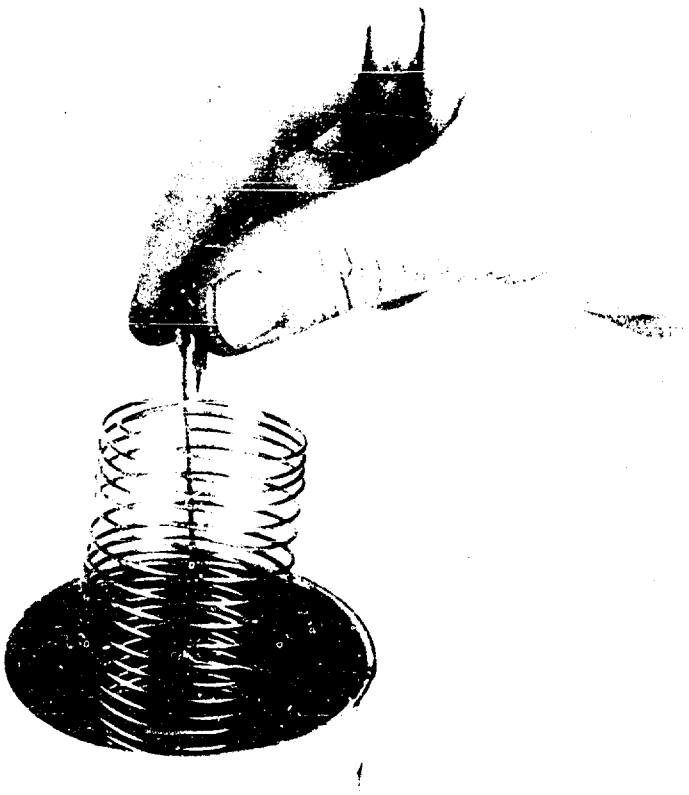
FIGURE 1

CALORIMETER SETUP FOR SPECIFIC HEAT DETERMINATION



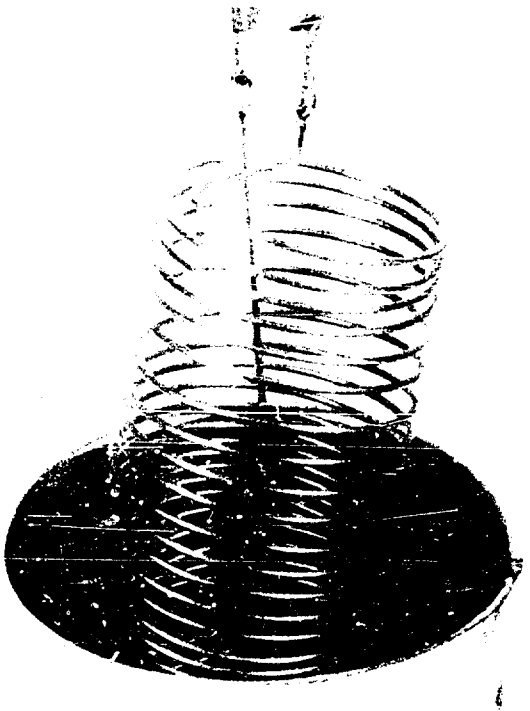
CALORIMETER VESSEL FOR
SPECIFIC HEAT DETERMINATIONS

FIGURE 2



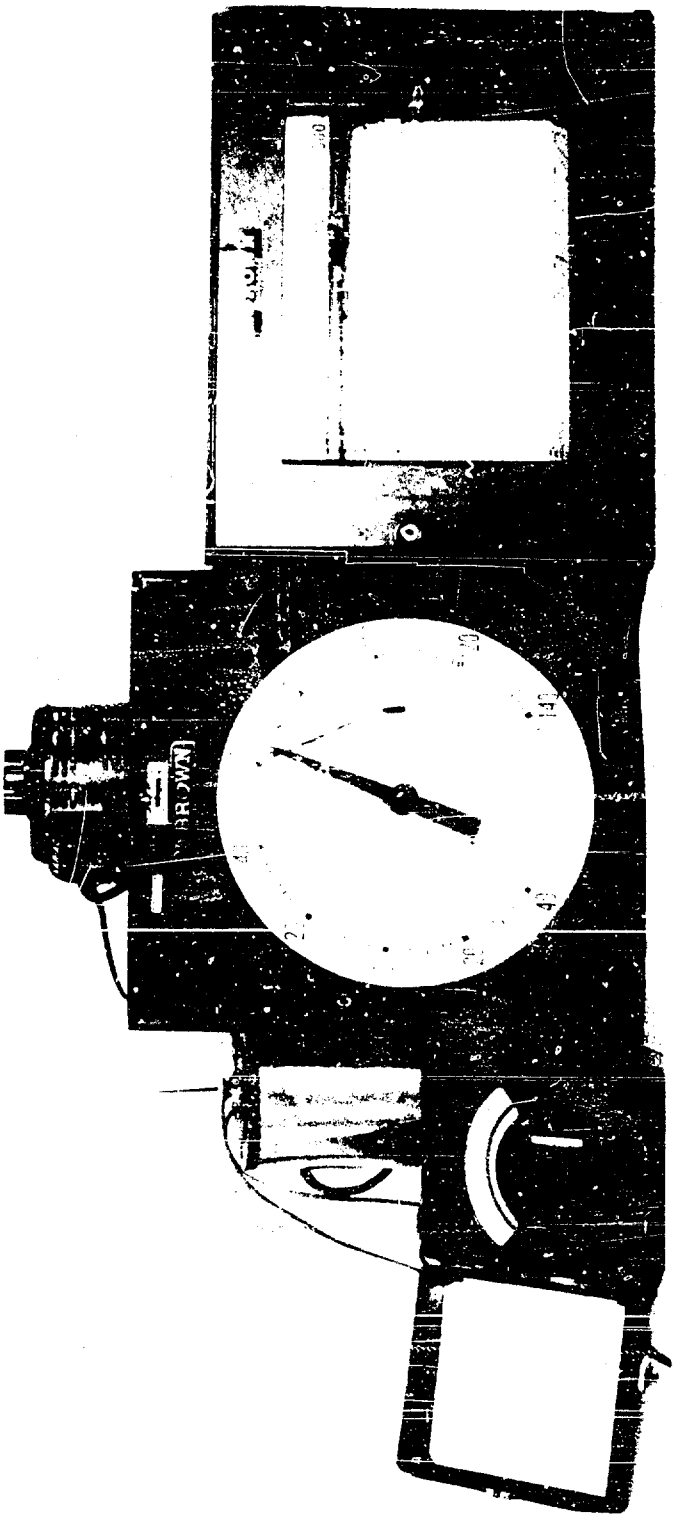
HEATING COIL BEING PLACED IN CALORIMETER VESSEL

FIGURE 3



HEATING COIL BEING PLACED IN CALORIMETER VESSEL

FIGURE 4

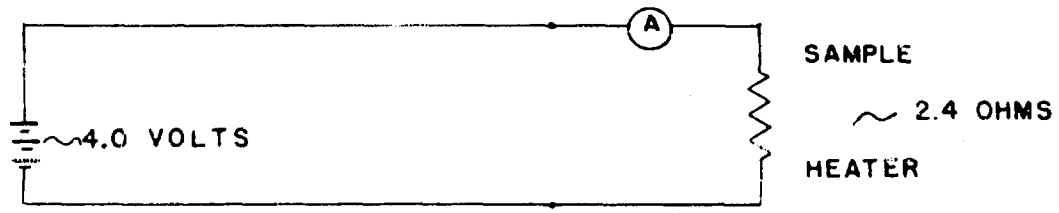


EXPERIMENTAL ARRANGEMENT SHOWING CALORIMETER, RECORDERS AND AMMETER.

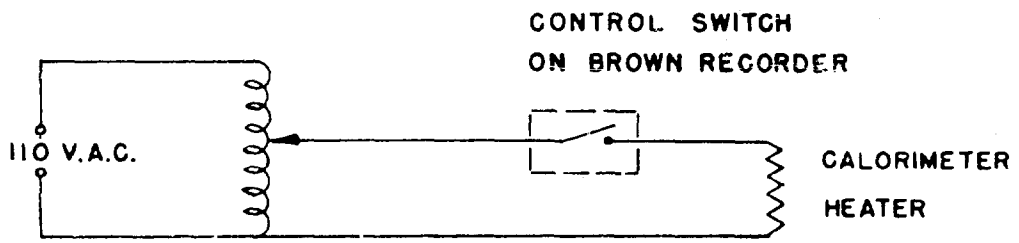
FIGURE 5

PRECISION
POTENTIOMETER

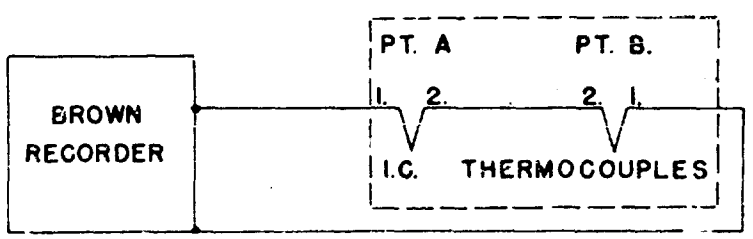
FIGURE 6



SAMPLE HEATING CIRCUIT



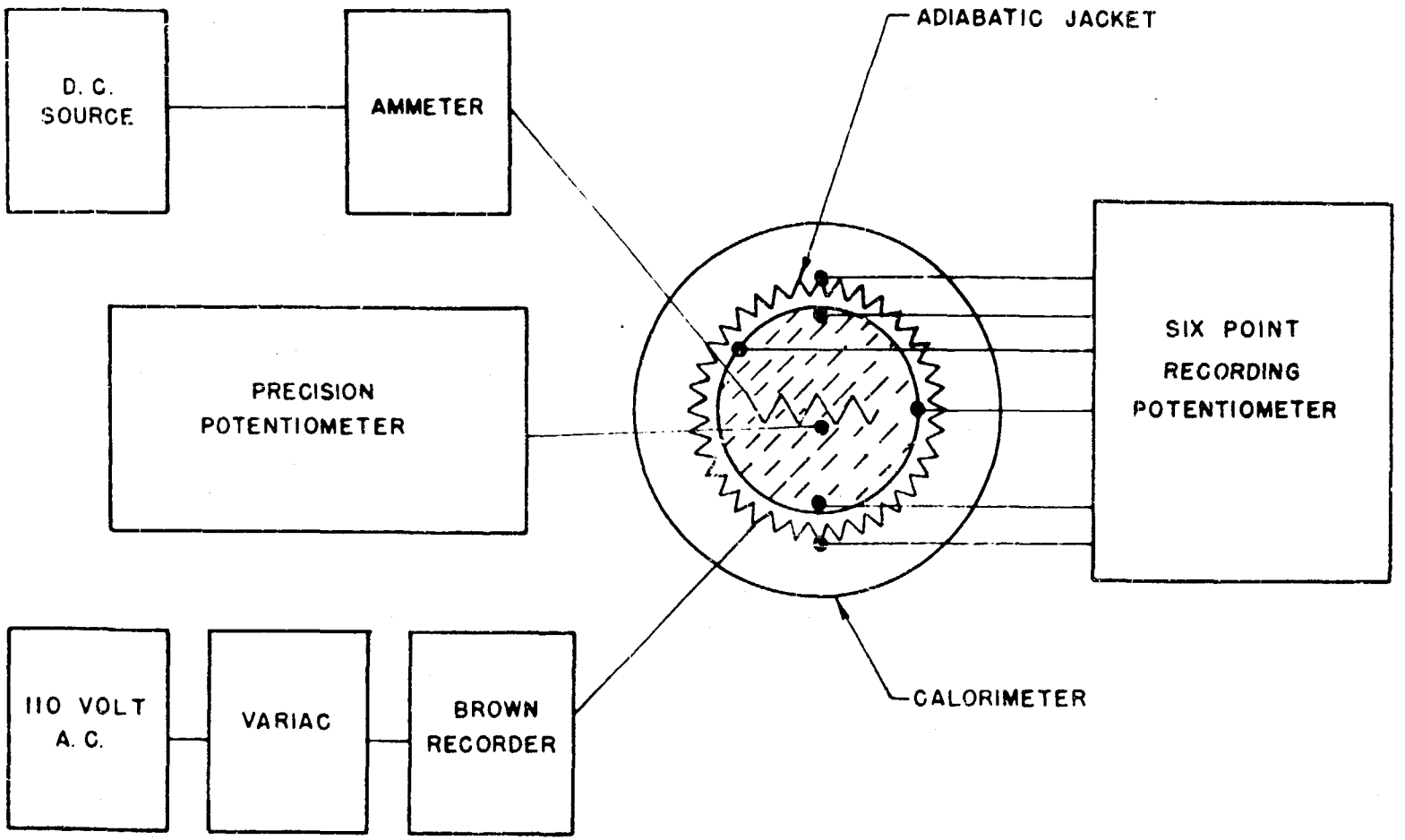
CALORIMETER HEATING CIRCUIT



CALORIMETER CONTROL CIRCUIT

CALORIMETER CIRCUIT DIAGRAMS

FIGURE 7



INSTRUMENTATION BLOCK DIAGRAM

FIGURE 8

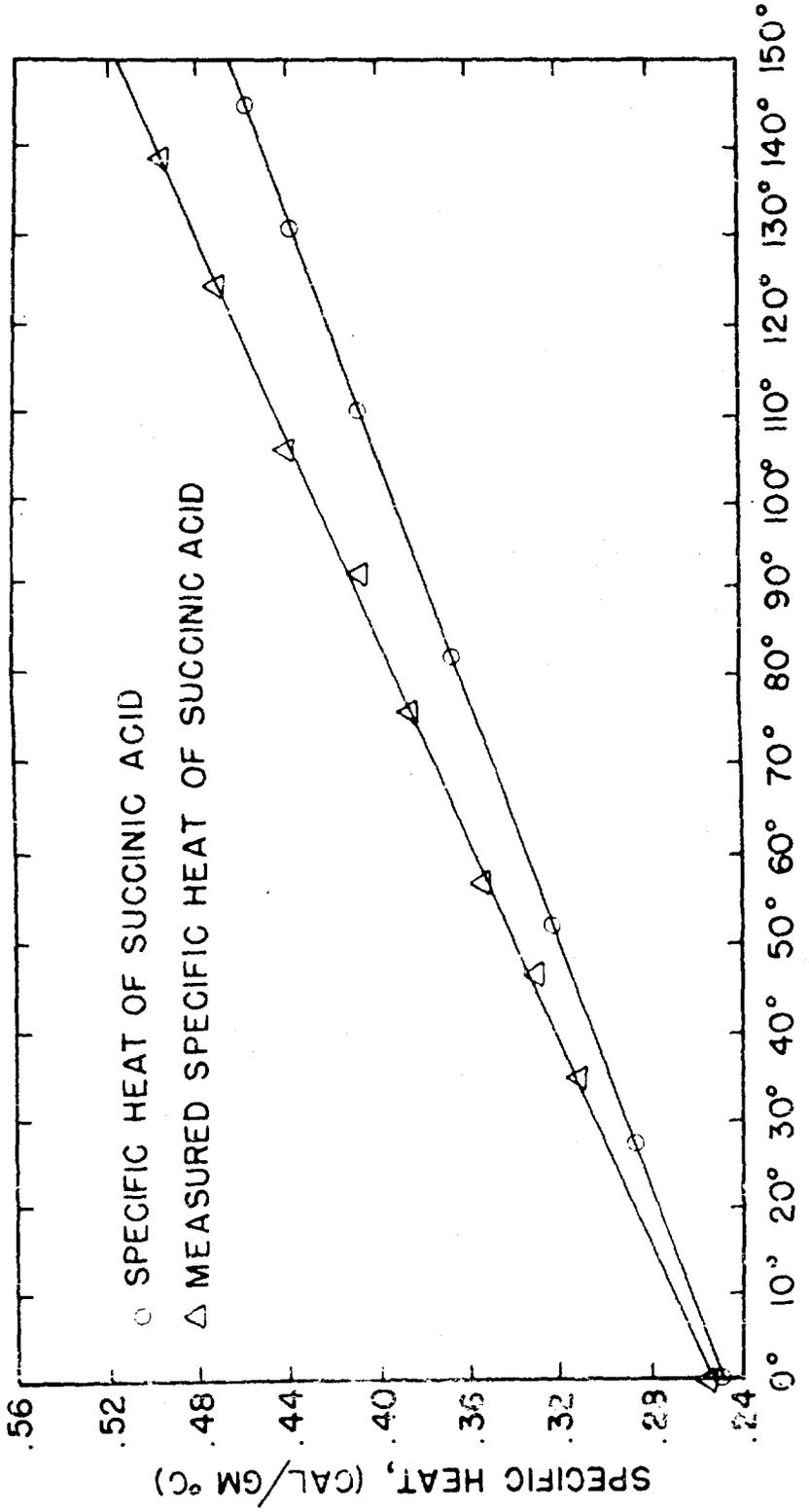
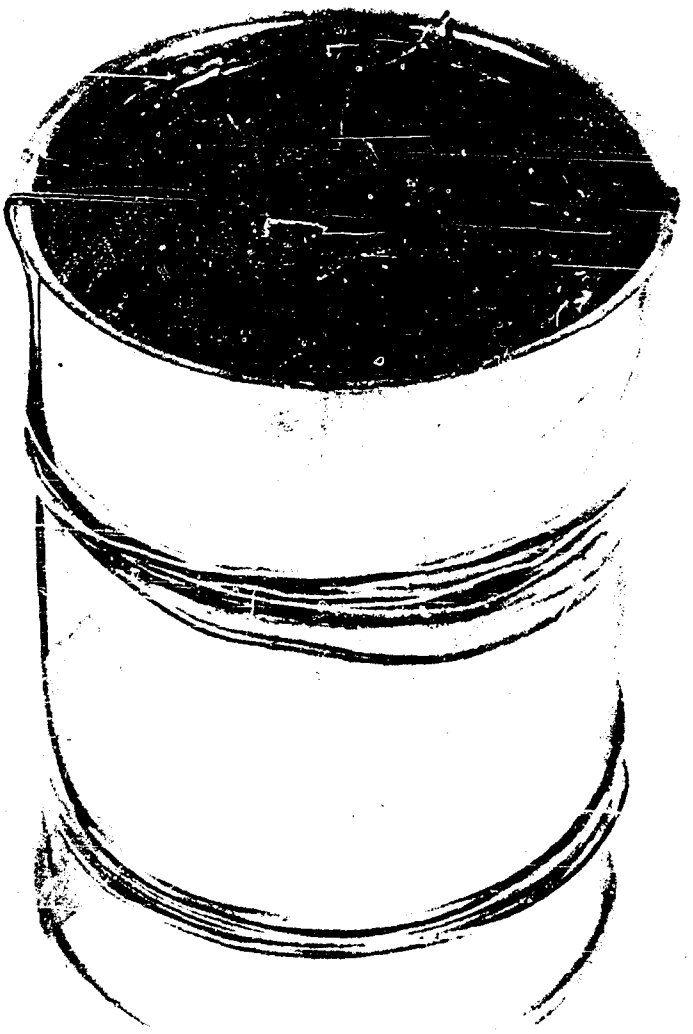
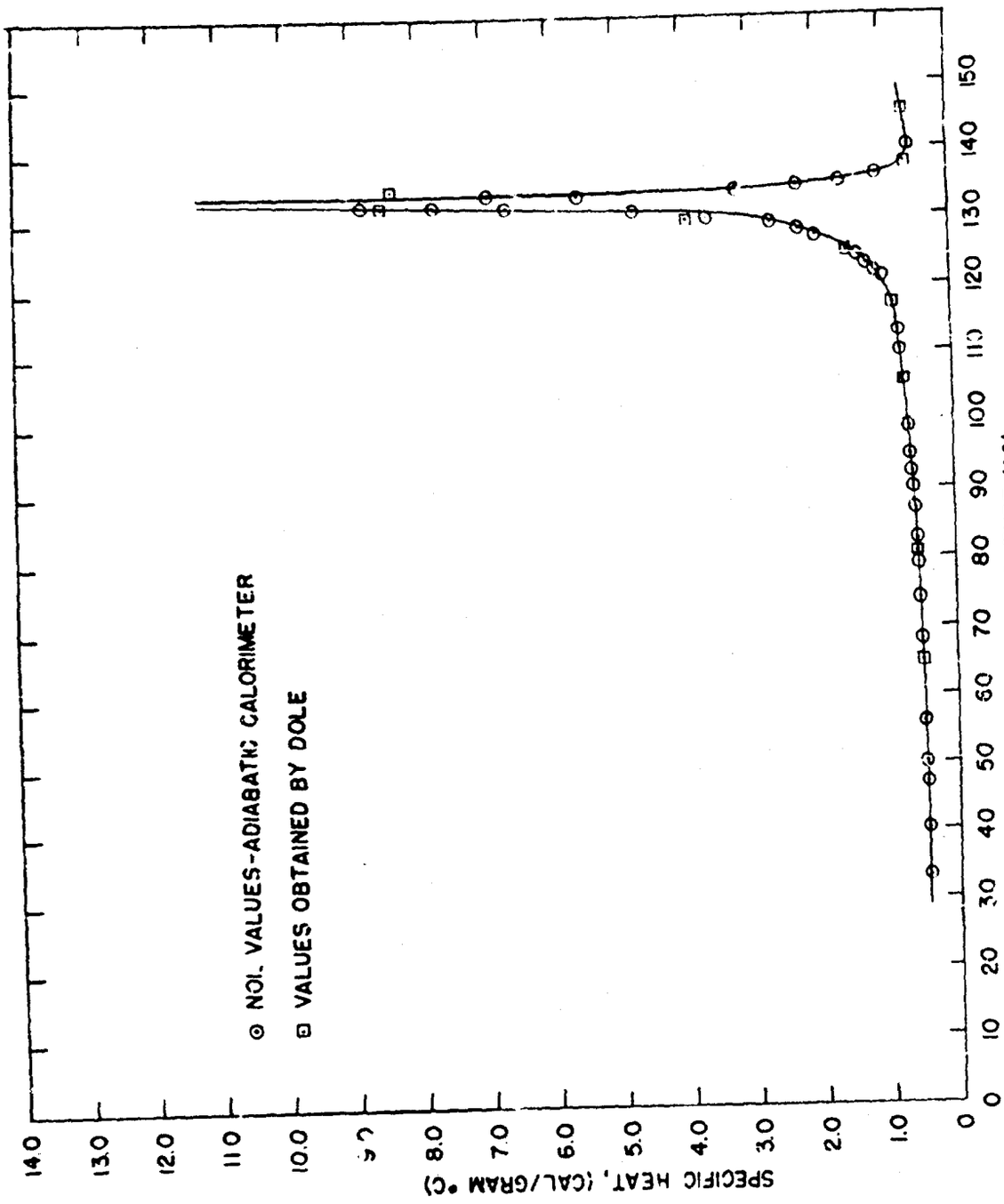


FIGURE 9

SPECIFIC HEAT OF SUCCINIC ACID AS A FUNCTION OF TEMPERATURE

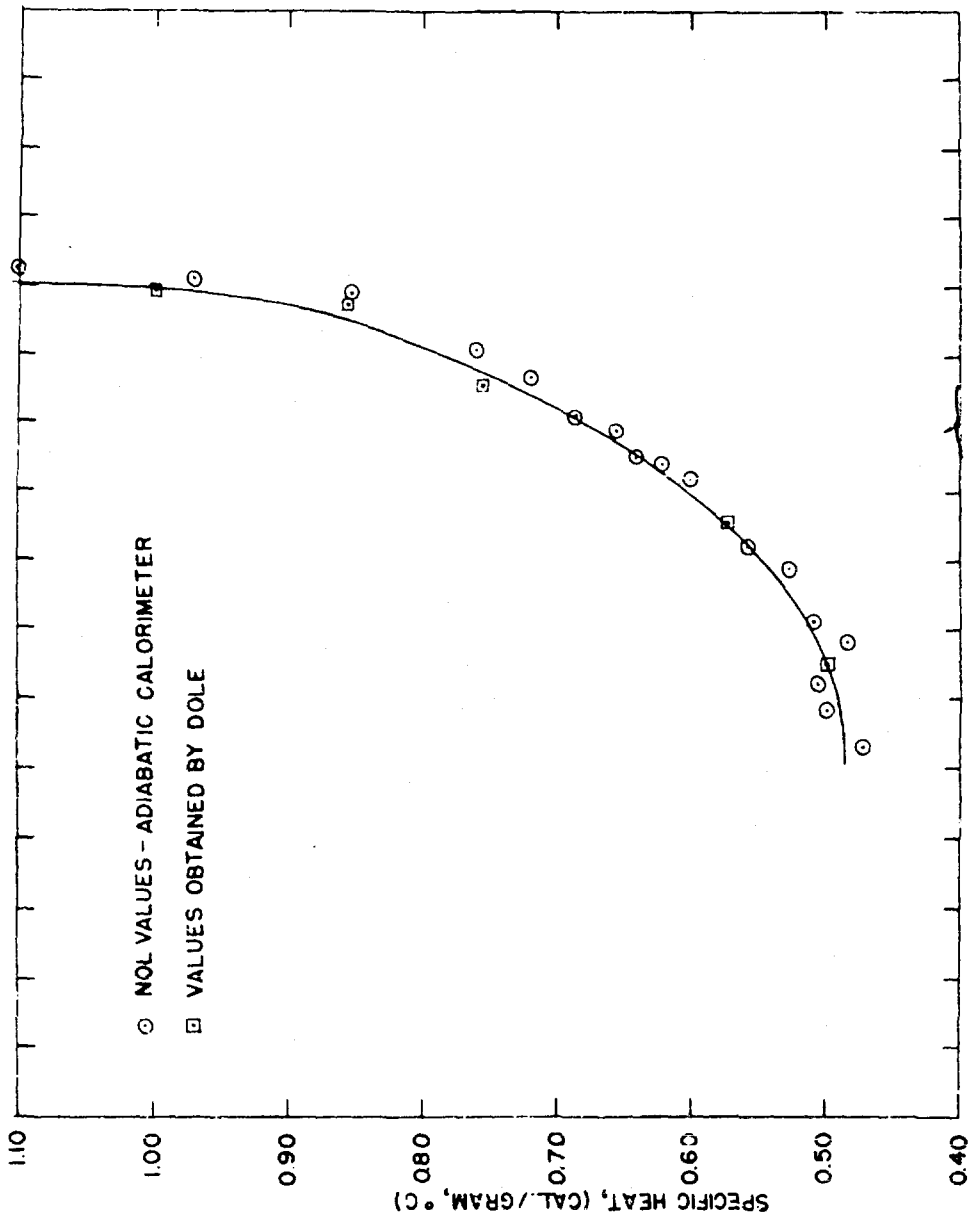


CALORIMETER VESSEL WITH ENCAPSULATED COIL
FIGURE 10



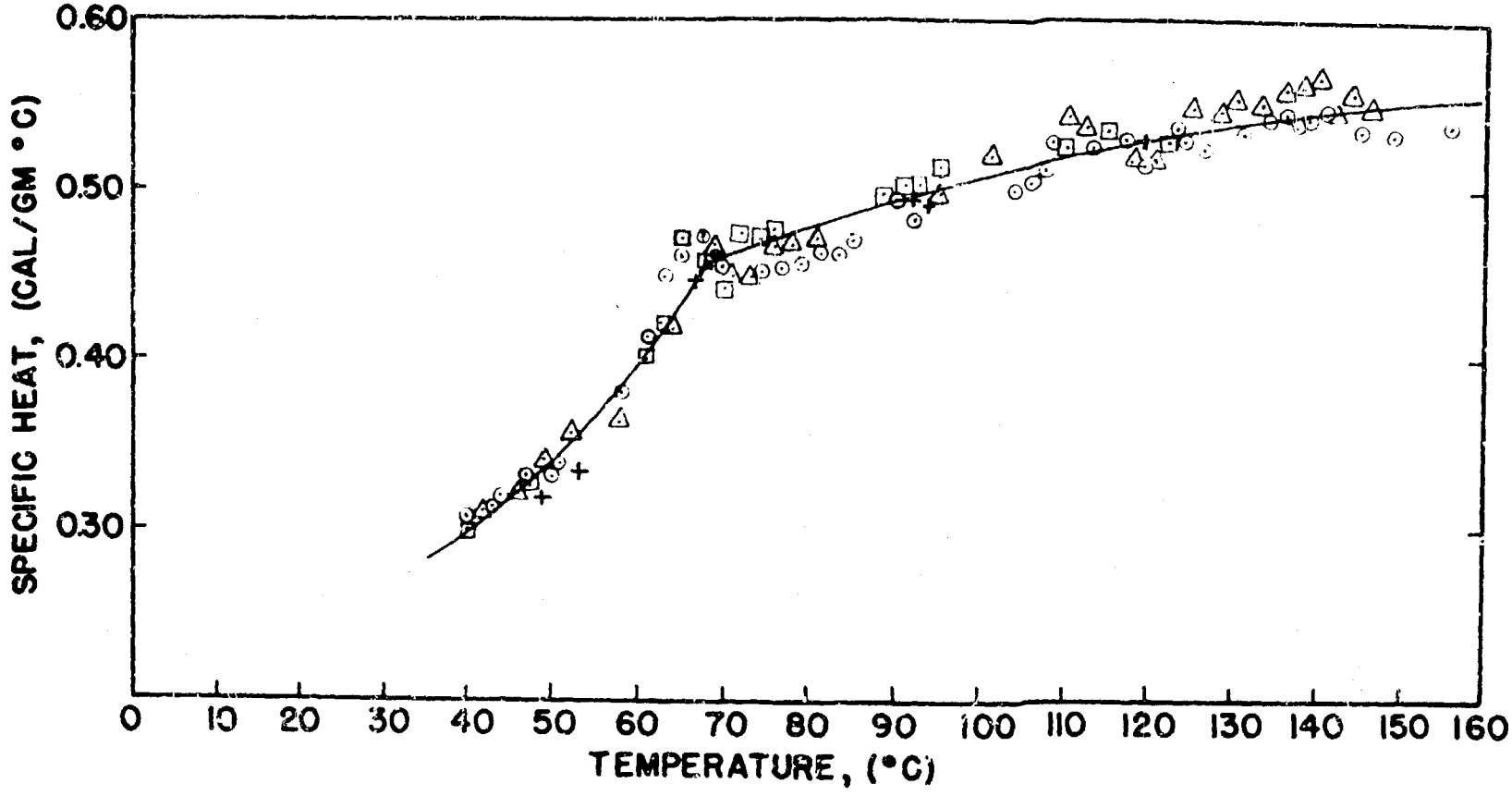
SPECIFIC HEAT OF ANNEALED MARLEX 50 POLY-ETHYLENE AS A FUNCTION OF TEMPERATURE

FIGURE 11



SPECIFIC HEAT OF ANNEALED MARLEX 50 POLY-ETHYLENE AS A FUNCTION OF TEMPERATURE

FIGURE 12



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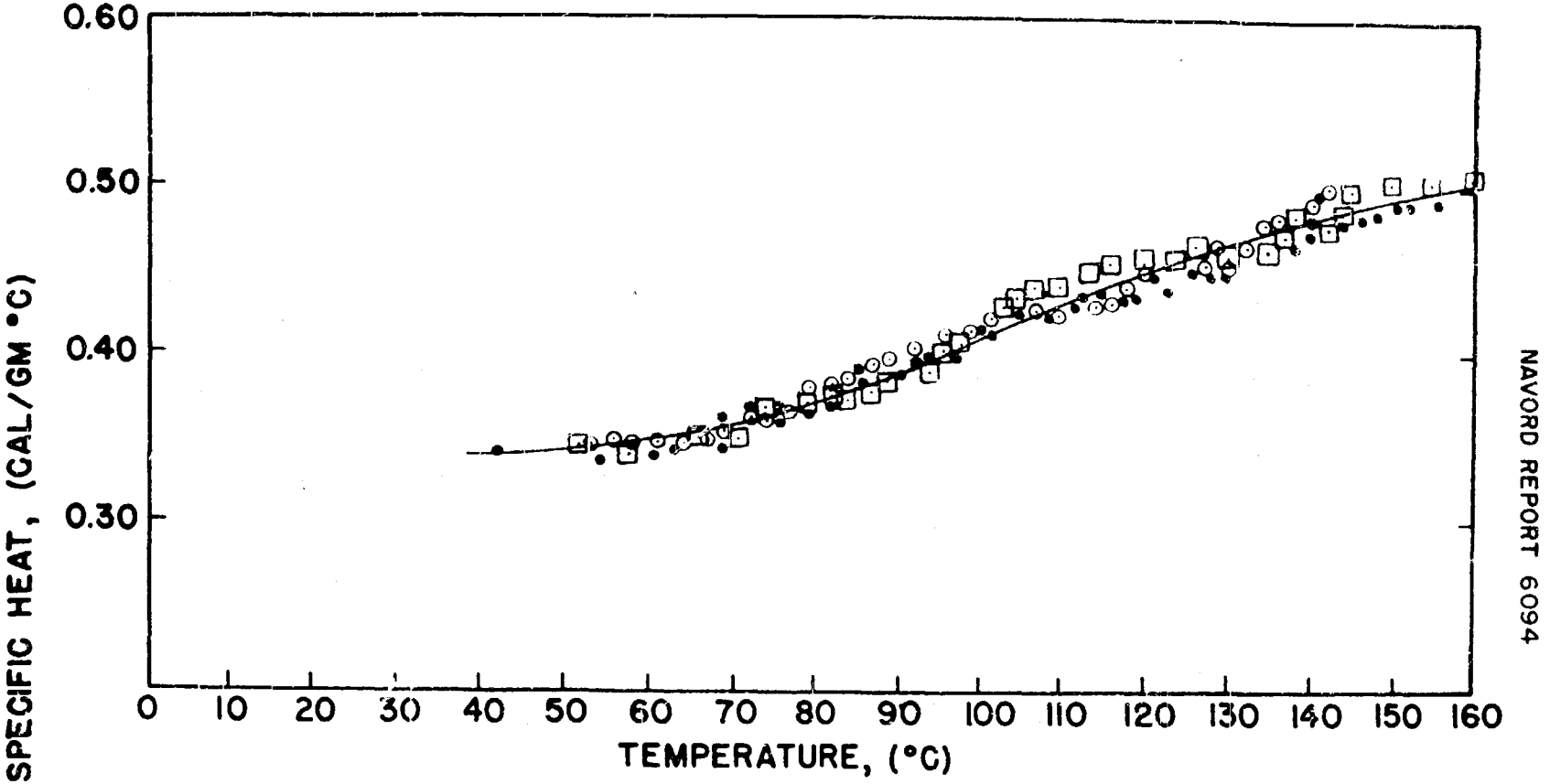
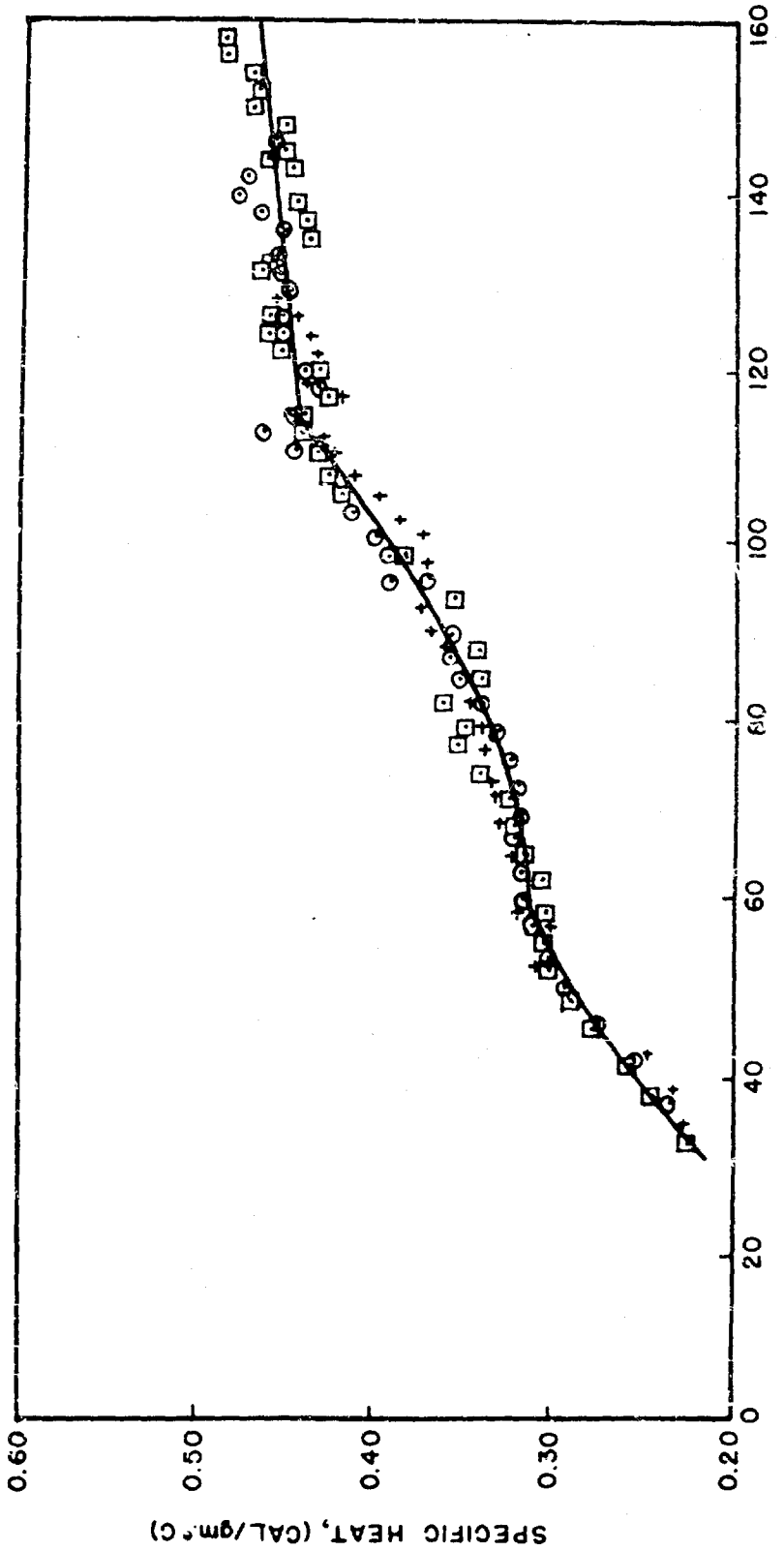
SPECIFIC HEAT OF EPON 828 CURED WITH 11.5% OF TRIS(DIMETHYLAMINOMETHYL) PHENOL TRI (2-ETHYL HEXOATE)

FIGURE 13



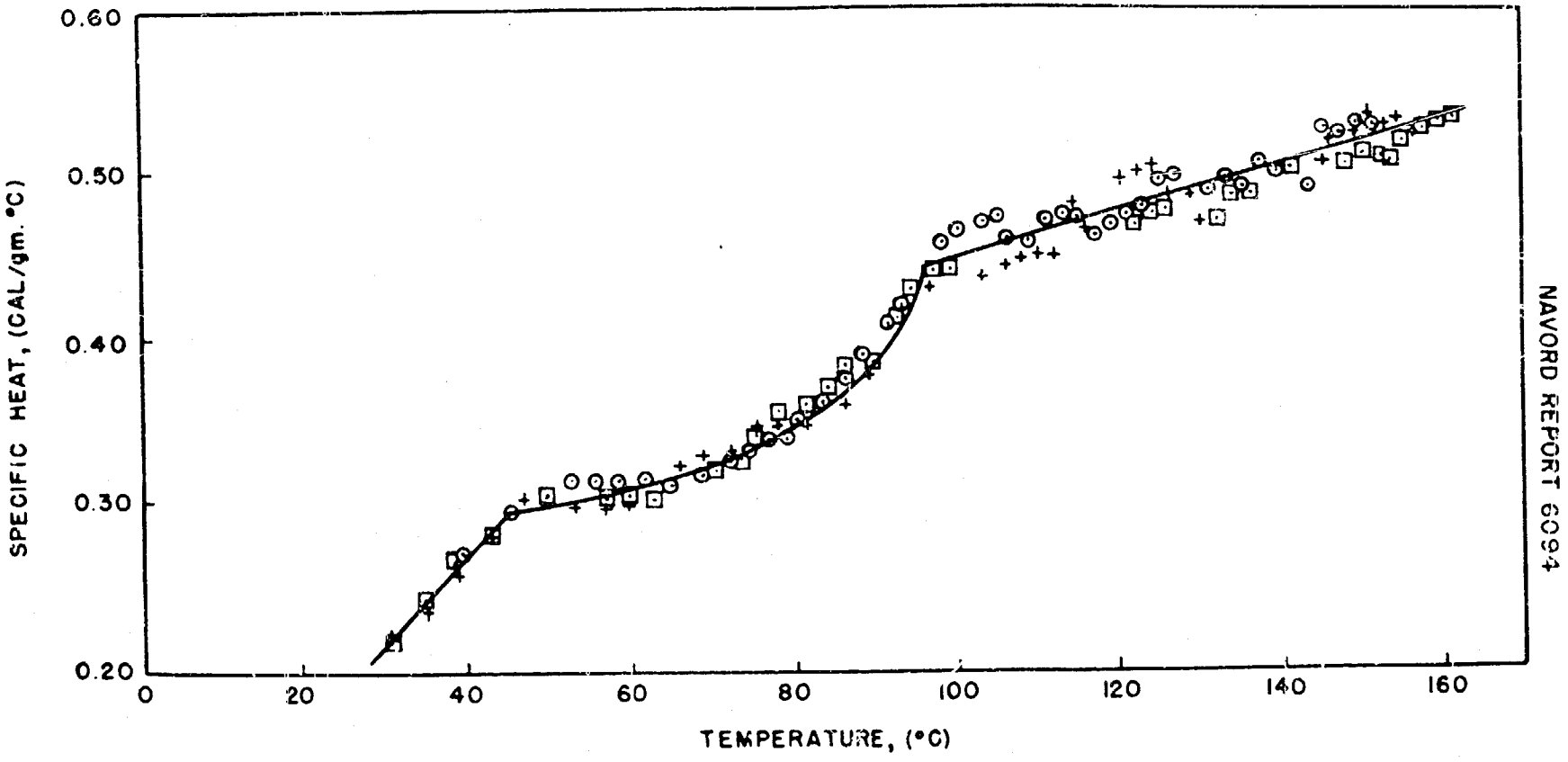
SPECIFIC HEAT OF EPON 828 CURED
WITH 12.6% OF M-PHENYLENEDIAMINE.

FIGURE 14



SPECIFIC HEAT OF EPON 828 CURED WITH 5.65% OF DIETHYLAMINOPROPYLAMINE.

FIGURE 15



SPECIFIC HEAT OF DU PONT 820-001 EPOXIDE RESIN WITH 15 % SiO_2 FILLER.

FIGURE 16