

Determination of Thermal Conductivity of Food
Materials Using a Bead Thermistor

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(ABSTRACT)

The ultimate goal of this research was to determine the feasibility of measuring thermal conductivity of food materials using the bead thermistor with particular reference to high temperature.

Feasibility was established by examining the effects of the input parameters and the measurement error associated with them on the ability to estimate the test medium thermal conductivity test medium. This study showed that estimation of effective radius and bead thermal conductivity, the probe parameters, had the most significant impact on the ability to estimate the thermal conductivity of food materials. The probe parameters were determined by standardizing the thermistor probe against materials of known thermal conductivity. The current lack of well defined thermal reference materials in the range of water and most food products is a primary source of error associated with the method.

The accuracy and coefficient of variation of the Bead Thermistor Method were statistically documented in 10° increments over the temperature range of 25°C to 125°C. These results showed the method to have better than 10% accuracy across the entire temperature range. Distinct differences in accuracy between probes at a given temperature were also discovered. Standardization with water and castor oil resulted in a more accurate method than was achieved using water, castor oil, and glycerin.

The minimum particle diameter necessary to maintain the infinite boundary condition assumption required by heat transfer theory was found to be ≥ 5 mm.

The methodology was evaluated by examining the effects of temperature on the thermal conductivity of milk of different fat contents. A prediction equation for each product was attempted from the experimental data, but the data appear best fit by assuming a constant value across temperature. Heat altered the product physically which likely affected temperature dependence.

Based on the results of this study, the bead thermistor method can be considered a practical method for determining thermal conductivity of food materials over the temperature range considered in this study.

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Dedication

This work is dedicated to my family, my father, , my late mother, , my brother, , and my sister . They were always interested in my work even though they didn't always understand what it was. Their love and support for all these years have finally paid off.

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Nomenclature

a	= temperature-resistance calibration coefficient
d ₋	= error associated with the given parameter
dT	= temperature step change (°C)
f	= fat content (%)
F	= fat to solids-non-fat ratio
k	= thermal conductivity (W/m-°K)
M	= void fractio of discontinuous phase
q(t)	= volumetric heat generation rate (W/m ³)
Q	= heat per unit length (W/m)
r	= effective radius (m)
R	= resistance (ohms)
s	= solids-non-fat content (%)
S	= solids (%)
t	= time (sec)
T	= temperature (°C)
v	= volume (m ³)
V	= voltage (volts)
W	= moisture content, wet basis (%)
β	= transient heat generation rate (W√s/m ³)
Γ	= steady-state heat generation rate (W/m ³)
δ	= partial derivative
φ	= evaporation ratio (weight of milk before to after evaporation)

Subscripts

b = bead

c = continuous phase

d = dispersed phase or particles

m = medium

ss = steady-state

T = thermistor

I. Introduction

Aseptic processing is defined as the filling of presterilized containers with sterile or commercially sterile product and hermetically sealing them under aseptic conditions (Lopez, 1986). The first aseptic process was devised in Denmark in the early 1920's. In 1933, the American Can Company developed the Heat-Cool-Fill (HCF) system. This system used pressurized saturated steam to sterilize the cans and lids. However, the method was not a commercial success. In the 1940's, the Dole-Martin Process was invented. This system uses super-heated steam to sterilize the cans. This method is still in use today (Hersom, 1985). The advent of plastic/board complexes sterilized with hydrogen peroxide proved a major breakthrough for aseptic processing. In Switzerland, milk packaged this way was sold in the early 1960's. The development of new packaging materials and improved process control technology has greatly expanded the range of products adaptable to aseptic processing.

Of current interest in the food industry is the development of aseptic processes for products containing discrete food particulates. There are several serious concerns which need to be addressed if an aseptic process containing particulates is to be developed. The most

important of these concerns is the ability to guarantee that the particulates are indeed commercially sterilized during the high-temperature-short-time (HTST) or Ultra High Temperature (UHT) process while maintaining product quality (de Ruyter and Brunet, 1973). To achieve this combination of safety and quality during sterilization, precise definition of process parameters is required (Lekwauwa and Hayakawa, 1986).

To determine the adequacy of a thermal process, it is necessary to document the time-temperature history of the slowest heating particle having the shortest residence time in the system. However, there is presently no practical method for physically measuring the rate of heat penetration into a food particulate suspended in a fluid moving through a heat exchanger. Therefore, modeling this heat transfer mathematically may become necessary.

In a continuous flow system, heat transfer from the liquid phase to the solid particulate occurs by convection, except in the case of high viscosity fluids where heat transfer is via conduction (Zuritz, et al., 1987a). Because a solid particulate heats at a slower rate than the liquid phase, more heat is required at the particle surface to raise the center temperature to a level necessary for sterilization. To calculate the center temperature, a surface temperature must be assumed (Gavin, 1985).

Investigators have proposed models for various conditions involving processing of particulate foods (de Ruyter and Brunet, 1973; Manson and Cullen, 1974; and Sastry, 1986). Application of these models requires that the thermal properties of the particulate itself be determined (Gavin, 1985; Sastry, 1986). Swartzel (1986), in modelling product constituent losses during UHT processing, required knowledge of thermal conductivity as a function of temperature. Since this data is not currently available, Swartzel assumed a constant value of $0.610 \text{ W/m-}^\circ\text{K}$ for whole milk.

At present there are several methods for the determination of the thermal conductivity of food materials (Reidy and Rippen, 1971). These include the Thermal Comparator method, the Guarded Hotplate method, the Fitch method, the Line Heat Source Probe method, and empirical equations. Each of these methods have their advantages. However, with the exception of the empirical methods, they also have the same limitations. That is, they are difficult to adapt to thermal processing temperatures, and are not applicable to use on small individual food particles. Some empirical equations do include temperature as a variable. However, these equations are only as accurate as the data used to generate them. And empirical equations are not reliable outside their designated ranges

of applicability. Presently, there are no thermal conductivity measurement methods applicable to small food particulates at thermal processing temperatures. The purpose of this work was to develop a method that would be capable of meeting this need.

The objectives of this study were:

- 1) to determine the feasibility of using a bead thermistor for the determination of thermal conductivity of high moisture and liquid food materials by examining the effects of the input parameters on the accuracy of thermal conductivity estimation
- 2) to statistically document the accuracy of the bead thermistor method over the temperature range of 25°C to 125°C
- 3) to determine the minimum particulate size which maintains the infinite boundary condition assumption for heat transfer
- 4) to document the functionality of the method by examining the effects of temperature and fat content of the thermal conductivity of milk products.

II. Literature Review

A. Introduction

A review of current methods for determining the thermal conductivity of food materials is presented. Empirical models for thermal conductivity, both temperature independent and temperature dependent, are summarized. The bead thermistor probe as a means of determining thermal properties of various materials is discussed, as is the present situation with regard to thermal standard reference materials. Representative thermal conductivity values for liquid, solid, and milk products are presented.

B. Measurement Techniques

1. Thermal Comparator Method

The thermal comparator is a simple device which consists of two thermocouples, each embedded in a phosphor-bronze sphere (Powell, 1957). These spheres are mounted in a block of balsa wood such that one ball extends below the block surface for contact with the solid to be tested, and the other is flush with the block surface. Two metal studs are positioned relative to the extended ball on the underside of the block to form a stable three point contact for the device. An important attribute of this design is that the contact area of the extended ball is very small,

only a few square millimeters, ensuring that heat transfer to the material being tested is primarily under steady-state conditions (Powell, 1957).

As with other measurement systems, the thermal comparator needs to be calibrated against at least two thermal conductivity standards (Powell, 1957). To begin, the test material is allowed to reach thermal equilibrium at a desired temperature. The comparator is heated, or cooled, to thermal equilibrium at a temperature different from the test material. The comparator is then placed in contact with the sample. The ball in contact with the sample changes temperature more rapidly than the ball suspended above the sample's surface. The difference between the microvolt outputs of the two thermocouples is read after 10 seconds, which results in a cooling or heating rate. Thermal conductivity is a linear function of this rate. After calibration with two standards to produce the standard curve, it can be used with cooling rates for unknown materials to determine the thermal conductivity of those materials (Powell, 1969).

The thermal comparator method has been adapted for powders and liquids in addition to solids. Its short equilibration time and minimal heating of the sample limit errors due to moisture loss or migration. In liquids, the short measurement time and top surface heating serve to

reduce problems associated with convection (Ziegler and Rizvi, 1985).

2. Guarded Hot Plate Method

The guarded hot plate method is a well established and commonly used steady-state procedure for the determination of thermal conductivity. The method has been described in detail by the American Society of Testing Materials (ASTM, 1987). Briefly, the method involves placing the test material and a standard reference material, both of known thickness, between a heat source and a heat sink. The sides of the apparatus are either insulated, heated, or both to minimize lateral heat loss, a major source of potential error, and to force the heat flux to be unidirectional. The temperature is carefully measured at all material interfaces. After the equipment has reached thermal equilibrium, the heat flux through the standard reference can be calculated and used to determine thermal conductivity of the test material through application of Fourier's law.

A variation of this method, the plane layer method, was developed by Filippov (1968). This technique involves placing the entire device in an autoclave, thereby making guard heaters on the top and lateral surfaces unnecessary. This arrangement also permits conductivity measurements at

temperatures up to 300°C, and pressures to 100 atm.

An automated version of the guarded hot plate was described by Zabawsky (1957). In this case, the system used was a modified version of the procedure described by ASTM (1987). There are several principle changes. The first is that the guard heaters are automatically controlled to maintain their temperature within 0.004°F of the test and reference materials. This serves to reduce the lateral heat loss error below that of standard guard heaters. Another modification is a redesign of the cold plate from a manifold to a labyrinth form. The entire assembly is held in a frame at a constant pressure of 50 lb/sq ft, and placed in a temperature-humidity controlled chamber. Results are reported to be within 1% of those measured by the National Bureau of Standards.

The guarded ring hot plate method is regarded as accurate and reliable. However, it is cumbersome, the apparatus is complicated, and the technique is slow (Bennett, et al., 1962).

3. Fitch Method

The Fitch method, or, more accurately, the Fitch apparatus, was invented by A.L. Fitch (1935). The instrument was originally designed to determine the thermal conductivity of poor heat conductors. The equipment, as

described by Fitch (1935):

"consists essentially of two highly polished, flat, nickel-plated copper blocks with suitable thermocouple connections and heat insulation. The lower block is approximately 4.4 cm in diameter and has a mass of 346 gm. It is imbedded in a good insulating material and has a copper-constantan thermojunction imbedded at its center. The copper wire runs out to a copper binding post and the constantan wire runs out to a second, insulated copper binding post with a constantan nut. The upper block serves as the bottom of a well-insulated vessel with a capacity of about 800ml. This block has a diameter of about 8.4 cm and an unknown mass. The thermal junction in this block is near one edge. The wires run out to copper binding posts and the constantan wire is held by a constantan nut as in the case of the lower block."

To achieve a desired temperature for study, the upper vessel is filled with a material, i.e. boiling water, dry ice ,etc., that will provide that temperature. The test material is positioned on the lower block, and the upper block and vessel are set on top of the test material to insure good thermal contact. A galvanometer is used as a measure of temperature difference between the upper and lower blocks. The slope of a semi-log plot of galvanometer deflection versus time is used to calculate thermal conductivity of the test material.

Several methods have been reported using modified versions of the Fitch method. Bennett, et al. (1962) altered the technique to estimate thermal conductivities of fruit and vegetable components. Their changes included completely surrounding the test specimen with thermal

insulation to insure unidirectional heat flow, and adding an adjustable plug to minimize plug-sample-plate contact resistance without compacting the sample, possibly altering its physical characteristics.

Another variation on the Fitch method was developed by Murakami and Sastry (1984) for the estimation of thermal conductivity of small food particulates. In this version, the copper plate was replaced with a 6.35 mm diameter rod. The copper plug was tapered to a diameter equaling that of the rod, and was insulated by embedding it in a block of wood with only the small end exposed. The test sample was mounted between the rod and the exposed plug. In this way, materials a minimum of 6.4 mm in diameter could be measured. Heat flow was assumed to be unidirectional through the sample. Tests were run for 440 seconds, and a 20°C temperature difference between the plug and rod was required to give a 1.5°C temperature change in the plug.

Zuritz, et al. (1987b) took a different approach to the Fitch method. They undertook to modify the theory involved rather than the equipment. Fitch's original theory allows for the apparatus to reach a quasi-steady state, neglecting transient or heat storage effects within the test material. However, there is an optimum sample thickness to be maintained if the quasi-steady state assumption is to be valid. Deviation from this optimum increases the error

associated with the measurement. For soft, compressible materials, the Fitch method may not be applicable due to compression below the optimum sample thickness.

To overcome these limitations, Zuritz, et al. (1987b) used the full solution to the transient heat transfer equation. They found that as long as there is unidirectional heat flow, the method is independent of sample thickness, and accurate thermal conductivities can be determined in the relatively short time of 100 seconds.

In general, the Fitch method is both simple to run and adaptable to a range of materials. It does not require complex instrumentation. And the reliability of the method is adequate for many applications (Bennett, et al., 1962).

4. Line Heat Source Method

The possibility of using a line-source technique for the determination of thermal conductivity was first recognized as early as 1888 (Nix, et al., 1969). The method was first used successfully by van der Held and van Drunen (1950).

The probe is an approximation of a line heat source of constant power placed in a homogeneous object at a uniform initial temperature (Hooper and Lepper, 1950). Construction of the probe, and the theory behind its performance has been thoroughly discussed by several

investigators (Hooper and Lepper, 1950; Nix, et al., 1967; Nix, et al., 1969; Sweat and Haugh, 1974; Sweat, 1976; Baghe-Khandan, et al., 1981a). Briefly, the probe is generally constructed from a needle mounted to a commercial thermocouple connector. Fitted either in a hypodermic needle or outside a solid needle are a constantan heater wire and a chromel-constantan thermocouple electrically isolated from each other and the needle. The thermocouple junction is located near the center of the needle length (Sweat, 1976).

While the probe is an approximation of a line source, corrections must be made for its finite length and width, as well as a nonhomogeneous cross section (Hooper and Lepper, 1950; Hooper and Chang, 1953; Nix, et al., 1967). The air space within the needle, thermocouple location, and heat conduction within the probe would seem to be potential sources of measurement error. However, this was found not to be the case (Sweat, 1976). A modification of the basic probe design (Baghe-Khandan, et al., 1981a) eliminated the air space problem by mounting the heater and thermocouple wires on the outer surface of the needle. This served to greatly simplify probe construction, and yielded longer lasting, more durable probes.

The working equation for using the line-source probe is given in equation (1) (Hooper and Lepper, 1950; Nix, et

al., 1967; Nix, et al., 1969; Reidy and Rippen, 1971; Lozano, et al., 1979; Baghe-Khandan, et al., 1981a&b).

$$T_2 - T_1 = (Q/4\pi k)\ln(t_2/t_1) \quad (1)$$

where T_2 and T_1 and t_2 and t_1 are the final and initial temperatures and times, respectively. A plot of temperature versus $\ln(\text{time})$ yields a straight line with slope, $s = Q/4\pi k$, and, therefore, $k = Q/4\pi s$.

The line-source probe is an "absolute" method not requiring calibration (Hooper and Lepper, 1950). However, to account for slight differences between probes, calibration of the probe against thermal standards is recommended (Sweat, 1976; Lozano, et al., 1979).

There are many advantages to using the line-source probe for estimating thermal conductivity:

- (1) Short test duration of typically 2-5 minutes (Nix, et al., 1967; Sweat and Haugh, 1974; Lozano, et al., 1979)
- (2) Little or no sample preparation is required as long as the sample maintains the infinite boundary condition assumption required to solve the heat transfer equation (Nix, et al., 1967; Sweat, 1976)
- (3) Only a small temperature increase during testing permits measurements on temperature sensitive materials (Nix, et al., 1967)

- (4) The method is applicable to a wide range of temperatures (Nix, et al., 1967)
- (5) Data collection is easily automated with a digital computer (Sweat, 1976)

The method does have some problems associated with it. Potential for error due to the relatively large temperature gradient between the thermocouple, the outer surface of the needle, and the test material, and the need to monitor the surface temperature of small samples for a temperature rise indicating loss of the boundary condition are two such problems (Sweat and Haugh, 1974). Overall, for estimating the thermal conductivity of most materials, the line heat source probe method is equal in accuracy to the guarded hot plate (Hooper and Lepper, 1950).

5. Empirical Methods

One of the most difficult problems associated with estimating the thermal conductivity of liquid foods is that of convection. To achieve accurate results, convection must be either eliminated or be accounted for in some way. An alternative to physically measuring the thermal conductivity of a food material is to estimate the value empirically based on its physical and chemical composition. Temperature may also be a factor in the empirical estimation.

Theoretical, empirical, and semi-empirical models for the estimation of thermal conductivity of liquid foods have been well summarized by Cuevas and Cheryan (1979). Some of these models are briefly reviewed below.

Cuevas and Cheryan classified the models into two categories: those that did not include temperature as a variable, and those that did. Models excluding temperature will be reviewed first.

a. Temperature Independent Models

One of the first empirical models of thermal conductivity was developed by Eucken (1940) as a modified version of an equation proposed by Maxwell (1904). This equation, shown below, is applicable to a system containing a distribution of particles.

$$k = k_c \left[\frac{1 - (1 - a(k_d/k_c))b}{1 + (a - 1)b} \right] \quad (2)$$

where:

$$a = 3k_c / (2k_c + k_d) \quad (3)$$

$$b = v_d / (v_c + v_d) \quad (4)$$

The Maxwell-Eucken equation has been adapted to predict the thermal conductivity of various food systems by Lentz and Van den Berg (1977). They made the following assumptions in adapting equation 2:

- (1) density of water in the system is 1.00 gm/ml.

- (2) density of carbohydrate is 1.55 gm/ml, fat is 0.92 gm/ml, protein is 1.35 gm/ml, and salts are 1.9 gm/ml.
- (3) mean density of non-fat solids is 1.5 gm/ml.
- (4) mean thermal conductivity of food solids is 1.7 mW/cm-°K.
- (5) at 20°C, water is the continuous phase, and the dispersed phase is the solids.

Most estimated thermal conductivities were found to be within 10% of the corresponding experimental values.

A three-dimensional, two component, isotropic physical model was suggested by Kopelman (1966). In this model, one component is randomly distributed throughout the other.

The Kopelman equation is as follows:

$$\frac{k}{k_c} = \frac{1 - M^2(1 - k_d/k_c)}{1 - M^2(1 - k_d/k_c)(1 - M)} \quad (5)$$

There are certain restrictions imposed on the model: the continuous and discontinuous components may form two phases or be the same state, the dispersed particle size must be small in comparison to the system, the particle distribution must be random, and the discontinuous component must be dispersed above the atomic level.

This model seems applicable to such systems as air-sucrose foams, butter, and ice cream.

A linear empirical equation not including temperature was developed by Sweat based on measurements taken with a line heat source probe. The model is:

$$k = 0.148 + 0.00493W \quad (6)$$

The model is restricted to water contents in excess of 60%, and is accurate to within 15% for estimating the thermal conductivity of fruits and vegetables.

b. Temperature Dependent Models

One of the first empirical equations that incorporated temperature as a variable was proposed by Riedel (1949). His model for estimating thermal conductivity (kcal/hr/m/°C), developed for cane and grape sugar solutions, as well as a variant for milk products, is as follows:

$$k = (0.486 + 0.00155T - 0.000005T^2)(1 - 0.0054S) \quad (7)$$

and, for milk products:

$$S = (10 + 2f)\frac{1}{2} \quad (8)$$

This model may be used for estimating thermal conductivity of fruit juices and sugar solutions of up to 60% solids. The model has an apparent accuracy of 1% for all varieties of liquid foods, independent of the material.

A variation of Riedel's model was developed by More and Prasad (1988). Their equation is given below. It was

$$k = (0.59 + 0.0012T)(1 - 0.0078S) \quad (9)$$

adapted for use on concentrated whole milk products, 37-72.4% total solids, over a temperature range of 40-90°C. Values calculated from the empirical equation are reported to fit experimental data to within 0.88%.

Another temperature dependent empirical equation has been developed by Kolarov and Gromov (1973). Their "universal equation", given below, is for estimating thermal conductivity in the ranges of 30-80°C and 4-60% solids.

$$k = \frac{(5.75 - 0.048S) \times 10^{-4}}{1.11 - 0.0036T} (993 + 5S - 0.57T) \quad (10)$$

For the study of syrups and fruit and vegetable juices, this model has an accuracy of up to 8% when compared to experimentally determined thermal conductivities.

Effects of composition and temperature on the thermal conductivity of milk products was studied by Fernandez-Martin and Montes (1972). By examining whole milk, skim milk, and half-and-half over a range of temperatures, they developed a quadratic equation for conductivity (cal/s-°C-cm) shown below.

$$k = A + BT + CT^2 \quad (11)$$

The coefficients A, B, and C are defined as follows:

$$A = A_0(1 + A_1F + A_2F^2) + A_3s + A_4(1 + A_5F)f \quad (12)$$

$$B = B_0(1 + B_1F + B_2F^2) + B_3s + B_4(1 + B_5F)f \quad (13)$$

$$C = C_0(1 + C_1F + C_2F^2) + C_3s + C_4(1 + C_5F)f \quad (14)$$

and equations 12, 13, and 14 are inserted into equation 11.

Values for empirical parameters A_0-A_5 , B_0-B_5 , and C_0-C_5 have been tabularized by Fernandez-Martin and Montes (1972).

This system of equations agrees within 1.5% to 3.0% of the experimentally determined values for milk for fat contents of 0.12-11.17% and temperatures of 5-75°C.

An equation developed for estimating thermal conductivity (cal-cm)/(s-°C-cm²) of cream is:

$$k=[12.63+0.051T-0.000175T^2][1-(0.843+0.0019T)V_d]10^{-4} \quad (15)$$

Equation 15 estimates thermal conductivity to within 10%.

Since the review by Cuevas and Cheryan (1979), another non-temperature dependent model for estimating thermal conductivity has been elucidated. The model is an adaptation of the Effective Medium Theory (EMT) (Mattea, et al., 1986). Briefly, a homogeneous, or effective, medium is assumed to exist with an overall conductivity of k_m . Local disturbances created by non-homogeneities are treated as fluctuations of the effective medium. The general form of the equation is:

$$\sum_{i=1}^n \varepsilon_i \frac{k_m - k_i}{k_i + 2k_m} = 0 \quad (16)$$

where ε_i and k_i are the volume fraction and thermal conductivity of the i-th component, respectively. Potatoes and sugar-based fruits are treated as two

component systems where the cellular material represents the continuous phase, and pores, or intercellular air spaces, are the discontinuous phase. In this way, volume fraction of the pores is total porosity, ϵ , and volume fraction of the cellular material is $\epsilon_c = 1 - \epsilon$.

The model is a good predictor of thermal conductivity at various stages of dehydration provided that a value for porosity can be obtained. For potatoes, the model was found accurate to within 7.5%. While for pears, empirically derived data agreed to within 4% of experimental values.

6. Bead Thermistor Probe

A thermistor, or "thermal resistor", is defined as a ceramic semiconductor which exhibits a change in electrical resistance as a function of temperature. Most thermistors, the negative thermal coefficient (NTC) type, display a decrease in resistance with an increase in temperature. A representative plot of thermistor resistance as a function of temperature is given in Figure 1. The NTC type thermistors are made of oxides of metals such as manganese, nickel, cobalt, copper, iron, and titanium (Veco, 1968;

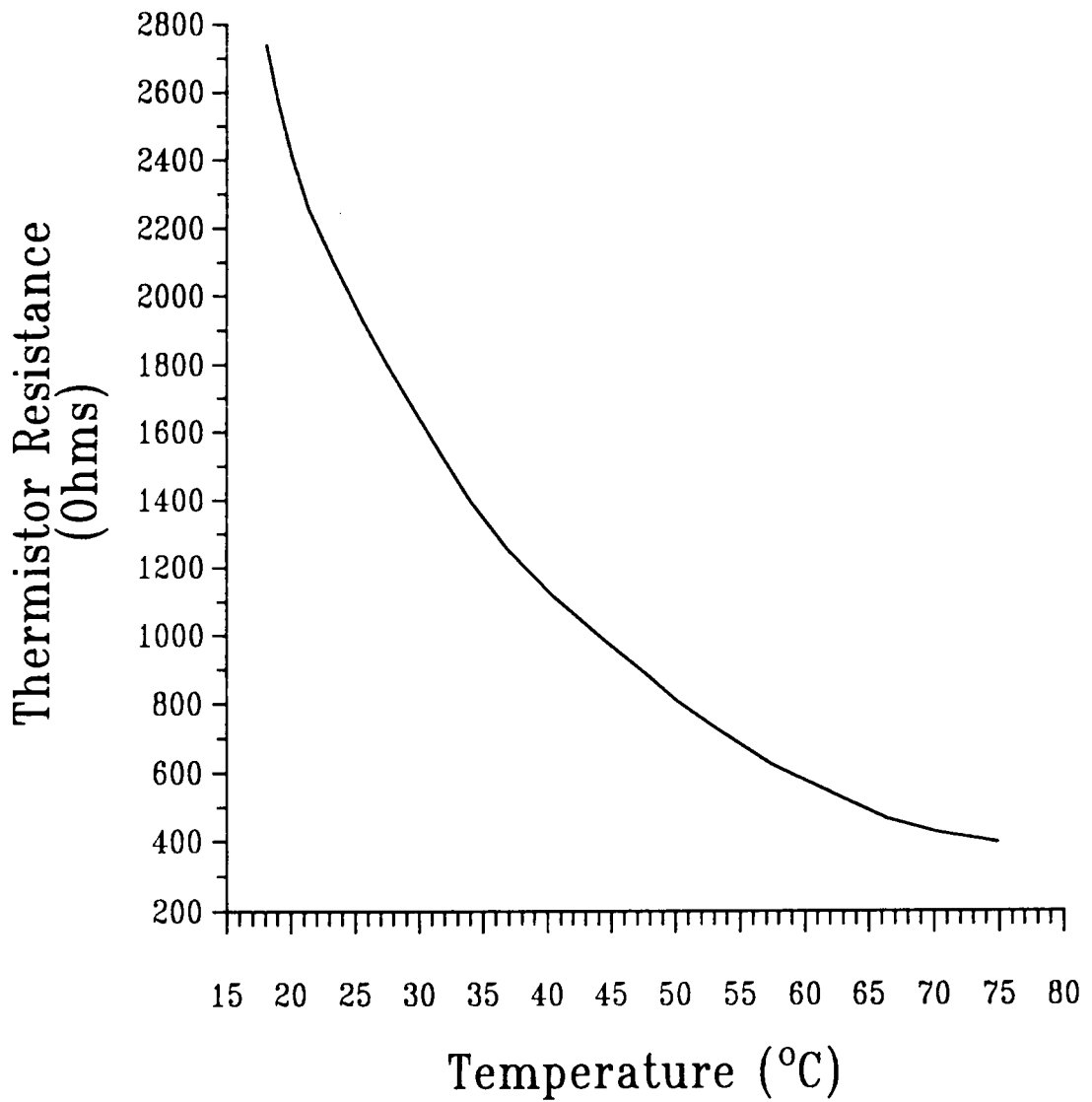


Figure 1. Representative Plot of Thermistor Resistance as a Function of Temperature

Thermometrics, 1986). Another property of thermistors is that, when supplied with a current, they are self-heating. Therefore, a single ovate thermistor bead can function as both a temperature sensor and a heat source (Balasubramaniam, 1975). To electrically isolate the thermistors from the test media, beads used for this study were encased in glass, and the leads from the beads were teflon coated. However, other shapes and encapsulation materials are available.

The solution to the heat transfer problem of a spherical heat source in an infinite medium and the theory behind that solution have been covered extensively by Balasubramaniam and Bowman (1974) and Valvano (1981). A major advantage of a thermistor-based thermal property measurement system is that a single test yields both thermal conductivity and thermal diffusivity of the test material (Balasubramaniam, 1975; Balasubramaniam and Bowman, 1977; Kravets and Larkin, 1986). As pertains to this study, a brief review of the solution to the proposed heat transfer problem as applies to thermal conductivity measurements is presented.

To fit the theory to a thermistor probe, several assumptions must be made. These include: 1) a perfectly spherical bead; 2) uniform heat generation within the bead; 3) sample of infinite boundary conditions; 4) minimal

contact resistance between the probe and the sample; and 5) a measurable bead effective radius and thermal conductivity (Kravets and Larkin, 1986). The solution of the heat transfer problem yields a simple algebraic expression for the thermal conductivity (equation 17) of the test medium. This equation is:

$$\frac{1}{k_m} = \frac{4\pi r R_T (dT)}{(V_{SS})^2} - \frac{1}{5k_b} \quad (17)$$

Heat generation rate within the bead, at $t > 0$, is expressed in equation 18 (Balasubramaniam and Bowman, 1974; Balasubramaniam, 1975; Valvano, 1981; Woodbury, 1984; Dougherty, 1987).

$$q(t) = \Gamma + \beta t^{-1/2} \quad (18)$$

As can be seen from this equation, heat generation within the bead is linearly proportional to the inverse square root of time. At equilibrium, power dissipated to the medium surrounding the bead is equal to power generated within the bead. This power can be expressed as (voltage)²/resistance. At equilibrium, however, resistance of the thermistor remains constant. Therefore, power output of the bead to the surrounding medium can be modeled as simply the square of the voltage drop across the thermistor. A plot of (voltage)² versus (time)^{-1/2} (Figure 2) yields a straight line with an intercept of $(V_{SS})^2$, the steady state voltage squared.

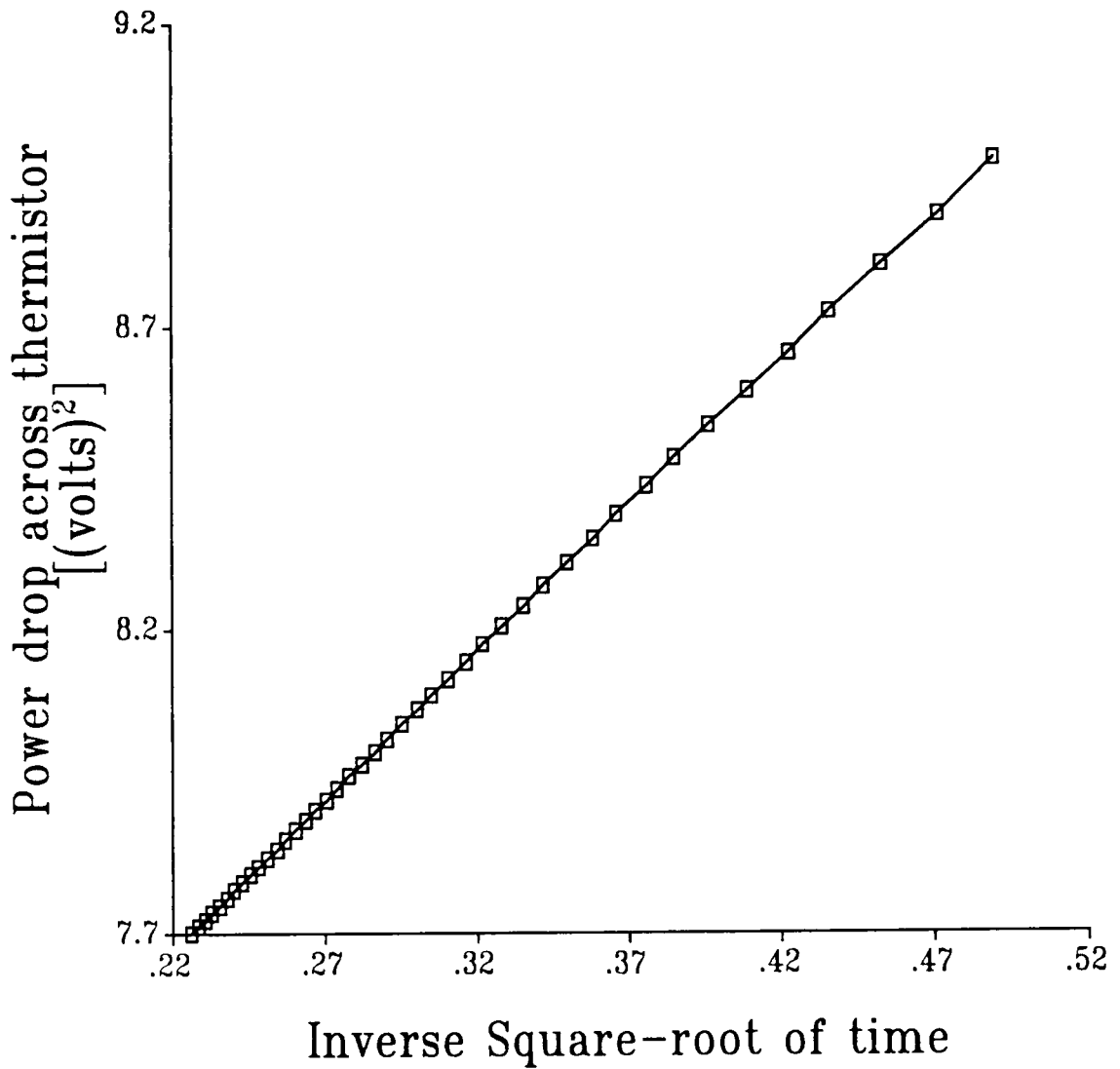


Figure 2. Voltage squared versus the inverse of the square root of time. A representative plot of experimental data.

Thermal conductivity (k_p) of the bead and its effective radius (r) are determined by standardization of the probe against standard reference materials of known thermal conductivity. The remaining variables in equation 2, final thermistor resistance, R_T , and temperature step change, dT , are obtained by direct measurements during the test procedure.

Early work on developing the use of the thermistor for determination of thermal properties was done by Chato (1968). This development centered on a transient method for taking thermal property measurements in biomaterials. The probe, a bare thermistor bead with insulated leads, was inserted by means of a hypodermic needle (Chato, 1968). While the technique is an invasive one, trauma resulting from the insertion of the thermistor probe via the hypodermic needle is minimal, and deep tissue rather than simply surface measurements can be obtained (Balasubramaniam, 1975). Balasubramaniam (1975) found the method both very accurate, within 1-2% of literature values at 70°F, and precise, small standard deviations, for determining thermal conductivity. He also found the thermistor technique compared favorably with literature values for biomaterials determined by both the line probe and thermal comparator methods.

The study of biomaterials using the thermistor technique was expanded by Valvano (1981) to quantify the rate of perfusion. Perfusion is defined as "blood flow through the capillary network", and is a factor in the heat transfer equation applied to biomaterials. Presence of fluid moving within living tissue changes the volumetric heat generation rate within the bead by altering the surface heat transfer coefficient of the bead. In this case power is no longer proportional to $(\text{time})^{-1/2}$ (Valvano, et al, 1984). The perfusion rate can be determined from both steady-state and transient solutions to the heat transfer equation. Both thermal conductivity and thermal diffusivity are estimated and considered "effective" values. These approximate values are used in an iterative solution to the heat transfer equations. It was found that three iterations usually gave convergence to within 1% of the effective thermal conductivity. This value of thermal conductivity was then used to determine thermal diffusivity and perfusion rate (Valvano, 1981; Valvano, et al., 1984).

Another adaptation of the thermistor technique was developed by Woodbury (1984). In this case, thermal conductivity was used as a means of estimating moisture content of a highly porous glass fiber insulation material. Insulation material was tested over a moisture content

range of zero to 100% on a dry mass basis (Woodbury and Thomas, 1985b). A major difficulty in this work was attaining a uniformly wetted sample ensuring that the desired moisture content was maintained around the probe during the test. Results of this investigation revealed a distinct nonlinear relationship between moisture content and thermal conductivity. However, the method proved to be accurate to within 5% over a wide range of low thermal conductivities (Woodbury, 1984; Woodbury and Thomas, 1985a; Woodbury and Thomas, 1985b).

A detailed study of the bead thermistor method was performed by Dougherty (1987). A computer-based data acquisition and process control system was developed to regulate the procedure. It was found that sampling rate had no effect on the estimate of thermal conductivity. Response time of the thermistor was also found to be of no importance except when free convection in liquids or stem losses in insulation materials were present.

Accuracy of the method was determined to be closely correlated to availability of adequate standard reference materials. Reference materials were used to ascertain the bead thermal conductivity and effective radius. Errors associated with the estimation of these parameters were the largest sources of error in the final estimation of thermal conductivity of the test medium (Kravets and Larkin, 1986).

C. Thermal Standards

A major concern when using thermal conductivity measurement equipment is calibration, or standardization, of the equipment against standard reference materials of known thermal properties. However, there is at present a serious lack of thermal conductivity standards in the region of most intermediate and high moisture content food materials. For potential liquid standard materials, accurate thermal conductivities and their temperature dependence are difficult to find (Woolf and Sibbitt, 1954). This deficiency of reliable thermal standards can often lead to significant measurement error (Tseiderberg, 1965; Chato, 1968). The National Bureau of Standards (NBS) thermal conductivity standard reference materials that most closely bracket water, $0.597 \text{ W/m-}^\circ\text{K}$, at 293°K (Touloukian, 1970) are $0.039 \text{ W/m-}^\circ\text{K}$ (fibrous glass batt) to $14.1 \text{ W/m-}^\circ\text{K}$ (stainless steel) (NBS, 1988). The NBS itself recognized this need, and recommended a search for new standard reference materials in the range of 0.015 to $5 \text{ W/m-}^\circ\text{K}$ to be tested over the temperature range of -200 to 1200°C (Rennex, 1985). However, at present, the NBS is not working to fill this need due to a lack of strong interest and a corresponding lack of funding for the necessary research (Dougherty, 1988).

In addition, values for materials to be used as thermal standards vary widely throughout the literature. This may be attributed to either inexact or inaccurate heat transfer models, or imprecise experimental techniques, or a combination of the two (Laurent, 1983). As an example, the thermal conductivity of water, in W/m-°K, at 20°C is reported to range from 0.590 (Batchelor, 1967) to 0.624 (Kaye and Higgins, 1928). This presents some difficulty in deciding which value to use for standardization. To minimize this potential source of error, the three standard reference values should be chosen from either the same reference or references using the same method of determination. While no one method can claim absolute accuracy, the error or bias associated with a given method should be systemic for all data determined by that method. These values are not necessarily the true values. However, the relative error between values should be smaller than the error between values determined by different methods.

D. Thermal Conductivity Values

There are many sources for thermal conductivity values of food materials. These compendia have been compiled by many researchers using a variety of measurement techniques. Representative lists of these data are presented in Tables 1 and 2. Summarized in Table 3 are values of thermal

Table 1. Thermal conductivity values for selected juice products (Choi and Okos, 1986)

food material	temperature (°C)	moisture content (%)	thermal conductivity (W/m-°K)
apple juice	20	87	0.559
	80		0.631
	20	70	0.504
	80		0.546
	20	36	0.389
	80		0.435
grape juice	20	89	0.567
	80		0.639
	20	68	0.496
	80		0.554
	20	37	0.396
	80		0.439
tomato juice	30	95.2	0.596
		70.9	0.472
		40.0	0.314
	60	95.2	0.630
		70.9	0.511
		40.0	0.358
	90	95.2	0.653
		70.9	0.542
		40.0	0.400
	120	95.2	0.665
		70.9	0.565
		40.0	0.439
150	95.2	0.665	
	70.9	0.581	
	40.0	0.474	
orange juice	15.5	89.0	0.553

Table 2. Thermal conductivity values for selected solid and semi-solid food products (Singh and Heldman, 1984)

food material	temperature (°C)	moisture content (%)	thermal conductivity (W/m-°K)
apple	2-36	85.6	0.393
applesauce	2-36	78.8	0.516
butter	46	15.0	0.197
cod	2.8	83.0	0.544
egg white	36	--	0.577
egg yolk	33	--	0.338
honey	2.0 69.0	12.6 14.8	0.502 0.623
raw potato	1-32	81.5	0.554
sausage mix	24	65.72	0.407
strawberry	14-25	--	0.675
turkey breast	3	74	0.502

Table 3. Thermal conductivity values for selected milk products

milk product	temperature (°C)	moisture content (%)	thermal conductivity (W/m-°K)
skim (Reidy, 1968)	7	90	0.547
	27		0.575
	47		0.600
	67		0.625
	87		0.642
whole (Reidy, 1968)	7	83	0.531
	27		0.558
	47		0.580
	67		0.600
	87		0.620
skim (empirical from Fernandez- Martin and Montes, 1972)	25	90	0.587
	35		0.604
	45		0.618
	55		0.632
	65		0.643
	75		0.654
	85		0.662
whole (empirical from Fernandez- Martin and Montes, 1972)	25	87.7	0.556
	35		0.573
	45		0.588
	55		0.601
	65		0.612
	75		0.620
	85		0.627
dairy products (Govorkov, 1950)	20	4% fat	0.592
		20% fat	0.382
		25% fat	0.370
		35% fat	0.348
		45% fat	0.324
		85% fat	0.201
		100% fat	0.168

conductivities both physically measured and empirically estimated, of some milk products, though additional sources, both empirical (Fernandez-Martin and Montes, 1977) and measured (Sweat and Parmelee, 1979) are available.

While much work has been done determining thermal conductivity, little work has been done on examining the temperature dependence of thermal conductivity in food materials. Work has been done in frozen foods due to the relative ease of handling a frozen product. However, at thermal processing temperatures, most food materials are very difficult to work with. With liquids, convection during measurement is a serious problem, and they need to be kept in a pressurizable container to prevent boiling. With either liquids or solids, moisture loss during the test is a concern. Current methods for measuring thermal conductivity are extremely difficult if not impossible to adapt to these high temperature conditions.

III. Materials and Methods

A. Introduction

The objectives of this study were accomplished in three phases, feasibility study, methodology documentation, and method evaluation. The feasibility study involved evaluation of the thermistor as a means of determining the thermal conductivity of food materials. Probes were temperature-resistance calibrated. Standardization of probes was required to find the probe's effective radius and thermal conductivity. Standard reference materials used were water, glycerin, and castor oil.

Methodology documentation was achieved by measuring thermal conductivity of ethylene glycol as a function of temperature. The 25°C to 125°C temperature range was divided into subranges with a different resistance thermistor used for each. Probes were calibrated and standardized at the temperatures at which they were used. A small pressurizable chamber was used to allow heating of the samples above 100°C.

The method evaluation phase was accomplished by evaluating the thermal conductivity of skim milk, whole milk, half-and-half, and cream as a function of temperature. Problems arose with the probes and control circuit, and efforts were made to correct these problems.

B. Feasibility Study

1. Methodology

Early work centered on testing the potential of the bead thermistor probe for determining thermal properties of liquid and high moisture food materials. Thermistors (Victory Engineering Co., Springfield, N.J.) chosen for this study were 2.54 and 1.52 mm diameter glass encapsulated beads with nominal resistances at 25°C of 1000 and 10000 ohms, respectively. Teflon coated leads and glass encapsulation electrically insulated the beads from the surrounding media. Probes were temperature-resistance calibrated in a Masterline 2095 Bath & Circulator (Marietta, OH) using a Keithley 195A Digital Multi-Meter (Cleveland, OH) and a 0.1°C division thermometer tested to meet National Bureau of Standards specifications (Fisher Scientific, Pittsburgh, PA). The temperature-resistance data was analyzed by linear regression to obtain coefficients, a_0 , a_1 , and a_2 in the following thermistor calibration equation:

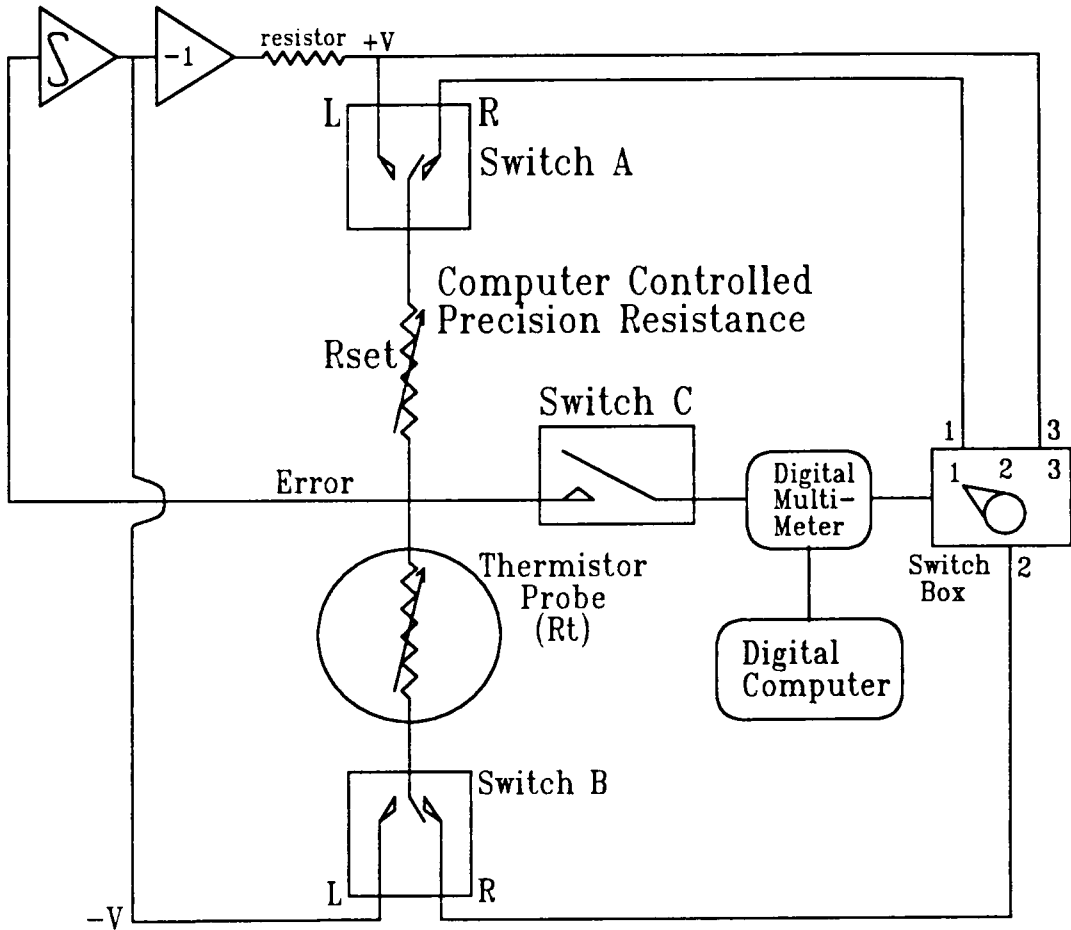
$$1/(T+273.15) = a_0 + a_1 \ln(R) + a_2 [\ln(R)]^3 \quad (19)$$

This equation, while containing non-linear terms for the independent variable, may be analyzed by linear regression because the coefficients are entered in a linear fashion (Myers, 1986).

After calibration, the probes were standardized against 3 reference media of known thermal conductivity. Standards used were glycerin (J.T. Baker Chemical), castor oil (Purchased at a local drug store), and distilled water. The thermal conductivities of these standards at 25°C are 0.285, 0.180, and 0.605 W/m-°K, respectively (Touloukian, 1970). CarboxyMethylCellulose (CMC type 7HCF, Hercules Incorp., Wilmington, DE) at 0.75%(w/v) was added to the water to inhibit convection around the bead during heating. This and all other data were analyzed in a spreadsheet programmed for linear regression, to determine the intercept of the voltage squared versus inverse square-root of time data, and the thermal conductivity bead equation.

The circuit used (Figure 3) is a modified version of that developed by Valvano (1981). A more detailed diagram of the integrator/inverter portion of the circuit is given in Appendix A3. The resistor placed after the integrator has a resistance of 0 ohms except where otherwise noted. The computer program used for process control and data acquisition is presented in Appendix A1. The test procedure was as follows: the computer read, via the multi-meter, initial thermistor resistance (R_T). By using equation (19), this resistance was converted to initial temperature. A temperature step-change of 2.5 - 5°C was added to the initial temperature, and this final desired

Integrator Inverter



Mode	Switch A	Switch B	Switch C	Switch Box
Idle	R	R	Off	-
Rset	R	L	On	1
Rt	L	R	On	2
Run	L	L	Off	3

Figure 3. Diagram of circuit design and switch settings for desired operational modes

temperature was run back through equation (19) to yield the final desired resistance. The computer-controlled precision resistors (R_{set}) were then set as close as possible to the desired final resistance. The resistance was then measured with the multi-meter to determine the actual final resistance (R_F), and, by equation (19), the actual final set temperature. Actual temperature step change was the difference between actual final temperature and initial temperature. When data collection began, voltages of opposite sign were applied across the thermistor and R_{set} . The circuit was designed such that voltages applied across the thermistor and set resistors equilibrated within 0.3 seconds. Equilibration was expressed as an increase in power (voltage) supplied to the probe as it heats to the desired temperature step change. Once the circuit was equilibrated and the desired temperature step change achieved, voltage across the probe began decreasing. With a voltage placed across the thermistor, it began to heat, and its resistance decreased. When the thermistor's resistance equaled the set resistance, and with the same magnitude voltage drop across both, the system was in equilibrium. As the thermistor heated the test medium, the voltage necessary to maintain a constant power output to the medium decreased. Data collection proceeded at 3 samples per second for 20 seconds

with voltage and time being recorded. The system was allowed to return to thermal equilibrium by stirring the medium and waiting one minute between measurements. Even though system equilibrium was reached in 0.3 seconds, the initial 4 seconds of data from each experiment were not used in the data analysis. This was to insure that only data from an equilibrated system was analyzed. The data was plotted as (voltage)² versus (time)^{-1/2} (Figure 2). The intercept of this line, as determined by linear regression, was the steady-state voltage squared achieved at infinite time. A representative plot of the residuals of the regression equation is shown in Figure 4. Each reference material was measured ten times. Mean values for the final resistance (R_F), temperature step-change (dT), steady-state voltage squared [$(V_{SS})^2$] and standard reference medium conductivity (k_m) were used in the following equation to determine the bead thermal conductivity ($1/k_b$) as a function of effective radius:

$$\frac{1}{k_b} = \frac{20\pi R_T(dT)}{(V_{SS})^2} r - \frac{5}{k_m} \quad (20)$$

This data was plotted for each standard reference material as shown represented in Figure 5. The intersection point of the three standardization lines yielded the effective radius and thermal conductivity of the thermistor bead probe. If the lines failed to intersect, the center of the

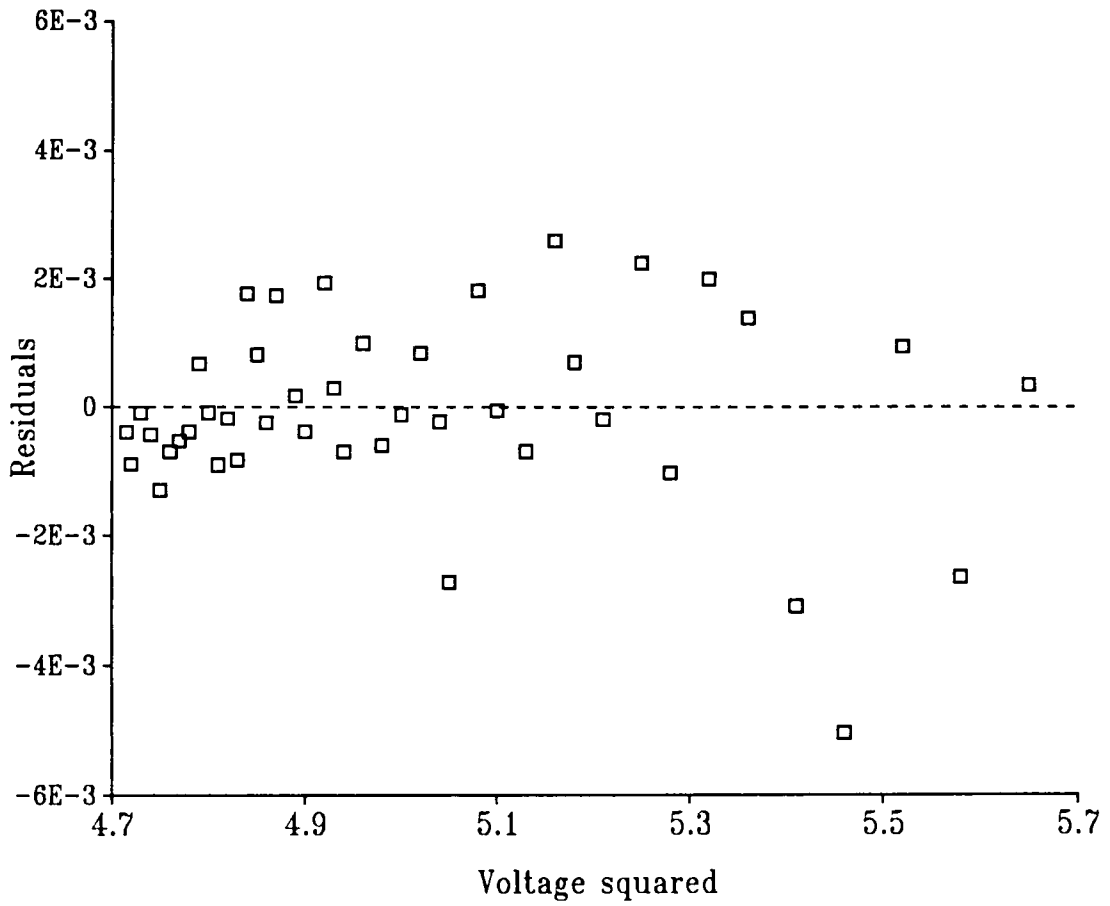


Figure 4. Representative plot of residuals of voltage squared versus inverse square root of time regression

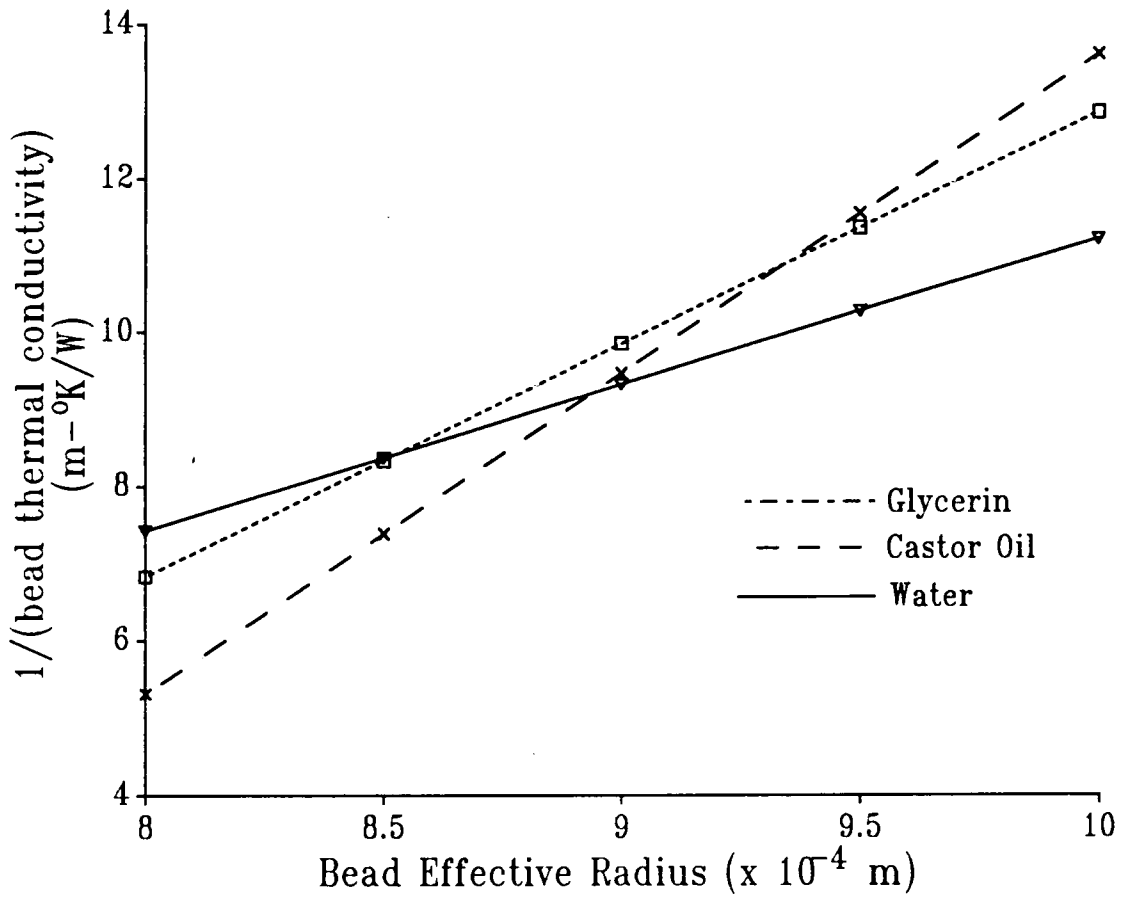


Figure 5. Thermistor bead probe standardization plot yielding probes thermal conductivity and effective radius

formed triangle, determined by averaging the values of the three vertices, was used to ascertain the bead parameters.

2. Minimum Particle Dimension

Minimum acceptable particle size which maintains the infinite boundary condition assumption was determined by drilling holes in a block of wood 8.6 x 8.6 x 45.5 cm. Ten holes with diameters ranging from 25.4 mm to 4.0 mm were drilled such that depth exceeded diameter to ensure that diameter was the limiting dimension. Pine was chosen for this experiment because its thermal conductivity (0.1413 W/m-°K, CRC, 1971) is different from that of the castor oil (0.180 W/m-°K, Touloukian, 1970) used to fill the wells. Using the test procedure, 10 measurements were made in each well using the 2.54 mm diameter 1 kOhm probe. Average thermal conductivity was plotted as a function of hole diameter.

C. Methodology Documentation

1. Thermistor Bead Calibration

Repeated attempts were made to construct encapsulated probes from bare beads. However, an adequate seal to prevent the test medium from coming in contact with the leads from the bead could not be maintained. Therefore, glass encapsulated thermistors with teflon coated wire leads (Victory Engineering Co., Springfield, N.J.) were chosen for the remainder of this study. These probes were 2.54 cm in length, were 1.52 mm in diameter, and had nominal resistances at 25°C of 1, 2, 10, and 20 kOhms. Thermal conductivity was examined at 10°C increments over a 25 to 125°C temperature range. Different resistance thermistors were used to evaluate different subdivisions of the 25 to 125°C temperature range. This range was subdivided into 25-35°C using the 1 kOhm probe, 45-55°C using the 2 kOhm probe, 65-95°C using the 10 kOhm probe, and 105-125°C using the 20 kOhm thermistor probe.

The 1 and 2 kOhm probes were calibrated as described in section III.B.1. The 10 and 20 kOhm probes were calibrated in a Haake A82 circulating bath (Haake-Buchler Instruments, Saddlebrook, NJ) filled with stabilized, high temperature bath oil (Fisher Scientific, Pittsburgh, PA). Probe resistance and temperature were recorded over a range 15°C below to 15°C above the range to be studied. Temperature-

resistance data was analyzed as for equation 19.

2. Thermistor Bead Probe Standardization

After calibration, each probe was standardized for each temperature the probe was to be used with the 3 standard reference media of glycerin (J.T. Baker Chemical), castor oil (Fisher Scientific), and distilled water. The thermal conductivities of the standard reference media as a function of temperature are presented in Appendix A2. CarboxyMethylCellulose (CMC type 7HCF, Hercules Incorp., Wilmington, DE) at 0.75%(w/v) was added to the water at 25°C and 35°C to inhibit convection around the bead during heating. At 45°C, CMC was added at the 1.25%(w/v) level. At 55-85°C, 1.5%(w/v) agar was used to inhibit convection in the water. For temperatures 95°C and above, Kelset Algin KSI-74292A (Kelco, Rahway, NJ) was used at the 0.9%(w/v) level. To prepare the Kelset, 800 mL of distilled water was acidified with 15 mL of 1N HCl. Kelset (7.5 gm) was slowly added to the water to insure complete hydration. The mixture was then neutralized with 15 mL of 1N NaOH. The thickened water was deaerated by heating to 80°C under vacuum. In all cases, thermal conductivity of water was taken to be the same as pure water. The use of thickeners to inhibit convection in water is a common and acceptable practice (Chato, 1968; Sweat and Haugh, 1974;

Balasubramaniam, 1975; Lozano, et al., 1979; and Valvano, 1981).

Several references were examined as potential sources for obtaining thermal conductivity data for the standard materials at the temperatures studied (Kaye and Higgins, 1928; Kern, 1950; Tseiderberg, 1965; Woodams and Nowery, 1968; Touloukian, 1970; Vargaftik, 1975; Mohsenin, 1980; and Holman, 1986). Upon evaluation of these sources, only Kaye and Higgins (1928) and Touloukian (1970) were used to supply the thermal conductivity data presented in Appendix A2.

Beginning at 55°C with the water sample, and at temperatures above 65°C for all samples, a small chamber (Figure 6), pressurizable to prevent sample boiling during heating and data collection, was used. All components of the chamber were constructed of stainless steel. The tube comprising the body of the chamber, end caps (16AMP-Solid End Cap), and clamp (13 MHHM type 304SS) were manufactured by Tri-Clover Corp. (Kenosha, WI). The pressure release valve was a Whitey Toggle Valve (#SS-1GS4, Whitey Co., Highland Heights, OH). The chamber bottom, side arm, 90° elbow (#SS-4-TSW-9, Cajon Co., Macedonia, OH), and vertical arm connections were silver soldered. Connections to the pressure release valve were made by Swagelok (Solon, OH)

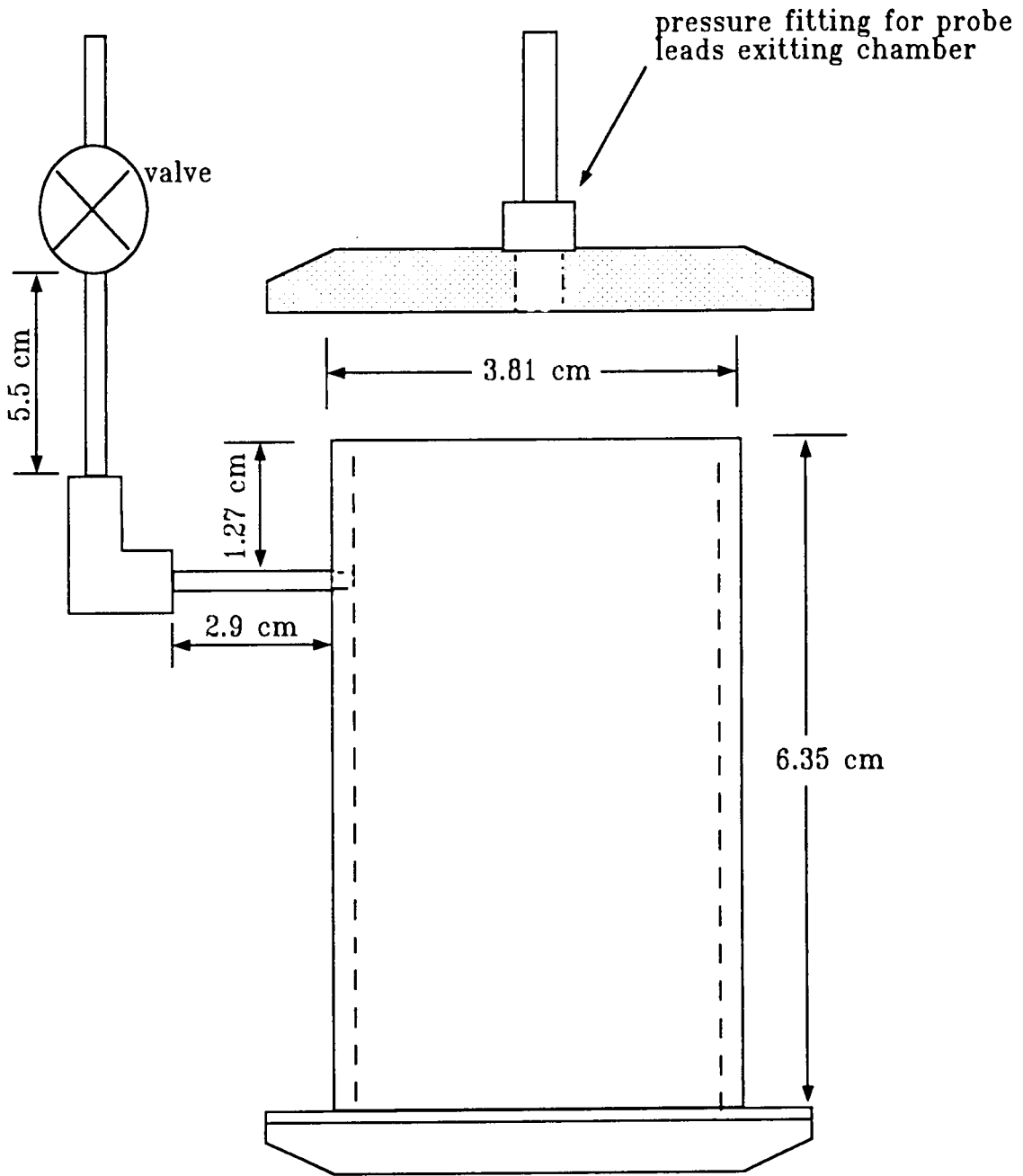


Figure 6. Diagram of pressurizable chamber

compression fittings. An advantage in using the chamber was that the sample could be completely submerged in the oil bath, thereby bringing the sample, air space above the sample, and the probe to thermal equilibrium at the temperature to be studied. This helped to avoid temperature gradients resulting from the difference between the chamber temperature and the ambient air temperature.

The circuit and experimental procedure used were the same as that described in section III.B.1.. This procedure was used as described for all temperatures up through 105°C. At 105°C, the linearity of the (voltage)² versus (time)^{-1/2} plots began to worsen for all 3 reference materials. Beyond this temperature, the viscosities decreased to the point where the 2.5 to 4°C step change was sufficient to allow convection to begin around the tip of the probe. Convection was expressed as curvature in the normally straight (voltage)² versus (time)^{-1/2} plot, and a corresponding decrease in the correlation coefficient as determined by regression of the voltage squared-inverse square-root of time data. At 115°C, it became necessary to reduce the test duration to 15 seconds to avoid the convection effects that became apparent near the end of the normal 20 second data collection period. To maintain the largest possible number of data points for the regression, the first 4 seconds of data were included in the analysis.

At 125°C convection became more pronounced and it was necessary to further shorten the test duration to 7 seconds. At this temperature, the initial 2 seconds of the data collection period were required for the circuit to reach equilibrium between the thermistor and the set resistance (R_{set}), and, therefore, the first 2 seconds of data were excluded from analysis.

3. Minimum Particle Size Experiment

Minimum particle diameter was reevaluated by measuring thermal conductivity at room temperature (20°C) in a series of decreasing diameter wells drilled in a block of aluminum and filled with glycerin. The wood block used previously was discarded when it was observed that the castor oil was absorbed into the wood causing it to swell. This absorption and swelling changed the hole diameters and blurred the boundary where the castor oil ended and the wood began. Holes were drilled in the aluminum such that the depth of each well exceeded its diameter to insure that diameter was the limiting dimension. Aluminum was chosen for the block material because its thermal conductivity (204 W/m-°K, Singh and Heldman, 1984) is much higher than that of glycerin (0.285 W/m-°K, Kaye and Higgins, 1928). Therefore, when the infinite boundary condition assumption on which the heat transfer equations are based was

violated, the change in measured thermal conductivity due to the boundary material was obvious. Hole diameters ranged from 12.7 to 2.78 millimeters. Thermal conductivity of the glycerin was measured ten times in each well, and the average thermal conductivity was plotted against hole diameter.

4. Accuracy and Coefficient of Variation Determination

Accuracy and percent coefficient of variation (%COV) of the method was determined by running the method at each temperature on a material of known thermal conductivity. Ethylene glycol (Fisher Scientific, Pittsburgh, PA) was chosen as the test reference. Thermal conductivity values for ethylene glycol over the 25-125°C temperature range were obtained from the Thermophysical Properties Research Center (TPRC) Data Series on the Thermophysical Properties of Matter (Touloukian, 1970), and are presented as the literature thermal conductivity values in Section IV.C.5 (Table 8). Due to the viscosity of ethylene glycol, it was necessary to inhibit convection during the test. To achieve this inhibition, 1 gm of Pyrex Brand Wool Filtering Fibre, or "angel hair" (Owens-Corning, Corning, NY), was placed in the chamber prior to filling with 50 ml of ethylene glycol. This use of angel hair, at 2%(w/v), to inhibit convection while determining the thermal

conductivity of liquid products is consistent with levels used by other researchers (3.5%(w/w), Baghe-Khandan and Okos, 1981; 4.4%(w/v), Choi and Okos, 1983). Conductivity measurements were taken 20 times at each temperature, and means and standard deviations at each temperature were calculated.

Accuracy is defined as "the closeness of a measured or computed value to its true value" (Sokal and Rohlf, 1973). For purposes of this study, literature values cited above will be accepted as the "true" values. Accuracy was determined as the absolute value of the literature thermal conductivity value minus the measured value and then divided by the literature value, times 100%.

Percent coefficient of variation was determined by dividing standard deviation by the mean of the 20 values, times 100%. This statistic is used to compare different populations of the same variable (Sokal and Rohlf, 1973). Degree of variability of the thermal conductivity measurements at each temperature were compared.

D. Methodology Evaluation

To substantiate the bead thermistor method, a study was run on a representative food product. Milk was chosen as the test medium because of the literature available on the properties of milk, and because, as a regulated food, milk

products are well standardized. Specific milk products chosen were skim milk, whole milk, half-and-half, and heavy cream purchased at a local grocery store.

Effects of fat content and temperature on the thermal conductivity of milk products were examined. Federal standards for fat levels in the products examined are: skim milk at 0.1 - 0.5%; whole milk at 3.3 - 3.7%; half-and-half at 10-12%; and heavy cream at 36% (Lampert, 1975). Values for the fat content of the samples were determined by the Babcock method (Richardson, 1985). Moisture content was determined using a Fisher Isotemp Vacuum Oven model 281 (Fisher Scientific, Pittsburgh, PA). The Solids-Non-Fat (SNF) content was found by difference from 100% after determining fat and moisture contents. These analyses were made on fresh product and on each replicate of each product following temperature treatment completion.

Since milk products were liquid, convection around the bead during heating was a concern. To inhibit convection, each product was placed in the test chamber with 1.5%(w/v) angel hair.

Ten measurements of thermal conductivity were made at each 10°C increment from 25 to 125°C. Data was also collected by 20°C increments as the products were cooled back down to 25°C from 125°C. At 25 to 55°C, moisture loss due to evaporation was not a concern, so the 1 and 2 kOhm

probes were mounted in the chamber by means of a rubber stopper. Because the chamber was not tightly sealed, it was submerged to a point just below its upper lip. For temperatures above 55°C, the 10 and 20 kOhm probe leads were sealed in a piece of stainless steel tubing using a silicon-based rubber sealant. This probe-tube complex was then mounted to a stainless steel cap with a Swagelok pressure fitting such that the tip of the probe extended below the cap 4.5 cm to ensure the probe would be submerged in the test material within the chamber. The cap was then clamped pressure tight onto the chamber, and the vessel was submerged in the oil bath to a point just below the pressure release valve. To switch between the 10 and 20 kOhm probes, the chamber was raised from the oil, the clamp removed, the cap replaced with another cap mounted with the appropriate probe, the clamp replaced, and the chamber resubmerged into the bath. This replacement was accomplished as quickly as possible to minimize changes in product temperature.

At each temperature, the product was given the time necessary to reach thermal equilibrium before data collection began. Due to the time required for thermal equilibration and for data collection, the full temperature treatment for each milk sample proceeded over an entire day, and, therefore, the data was analyzed on the following

day. Triplicates of each product were run with all samples for a given product coming from a single carton within the freshness date for that carton. Moisture content determinations were done immediately following the completion of each replicate. The remaining sample material for each replicate of a product was stored in a refrigerator until all three replicates were completed. Upon completion of the thermal conductivity measurements of the replicates, all three samples (after) and the fresh sample (before) were analyzed for fat content. This delay in the Babcock fat analysis had no effect on the determined fat levels (Bishop, 1988).

At this point in the study, a problem arose concerning the 10 kOhm and 20 kOhm probes used at the higher temperatures. Overpowering of the probes beyond their power dissipation capacity at a given temperature led to degradation of the bead material over time, and the ultimate failure of the probes due to a condition defined as thermal runaway (Pittfield, 1988). With the present means of control, it was not possible to fix the power level supplied to the probes within their specifications. Therefore, in an attempt to decrease power output to the probes at the higher temperatures, a resistor was added to the circuit immediately following the inverter (Figure 3). By dropping some of the power supplied by the circuit

across the inserted resistor, the voltage drop across the thermistor was reduced to less than 5 volts. Each probe at each temperature where the additional resistor was used required restandardization because, while the probe resistance and temperature step change stayed the same, the diminished power output of a probe resulted in a change in the steady-state voltage squared value obtained from the power squared-inverse square root of time plot. This in turn altered the estimates of the probe's effective radius and thermal conductivity.

E. Statistical Analysis

All statistical analyses, other than linear regression, were run using the Statistical Analysis System (SAS) (Cary, NC). Unless otherwise stated, the analyses done were Duncan tests at the 5% confidence level.

IV. Results and Discussion

A. Introduction

The results of the three phases of this study are presented. In the first phase, feasibility study, an analysis of error associated with the input parameter measurements showed the bead thermistor method to be a viable means of measuring thermal conductivity. Minimum sample diameter required to maintain the infinite boundary condition assumption was determined to be 7.2 mm.

In the methodology documentation phase, accuracy and variability of the method over the 25°C to 125°C temperature range was evaluated. Error analysis was repeated using data from 25°C and 125°C. Effects of probe differences, thermistor resistance, and standardization material were evaluated. Minimum sample diameter was reevaluated and found to be 5 mm.

In the final phase, method evaluation, the method was used to measure thermal conductivity of milk, a representative food material, as a function of fat content and temperature. Problems that arose with the thermistor probes and the control circuit are discussed. Functionality of the method is evaluated in the context of these problems.

B. Feasibility Study

Linear regression analysis of the temperature-resistance calibration data for the two thermistor beads used in this study yielded the following coefficients (and standard errors) for equation 19: for the 1.52 mm diameter 10 kOhm bead, $a_0 = 9.43E-4$ ($4.23E-5$), $a_1 = 2.27E-4$ ($8.28E-6$), $a_2 = -1.16E-7$ ($4.58E-8$); for the 2.54 mm 1 kOhm bead, $a_0 = 1.38E-3$ ($2.91E-4$), $a_1 = 2.91E-4$ ($6.62E-5$), $a_2 = 1.42E-8$ ($5.04E-8$).

Standardization of the two thermistor probes at room temperature gave the following values for their thermal conductivity and effective radius: for the 10 kOhm probe, $k_b = 0.09450$ W/m-°K, and $r = 7.87E-4$ m; for the 1 kOhm probe, $k_b = 0.04774$ W/m-°K, and $r = 1.39E-3$ m.

1. Error Analysis

To determine the feasibility of the thermistor method it was necessary to ascertain how the error in measuring input parameters affected the accuracy of test medium thermal conductivity estimation. Based on values obtained from the calibration and standardization procedures, representative values for each parameter were assumed (Table 4). Errors associated with effective radius, bead thermal conductivity, and steady-state voltage squared were assumed to be 2.5%, 2.5%, and 0.275%, respectively. Values

Table 4. Assumed values for the error-mean-square variables, their standard errors, and the value of the partial derivatives with respect to each variable

<u>Variable</u>	<u>Average value</u>	<u>Standard error</u>	<u>Partial derivative</u>
r	0.0015 m	3.75E-5 m	-282
k_b	0.05 W/m-°K	0.0012 W/m-°K	-3.98
dT	4.5 °C	0.05 °C	-0.094
V_{ss}^2	8.0 volts ²	0.022 volts ²	0.211
R	800.0 ohms	0.05 ohms	-5.28E-4

for standard error for these variables (Table 4) reflect these percentages of the assumed values for the respective variables. The standard errors presented for temperature step change and resistance were assumed based on the accuracy of the thermometer and the Keithley Multi-Meter, respectively, used to do the temperature-resistance calibration. The partial derivative of Equation 17 with respect to each input parameter was derived and evaluated using the assumed values and standard errors (Table 4). The root-mean-square error of each parameter was then calculated using the format of Equation 20.

$$dk_m = \left[\left[\frac{\delta k_m}{\delta k_b} dk_b \right]^2 + \left[\frac{\delta k_m}{\delta r} dr \right]^2 + \dots + \left[\frac{\delta k_m}{\delta v^2} dv^2 \right]^2 \right]^{1/2} \quad (20)$$

All subsequent error analysis figures were generated in the same manner. Partial derivatives were solved over a range of values for the appropriate independent variable. Ranges of partial derivative values were then used in equation 20 solved for the appropriate dependent variable. For example, in Figure 7 discussed below, each of the partial derivatives for the five input parameters were evaluated over a range of values for bead thermal conductivity. At each value of k_b the numerical value of

each partial was used in equation 20 to generate a value for the error in estimating medium thermal conductivity. Error in estimating k_m was then plotted as a function of k_b .

Error in medium thermal conductivity was plotted against bead thermal conductivity for each parameter, assuming a value for k_m of 0.6 W/m-°K (Figure 7). This figure demonstrates the relative impact of the error associated with each input parameter. The overall inaccuracy in estimating k_m , dk_m , is presented for comparison purposes. The largest sources of this overall error are those associated with effective radius and thermal conductivity, the bead parameters. The effects of the remaining parameters, in decreasing order of importance, are temperature step change, steady-state voltage squared, and final thermistor resistance. In comparison to the bead parameters, these three terms have a minimal effect on the estimation of medium thermal conductivity within the experimental range of error for all the parameters.

Another aspect of Figure 7 is that the error associated with each parameter, as well as the overall accuracy of the method, improves as bead thermal conductivity increases. This led to use of only the higher conductivity, thinner encapsulated 1.52 mm diameter probes in subsequent work.

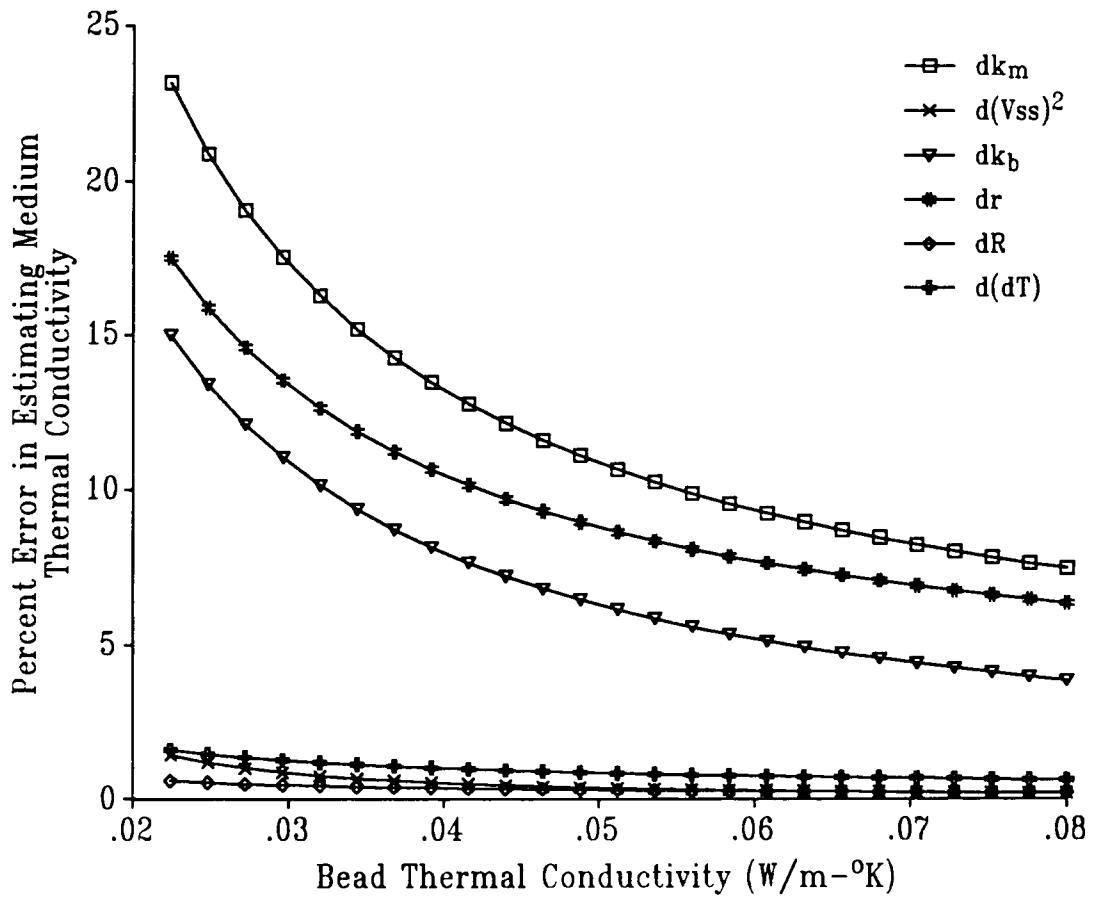


Figure 7. Error in estimation of medium thermal conductivity versus bead thermal conductivity for the 2.54 mm diameter 1 kOhm thermistor probe at 25C

Significance of the standardization is demonstrated in Figure 8 and Figure 9. Both of these figures show that, for a given medium conductivity, decreasing the error associated with the respective parameter estimate decreases the error associated with the estimate of medium thermal conductivity. Therefore, the more accurate the standardization, the more accurate the method. Another aspect of Figures 8 and 9 is that for a given error level in estimating each bead parameter, the error in estimating medium thermal conductivity increases as the medium conductivity increases.

The only other input parameter that displayed any effect on method accuracy was temperature step change (Figure 10). Maintaining a step change of 2.5°C or larger is necessary to minimize the error in estimating medium thermal conductivity. This agrees with studies by Chato (1968) that found maximum accuracy was obtained by maintaining the temperature step change between $3-7^{\circ}\text{C}$.

Figure 11 is a plot of the error in estimating medium thermal conductivity versus resistance. A similar plot for steady-state voltage squared showed the same basic trends. Error in measuring these input parameters showed no detectable effect on method accuracy.

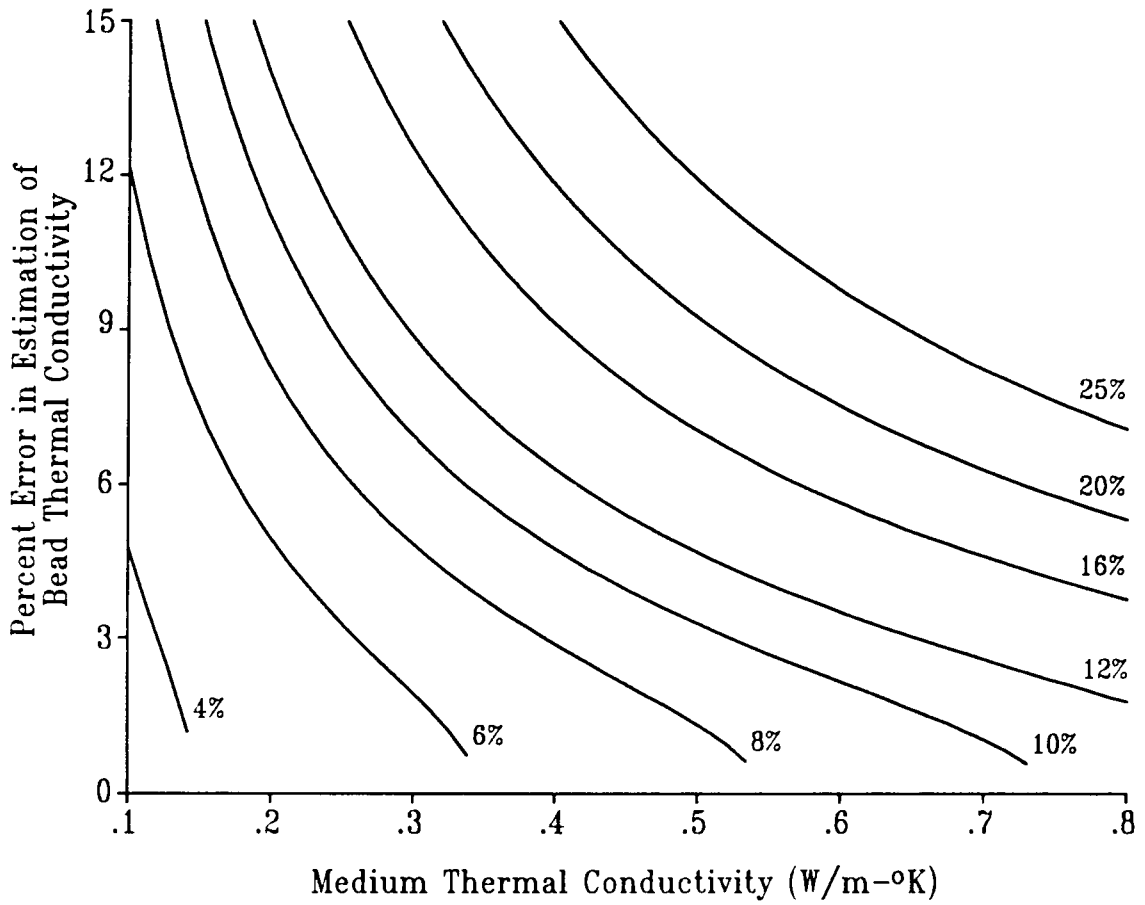


Figure 8. Percent error in estimating bead thermal conductivity versus medium thermal conductivity for varying levels of error in estimating medium thermal conductivity based on assumed values for the input parameters

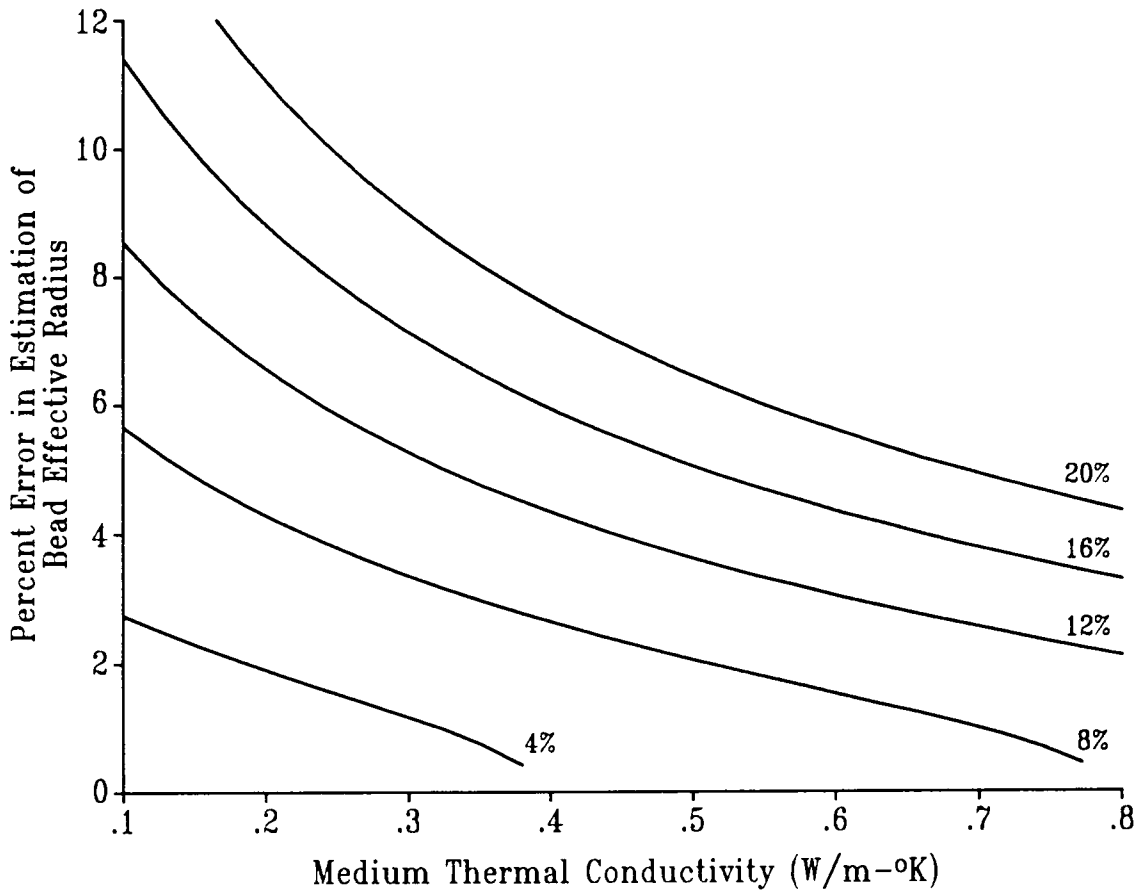


Figure 9. Percent error in estimating bead effective radius versus medium thermal conductivity for varying levels of error in estimating medium thermal conductivity based on assumed values for the input parameters

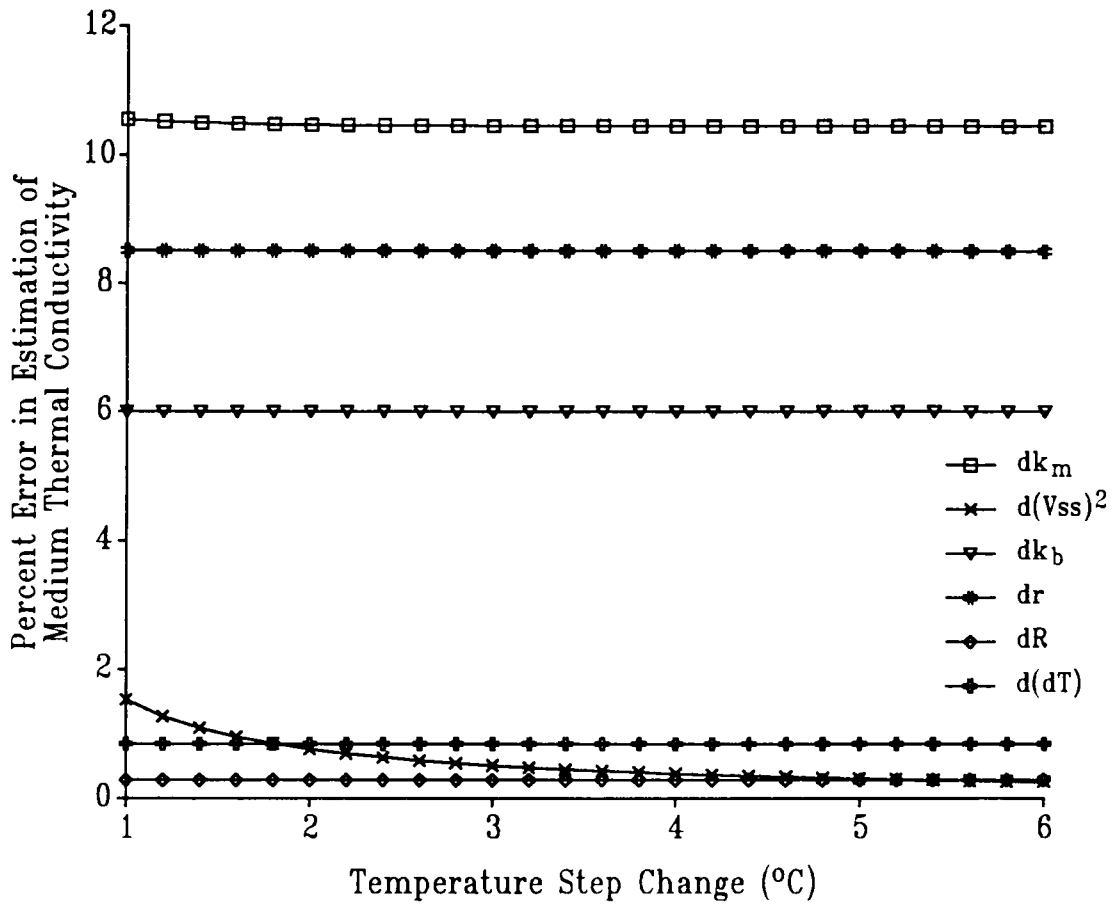


Figure 10. Percent error in estimation of medium thermal conductivity versus temperature step change for the 2.54 mm diameter 1 kOhm probe at 25C

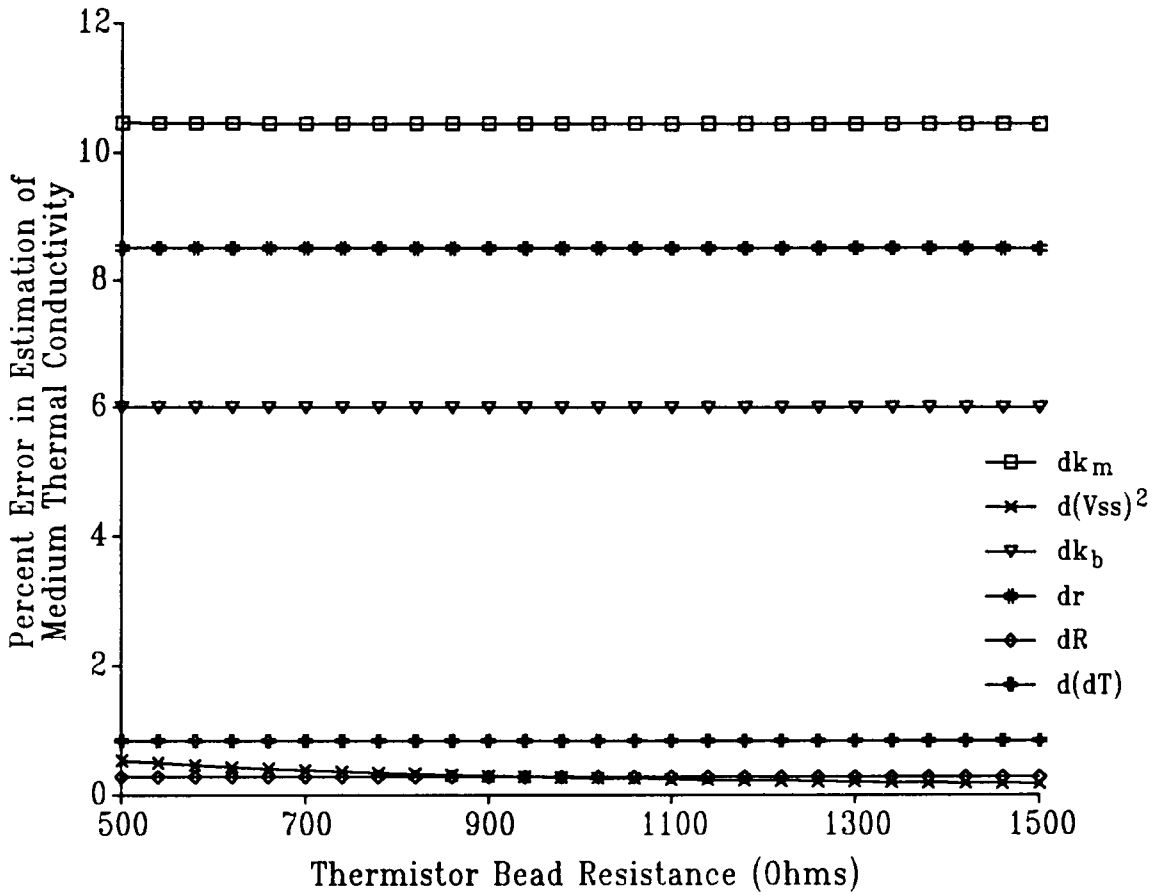


Figure 11. Percent error in estimation of medium thermal conductivity versus thermistor bead resistance for the 2.54 mm diameter 1 kOhm probe at 25C

From these results, it was concluded that the method was feasible as a means of estimating thermal conductivity of food materials. However, the following adaptations were made: 1) temperature step change needed to be maintained above 2.5°C, 2) thermal conductivity of the probes needed to be greater than 0.1 W/m-°K, i.e. probes with thinner glass encapsulations, and 3) more accurate thermal conductivity reference materials are needed to minimize standardization error associated with the bead parameters, thermal conductivity and effective radius.

2. Minimum Particle Size Determination

Results of this experiment are presented in Figure 12. Minimum diameter, for the 2.54 mm diameter 1 kOhm probe, which maintains the required infinite boundary condition assumption is 7.16 mm. Loss of this boundary condition is clearly illustrated by the large increase in the measured value of thermal conductivity of castor oil. The thermal conductivity appears to increase because the conductivity of the wood block is less than that of castor oil. This lower value for the boundary material causes an apparent increase in the conductivity of the medium being measured.

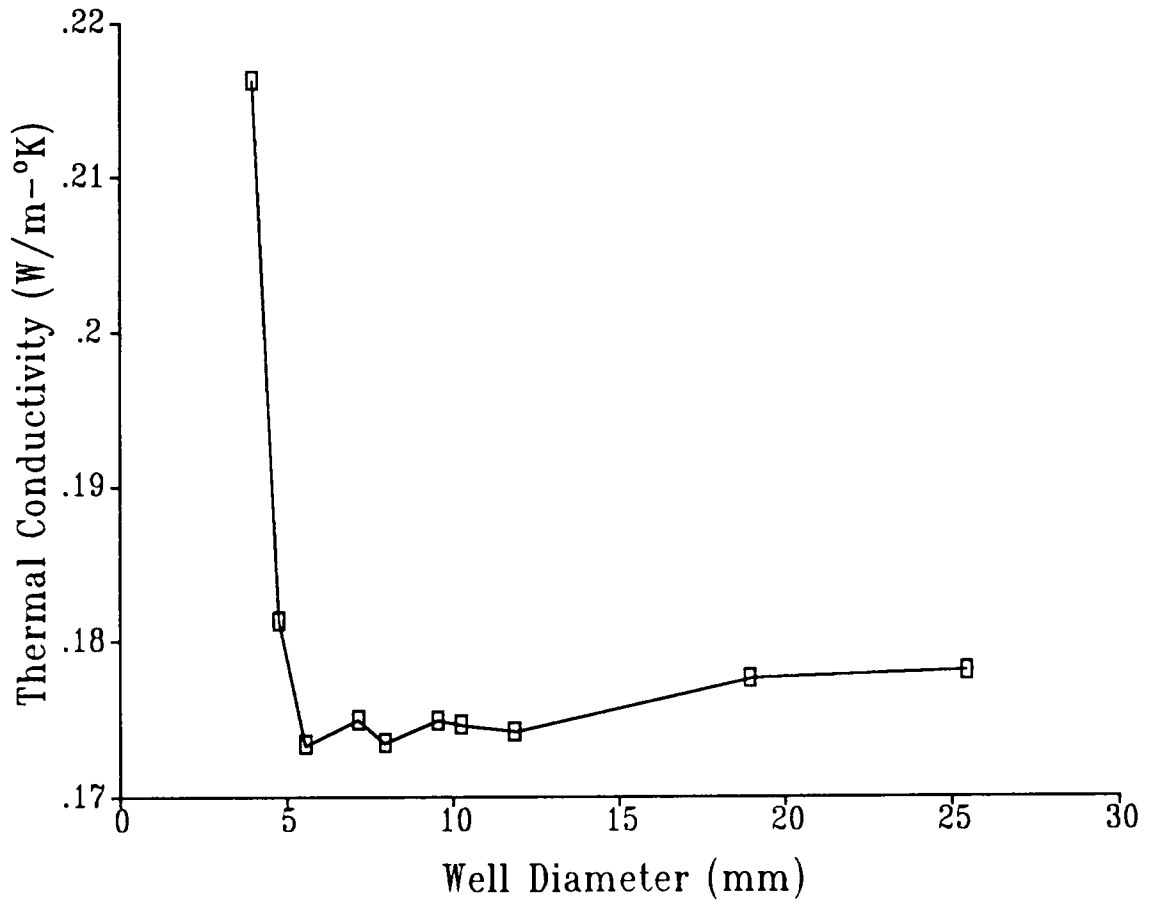


Figure 12. Measured thermal conductivity of castor oil ($0.180 \text{ W/m-}^\circ\text{K}$) versus well diameter for the 2.54 mm diameter 1 kOhm thermistor probe

C. Methodology Documentation

1. Thermistor Bead Calibration

Allowing for the fact that a given probe might be defective or may be damaged during use, more than one probe of each resistance was calibrated. Calibration coefficients and standard deviations for all probes are presented in Table 5, along with each probe's calibration range and overall fit to the calibration equation (equation 19). It was later realized that since the standard errors of most of the a_2 coefficients are greater than the coefficients themselves, the a_2 coefficients were not significant and may have been better modelled as zero. However, because this observation was made after the data had been collected, the coefficients are presented as used. Each probe was calibrated from 15°C below to 15°C above the temperature range over which it was to be used to ensure that range was accurately examined. Temperature-resistance calibration residuals are plotted against temperature for probes 10k1 and 10k2 in Figures 13 and 14, respectively. Selection of probes for use in the remainder of this study was based on which probe at each resistance had the smallest standard deviations on their calibration coefficients.

Table 5. Calibration coefficients and standard errors for all thermistor probes

probe	range (°C)	coeff- icient	value	standard error	r ²	
1 kOhm	1	10-50	a ₀	1.30E-3	6.30E-5	0.9998
			a ₁	2.96E-4	1.38E-5	
			a ₂	-7.54E-8	9.59E-8	
	2		a ₀	1.42E-3	6.35E-5	0.9998
			a ₁	2.81E-4	1.43E-5	
			a ₂	4.60E-8	1.06E-7	
	3		a ₀	1.39E-3	7.37E-5	0.9998
			a ₁	2.80E-4	1.64E-5	
			a ₂	6.84E-8	1.18E-7	
2 kOhm	1	30-70	a ₀	1.28E-3	2.79E-5	0.9999
			a ₁	2.66E-3	6.16E-6	
			a ₂	1.08E-7	4.35E-8	
	2		a ₀	3.42E-3	1.64E-4	0.9997
			a ₁	-1.56E-3	6.76E-5	
			a ₂	6.13E-5	1.64E-6	
	3		a ₀	1.34E-3	6.11E-5	0.9988
			a ₁	2.52E-4	1.34E-5	
			a ₂	2.03E-7	9.46E-8	
10 kOhm	1	50-110	a ₀	1.05E-3	3.33E-5	0.9999
			a ₁	2.51E-4	6.96E-6	
			a ₂	3.56E-9	4.40E-8	
	2		a ₀	1.06E-3	3.65E-5	0.9998
			a ₁	2.47E-4	7.58E-6	
			a ₂	2.99E-8	4.75E-8	
20 kOhm	1	90-140	a ₀	1.00E-3	5.20E-5	0.9998
			a ₁	2.29E-4	1.14E-5	
			a ₂	1.31E-7	7.92E-8	
	2		a ₀	9.47E-4	5.72E-5	0.9997
			a ₁	2.42E-4	1.25E-5	
			a ₂	3.67E-8	8.75E-8	
	3		a ₀	1.00E-3	6.15E-5	0.9997
			a ₁	2.30E-4	1.35E-5	
			a ₂	1.24E-7	9.43E-8	
	4		a ₀	8.92E-4	7.88E-5	0.9995
			a ₁	2.49E-4	1.70E-5	
			a ₂	-1.96E-8	1.15E-7	

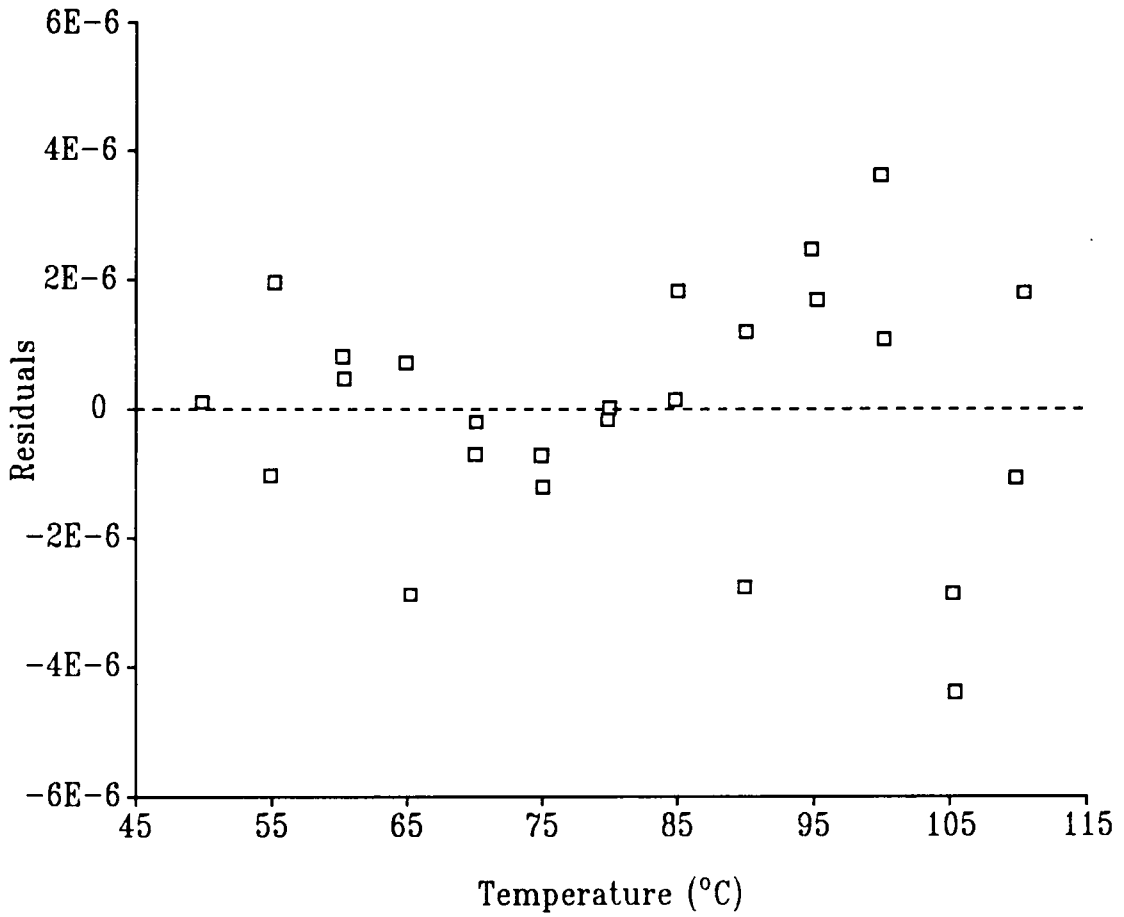


Figure 13. Residuals of temperature-resistance calibration equation versus temperature for probe 10k1

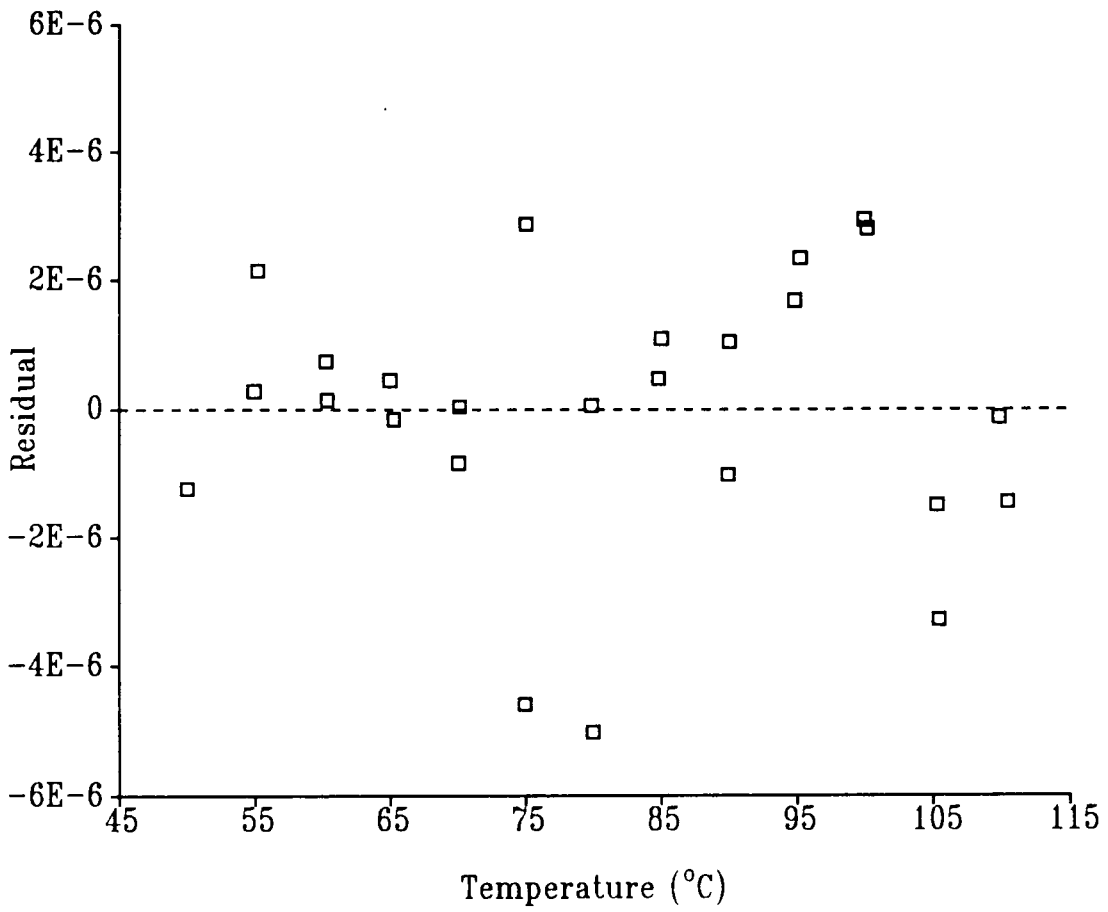


Figure 14. Residuals of temperature-resistance calibration equation versus temperature for probe 10k2

2. Thermistor Bead Probe Standardization

Standardization values for effective radius as a function of temperature are presented in Table 6. Values for each combination of the three reference materials is given along with the average of the three values. Effective radius of the thermistor beads appears to independent of temperature. There is no statistically significant difference between the mean values of effective radius across temperature with the exception of the radii at 95°C and 105°C which were different from the others but not from each other.

All the probes were manufactured to the same physical specification, a diameter of $1.52\text{E-}3$ m, or 0.06 in.. Actual radius is, in all cases, larger than effective radius due to the thickness of the glass encapsulation. Effective radius is more an estimation of thermistor bead diameter than of probe diameter.

Standardization values for bead thermal conductivity as a function of temperature are presented in Table 7. Values for each combination of the three reference materials are given along with the average of the three values.

Thermal conductivity is a temperature dependent property. However, while there are fluctuations in the bead thermal conductivity data, the values do not show any

Table 6. Average values of effective radius for glycerin-water (GW), glycerin-castor oil (GC), water-castor oil (WC), and glycerin-water-castor oil (GWC). All values are $\times 10^{-4}$ m

Probe	Temperature	GW	GC	WC	GWC
1k1	25	7.68	8.66	8.18	8.17
	35	7.87	8.30	8.08	8.08
2k1	45	8.63	9.34	8.99	8.99
	55	8.66	9.91	9.29	9.29
10k1	65	8.33	9.04	8.70	8.69
	75	11.0	8.28	9.35	9.56
	85	6.36	10.8	8.20	8.44
	95	8.48	5.69	6.67	6.95
20k1	105	6.43	6.10	6.24	6.26
	115	9.00	8.79	8.88	8.89
	125	8.48	7.93	8.16	8.19

Table 7. Average values of thermal conductivity for glycerin-water (GW), glycerin-castor oil (GC), water-castor oil (WC), and glycerin-water-castor oil (GWC).

Probe	Temperature	GW	GC	WC	GWC
1k1	25	0.122	0.087	0.108	0.106
	35	0.111	0.095	0.105	0.104
2k1	45	0.114	0.092	0.106	0.104
	55	0.116	0.081	0.102	0.100
10k1	65	0.082	0.068	0.077	0.076
	75	0.044	0.079	0.055	0.059
	85	0.106	0.036	0.070	0.071
	95	0.049	0.123	0.069	0.080
20k1	105	0.120	0.142	0.127	0.130
	115	0.124	0.133	0.127	0.128
	125	0.203	0.284	0.224	0.237

significant trend as a function of temperature. This is probably due to a combination of factors. Thermal conductivity of glasses and ceramics show, in general, a small temperature dependence over the relatively narrow examined 100°C temperature range (CRC, 1971). Another important factor is that different resistance beads have slightly different compositions responsible for those resistances. This varying composition may counter any temperature effects that would be more noticeable had one probe been used over the entire range. Finally, there is the accuracy of the bead thermal conductivity values themselves which will account for some the exhibited variability.

3. Error Analysis

After completing probe standardizations, error analysis on the effects of input parameters (Section IV.B.1) was repeated using collected data from 25°C and 125°C (Table 8). Average values and standard errors for temperature step change, steady-state voltage squared, and resistance are of ten measurements made in the water standard at a respective temperature. Values for effective radius and bead thermal conductivity and their standard errors are the determined standardization values at each

Table 8. Average values for the error-mean-square variables, their standard errors, and the value of the partial derivatives with respect to each variable at 25°C and 125°C

<u>Variable</u>	<u>Average value</u>	<u>Standard error</u>	<u>Partial derivative</u>
at 25°C			
r	8.17E-4 m	0.404E-4 m	-1692
k _b	0.106 W/m-°K	0.014 W/m-°K	-7.08
dT	3.48 °C	0.040 °C	-0.397
V _{ss} ²	10.3 volts ²	0.084 volts ²	0.254
R	997.3 ohms	1.40 ohms	-1.39E-4
at 125°C			
r	8.19E-4 m	0.228E-4 m	-1256
k _b	0.237 W/m-°K	0.0344 W/m-°K	-1.55
dT	2.54 °C	0.035 °C	-0.405
V _{ss} ²	6.53 volts ²	0.034 volts ²	0.133
R	588.6 ohms	0.561 ohms	-1.75E-3

temperature.

Plots of error in estimating medium thermal conductivity as a function of bead conductivity for each input parameter are given in Figure 15 for 25°C and Figure 16 for 125°C. It is readily apparent from both figures that standardization error (the error in estimating effective radius and bead thermal conductivity) is more critical than was assumed based on the feasibility study results. It was also noted that method accuracy improves as bead thermal conductivity increases. The remaining three parameters were examined as before and found to have no detectable effect on method accuracy.

Error associated with effective radius versus medium thermal conductivity at varying levels of error in estimating medium conductivity is presented in Figures 17 and 18 for 25°C and 125°C, respectively. Results in both cases are the same as in the feasibility study. The more accurately effective radius is determined, the more accurate the method. However, it is important to note that at 125°C, method accuracy is less sensitive to effective radius error than at 25°C. At a thermal conductivity of 0.6 W/m-°K and at an error level of 2% for effective radius, error in estimating medium conductivity is 16% at 25°C but only 8% at 125°C. The method appears more accurate at the higher temperature because, for a given error level in estimating effective radius, the error in

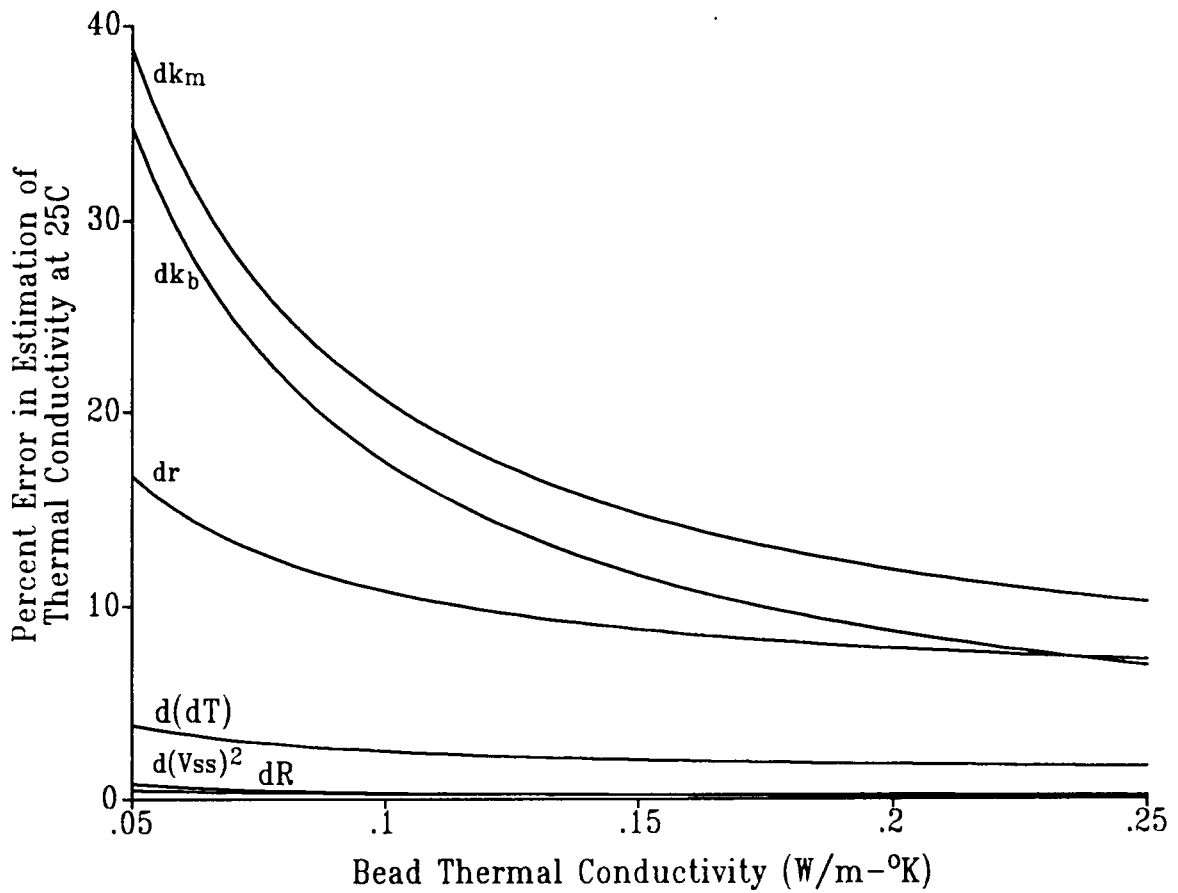


Figure 15. Error in estimating medium thermal conductivity versus bead thermal conductivity for the error associated with each parameter at 25C

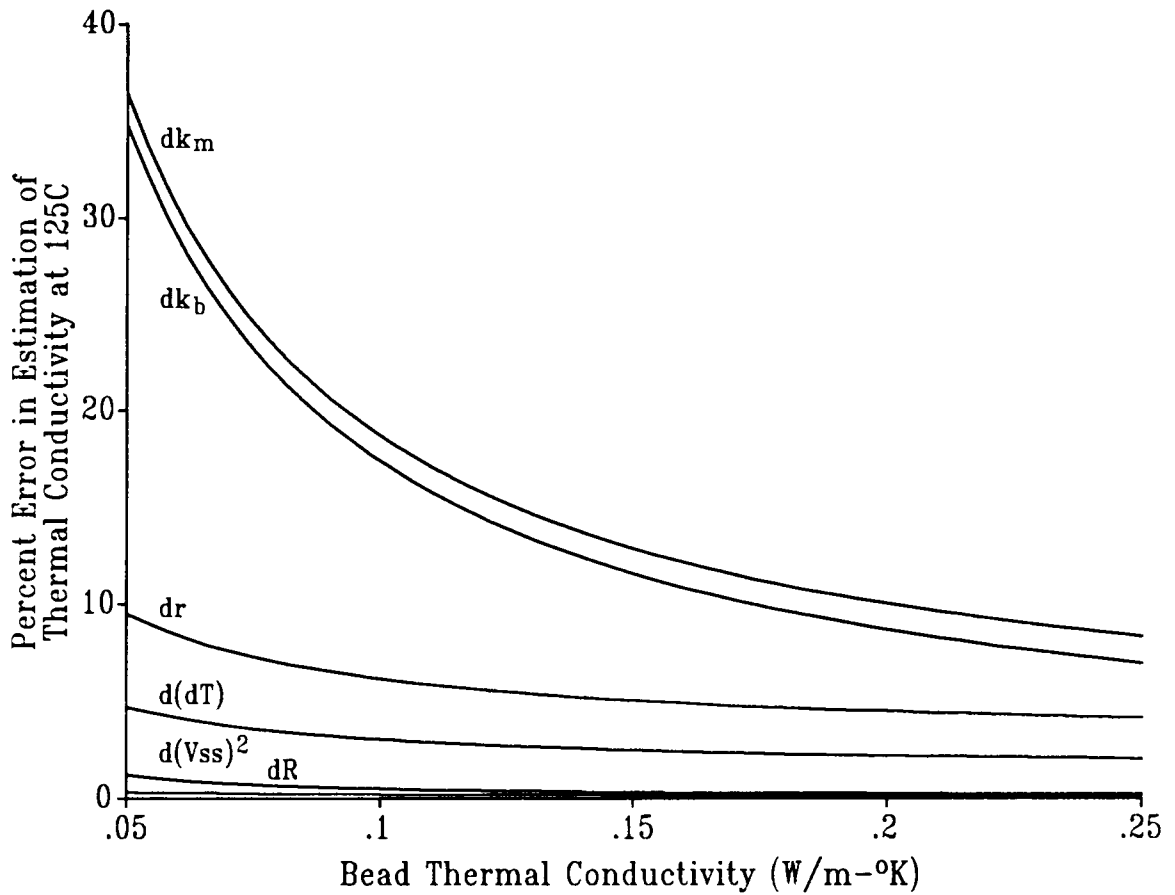


Figure 16. Error in estimating medium thermal conductivity versus bead thermal conductivity for the error associated with each parameter at 125C

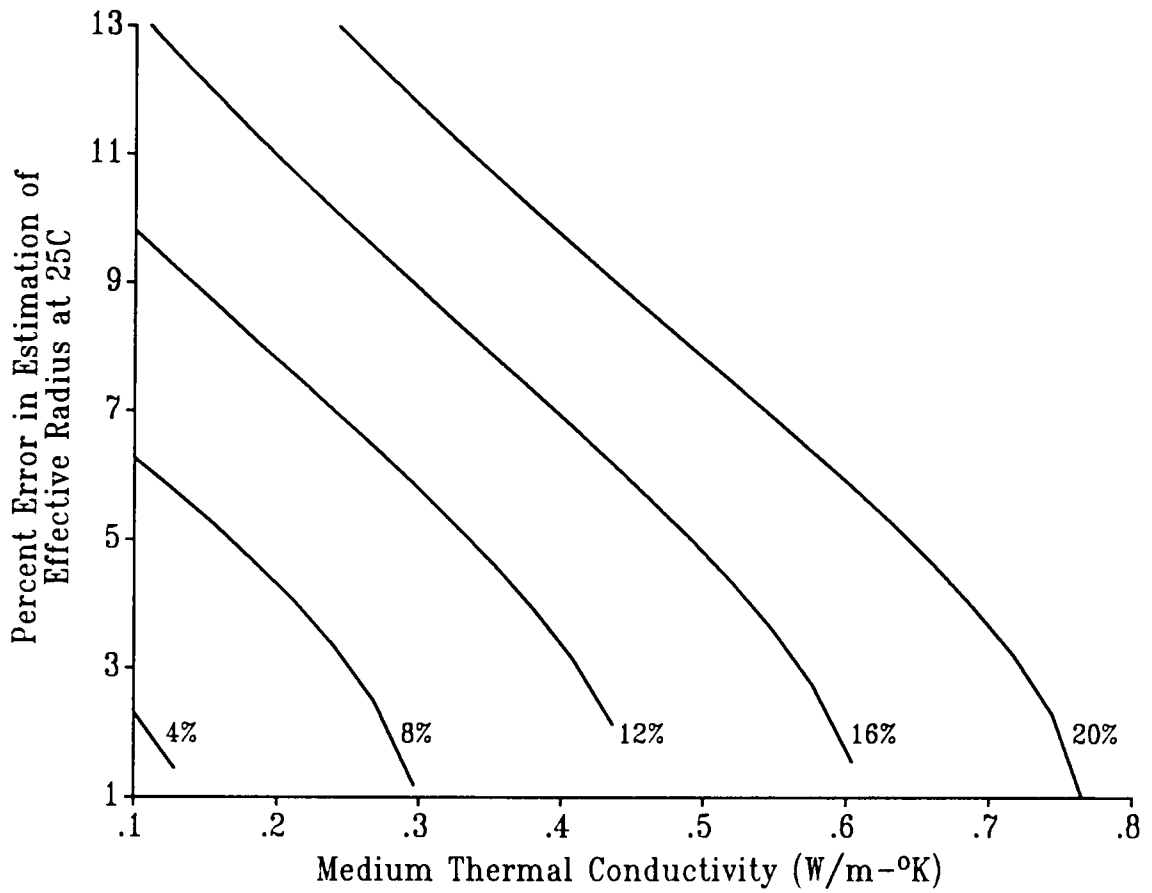


Figure 17. Percent error in estimating effective radius versus medium thermal conductivity for different levels of error in estimating k_m at 25C

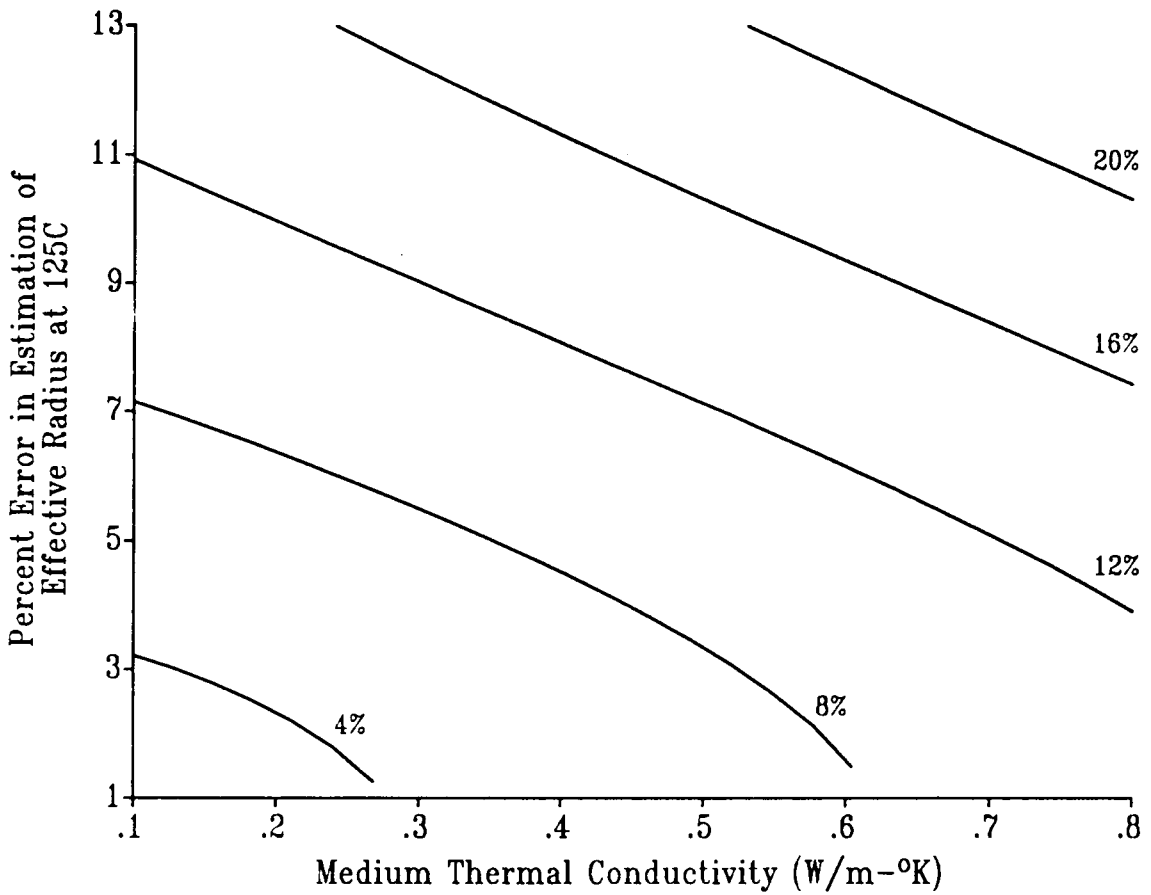


Figure 18. Percent error in estimating effective radius versus medium thermal conductivity for different levels of error in estimating k_m at 125C

estimating medium thermal conductivity at 125°C is less than the error in estimating medium thermal conductivity at 25°C.

The same result holds true for the error in estimating bead thermal conductivity versus medium thermal conductivity data presented in Figures 19 and 20 for 25°C and 125°C, respectively. Again, the more accurate the standardization used to determine bead conductivity, the more accurate the method. And, as with the effective radius data, the method appears more accurate at high temperature. For a medium thermal conductivity of 0.6 W/m-°K and an error in estimating bead conductivity of 5%, the error in estimating medium conductivity is 12% at 25°C but only 5% at 125°C.

4. Minimum Particle Size Determination

Minimum particle size necessary to maintain the infinite boundary condition assumption for heat transfer was determined for the 1 kOhm thermistor probe number 1kl. Results of this experiment are presented in Figure 21. Accounting for the accuracy of the method at 25°C, wells down to 5 mm could be tested before the measured thermal conductivity of the glycerin varied from the literature value. Using the thinner 1.52 mm diameter probe gave a reduction of 30% in the size of the smallest measurable

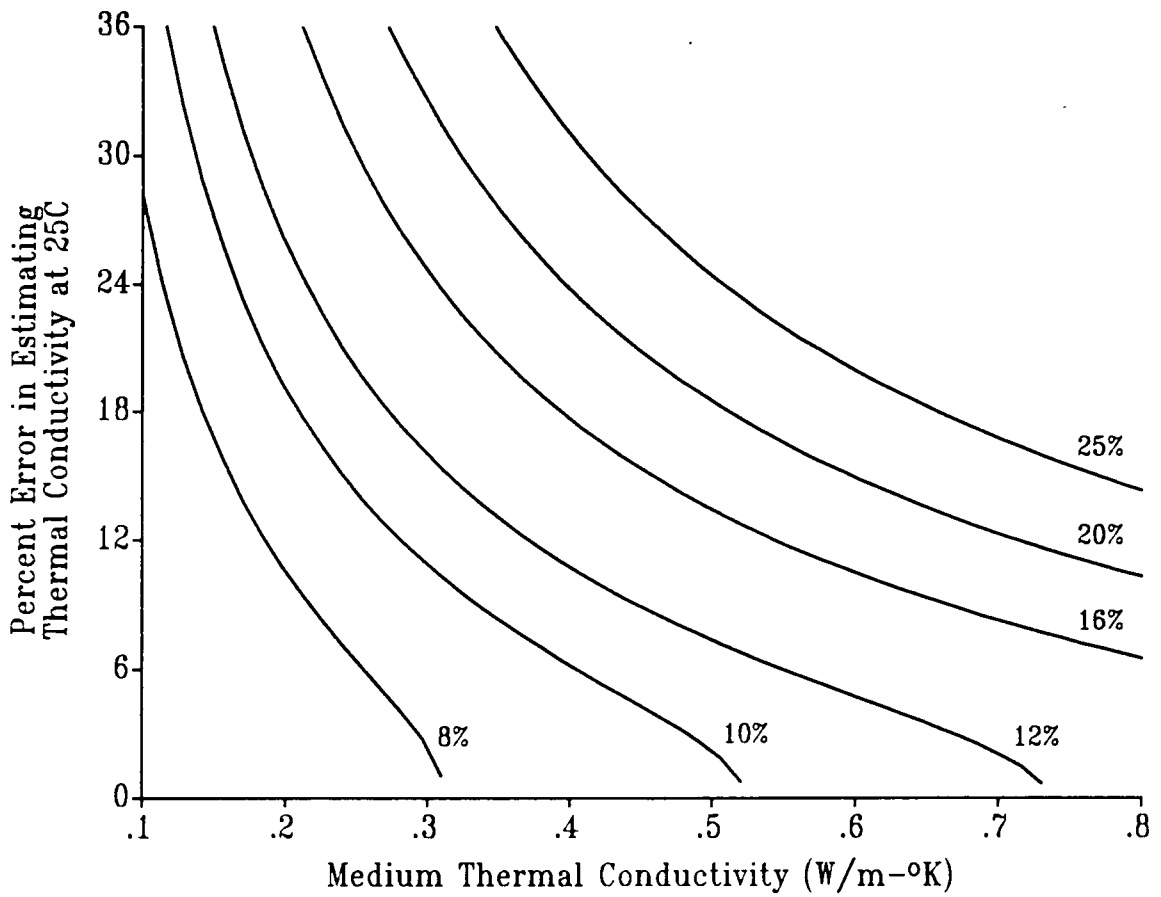


Figure 19. Percent error in estimating bead thermal conductivity versus medium thermal conductivity for different levels of error in estimating k_m at 25C

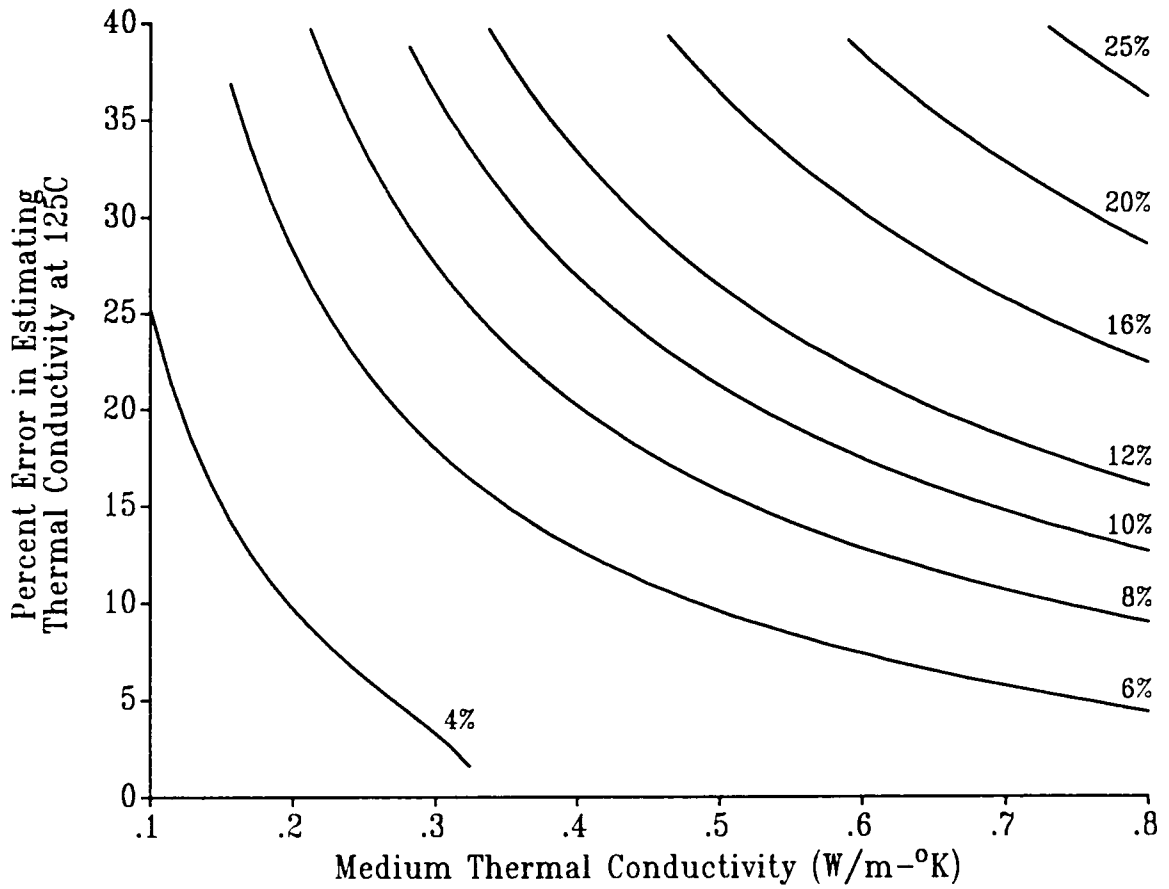


Figure 20. Percent error in estimating bead thermal conductivity versus medium thermal conductivity for different levels of error in estimating k_m at 125C

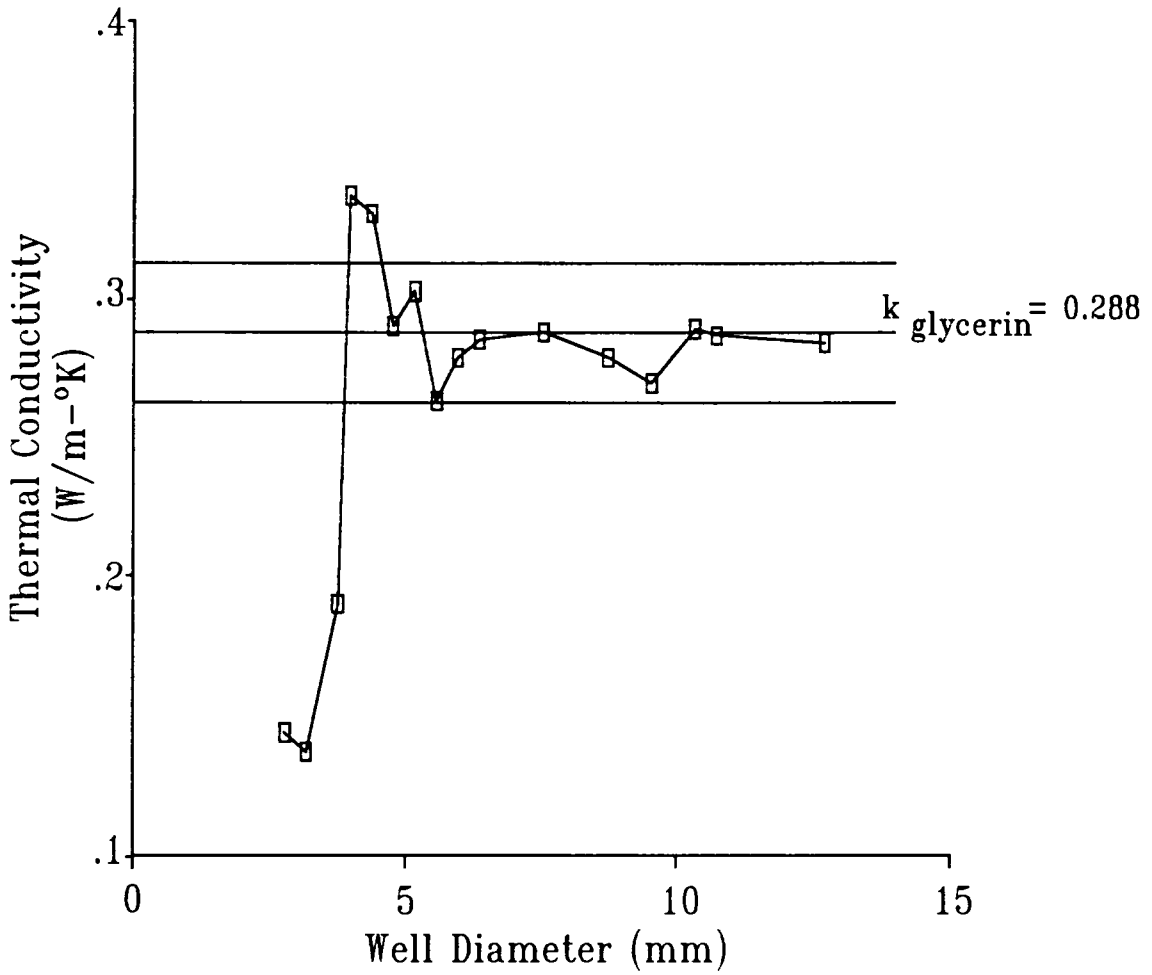


Figure 21. Minimum particle size determination showing $\pm 6.0\%$ band of accuracy about the thermal conductivity of glycerin

particulate from the thicker 2.54 mm diameter probe. A diameter of 5 mm is smaller than the 7.1 mm diameter of a size 1 canned pea, and, therefore, small enough to determine the thermal conductivity of almost any food particulate that might be thermal processed.

In the earlier minimum particle size determination experiment, thermal conductivity of the wood block was less than that of the test medium, and loss of the boundary condition was expressed as an apparent increase in medium thermal conductivity. In this case, the block conductivity was greater than the test medium, and so the loss of the boundary condition manifested as an apparent decrease in medium conductivity.

5. Accuracy and Coefficient of Variation Determination

Table 9 summarizes the data associated with this aspect of the method development. Measured thermal conductivity values for ethylene glycol are presented with literature values, and the calculated accuracy and percent coefficient of variation (%COV) for each temperature.

Accuracy of the method over the full temperature range was very encouraging. The high level of inaccuracy at 75-95°C was a concern, and is discussed below.

Table 9. Accuracy and percent coefficient of variation with temperature for ethylene glycol thermal conductivity measurements

Probe	Temp. (°C)	k_m (W/m-°K)	$k_{literature}$ (W/m-°K)	accuracy (%)	%COV
1k1	25	0.273	0.257	6.00	0.516
	35	0.275	0.259	6.31	0.502
2k1	45	0.281	0.260	7.88	0.741
	55	0.270	0.261	3.09	0.584
10k1	65	0.255	0.263	3.06	5.14
	75	0.231	0.264	12.6	7.12
	85	0.225	0.265	15.4	2.64
	95	0.139	0.267	47.8	4.29
20k1	105	0.254	0.268	5.20	5.14
	115	0.252	0.270	6.48	4.85
	125	0.276	0.271	1.77	1.32

The conductivity estimates at 25-55°C were greater than the literature values, while the remaining measured values, with the exception of 125°C, were below the accepted values. The worst accuracy occurred at temperatures where the bead thermal conductivity of probe 10k1 was at its lowest. This substantiates the error analysis that showed accuracy decreased with an decrease in bead conductivity. Also, the best accuracy was achieved with the bead that has the highest thermal conductivity, probe 20k1 at 125°C.

The percent coefficient of variation data showed the four lowest temperatures had the smallest %COV, or the best reproducibility of measurement. At 65°C and above, probes tended to show more susceptibility to fluctuations in the oil bath temperature. This is may be because as bath temperature increased, oil viscosity decreased, the rate of heat transfer to the chamber increased, and probes began detecting the normal temperature cycling as the bath tried to maintain a constant temperature.

Overall, the accuracy and reproducibility of the method across the full 100°C temperature range appeared excellent. However, there was still the problem of the accuracy at over the 75-95°C range measured by probe 10k1. As a means of verifying the 75-95°C results, a second 10 kOhm probe, probe 10k2 was standardized over the

standardization temperature range of 65-95°C. Effective radius and bead thermal conductivity at each temperature for probe 10k2 are shown in Table 10. Effective radii were in the same range as the values for the other probes. Bead thermal conductivity values for probe 10k2 at 65°C, 75°C, and 95°C were 37%, 93%, and 16% higher than 10k1, respectively, but 4% lower at 85°C. However, when probe 10k2 was used to measure the thermal conductivity of ethylene glycol, the %COV values showed an average improvement of 13%, while the accuracy at 75°C, 85°C, and 95°C improved by 57%, 63%, and 37%, respectively (Table 11). Although probe 10k1 had smaller standard deviations on its calibration coefficients, probe 10k2 was a more accurate probe. This indicated that to obtain the most accurate measurements of medium thermal conductivity, several probes should be evaluated for both accuracy and reproducibility to determine the best choice for taking the measurements.

In spite of the increased accuracy with probe 10k2, an accuracy of 30% showed room for further improvement of the method at 95°C. Probe resistance at 95°C was 733 Ohms. For a 10 kOhm probe, this resistance falls in the bottom, flatter portion of the temperature-resistance curve. This decrease in probe sensitivity may be responsible for the inaccuracy of the method. Therefore, the 20 kOhm probe,

Table 10. Effective radius ($\times 10^{-4}m$) and thermal conductivity for probe 10k2 at 65-95°C

Parameter	Temperature	GW	GC	WC	GWC
effective radius	65	7.06	8.26	7.66	7.66
	75	6.00	8.27	7.05	7.11
	85	6.94	9.12	7.98	8.02
	95	6.15	4.98	5.44	5.53
thermal conductivity	65	0.124	0.081	0.107	0.104
	75	0.162	0.067	0.116	0.115
	85	0.086	0.048	0.069	0.068
	95	0.071	0.122	0.086	0.093

Table 11. Accuracy and percent coefficient of variation with temperature for probe 10k2

Temperature (°C)	k_m (W/m-°K)	$k_{literature}$ (W/m-°K)	accuracy (%)	%COV
65	0.276	0.263	4.96	1.75
75	0.250	0.264	5.40	3.22
85	0.250	0.265	5.69	4.81
95	0.187	0.267	30.0	3.66

20kl, was standardized and tested at 95°C where its resistance was 1350 Ohms and still in the steeper, more sensitive part of its response curve. As can be seen in the results of this examination (Table 12), the %COV improved by 21%, and there was a ten-fold improvement in accuracy. It became apparent that closer examination of the temperature-resistance profiles of the different resistance probes was necessary to insure that the proper probe was chosen for each temperature.

To investigate this theory, temperature-resistance data collected for probe 10kl was examined to determine how the error in measuring resistance affected the temperature estimate. This probe had been calibrated from 50°C to 110°C, and was, therefore, examined at the corresponding resistance extremes of 3300 and 490 ohms, respectively. Mean square error (MSE) was to be used to calculate the 95% confidence interval about the resistance value. However, the MSE was 9.44E-11, and the confidence interval of 3.77E-6 was too small to produce a significant range about a resistance value. Therefore, a worst case range of ±1% was used around the extreme resistance values. These results are summarized in Table 13. The slope decreased 89% going from 3300 to 490 ohms. However, average percent errors in the temperatures for the ±1% error in resistance were 0.525% at 3300 ohms and 0.336% at 490 ohms. It appears the

Table 12. Effective radius, bead thermal conductivity, accuracy, and percent coefficient of variation for probe 20k1 at 95°C

quantity	GW	GC	WC	GWC
effective radius	6.32	7.25	6.80	6.79
thermal conductivity	0.141	0.094	0.122	0.119
accuracy ($k_m=0.257 \text{ W/m-}^\circ\text{K}$)	--	--	--	3.58
%COV	--	--	--	2.88

Table 13. Resistance, temperature, and slope of the temperature-resistance curve of probe 10k1 at $\pm 1\%$ error in measuring resistance

resistance (ohms)	temperature ($^{\circ}\text{C}$)	error in temp (%)	slope (ohms/ $^{\circ}\text{C}$)
3267	50.48	0.528	-123.8
3300	50.22	---	-125.2
3333	49.96	0.522	-126.7
485.1	110.1	0.338	-13.12
490.0	109.7	---	-13.28
494.9	109.3	0.334	-13.44

loss of 10k probe sensitivity by itself is insufficient to account for the decrease in accuracy of the method at higher temperatures in the 65-95°C range. However, the full 25-125°C temperature range may need to be subdivided into more and perhaps different subranges than the four divisions used in this study to best optimize the sensitivity of the method itself. The accuracy and %COV data for the final probe and temperature range selections are shown in Table 14. The measured ethylene glycol thermal conductivity values for the initial and final probe selections are plotted with the literature values as a function of temperature in Figure 22.

During the evaluation of the above data, it was noted that Valvano (1981) used only water and glycerin as standard reference materials for probe standardization. From this observation arose the question of why is the three-point calibration "better" than any of the three possible two-point calibrations. To investigate this hypothesis, the bead parameters for each combination of two reference materials were used to recalculate the thermal conductivity of ethylene glycol. Using probe 10k2 for 65-85°C and probe 20k1 for 95°C, the accuracy of the method for each combination was tested for any statistically significant differences between combinations for each temperature. These results are summarized in Table 15. Considered across the full temperature range, the best

Table 14. Accuracy and percent coefficient of variation with temperature for ethylene glycol thermal conductivity measurements for final probe and temperature range selections

Probe	Temp. (°C)	k_m (W/m-°K)	$k_{literature}$ (W/m-°K)	accuracy (%)	%COV
1k1	25	0.273	0.257	6.00	0.516
	35	0.275	0.259	6.31	0.502
2k1	45	0.281	0.260	7.88	0.741
	55	0.270	0.261	3.09	0.584
10k2	65	0.276	0.263	4.96	1.75
	75	0.250	0.264	5.39	3.22
	85	0.250	0.265	5.69	4.81
20k1	95	0.257	0.267	3.58	4.29
	105	0.254	0.268	5.20	5.14
	115	0.252	0.270	6.48	4.85
	125	0.276	0.271	1.77	1.32

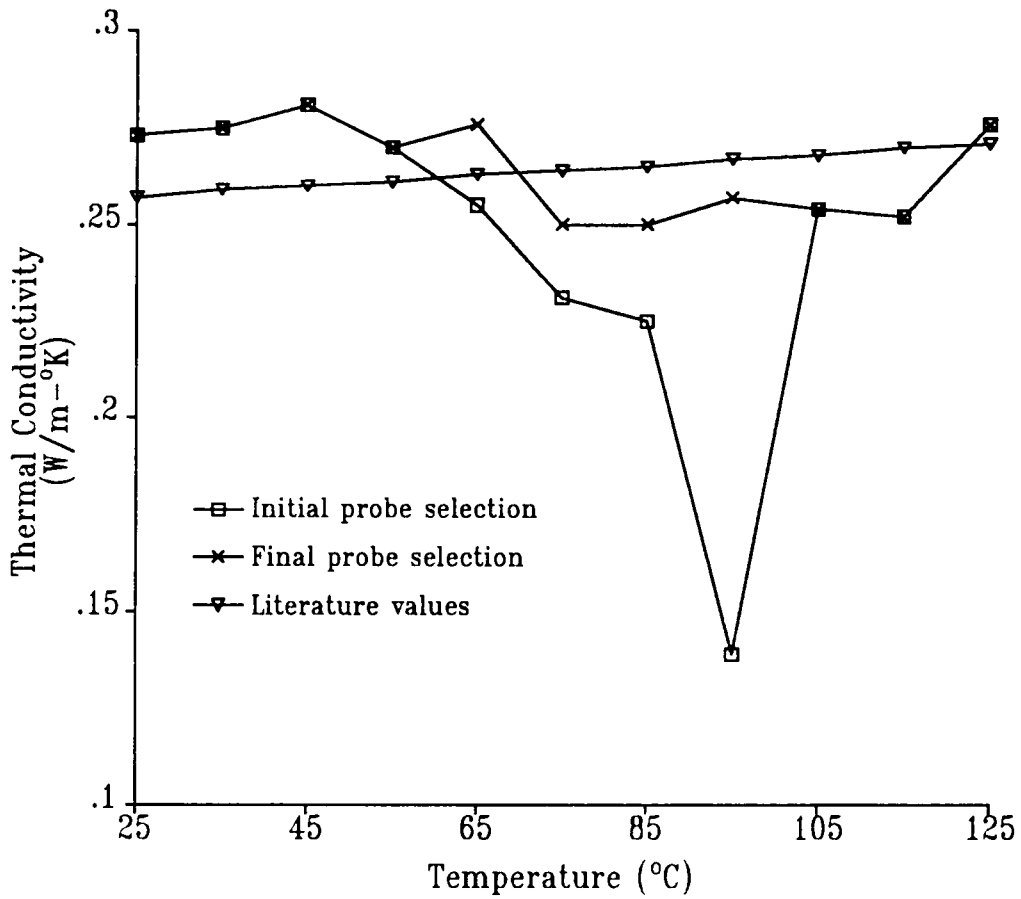


Figure 22. Thermal conductivity of ethylene glycol for the initial and final probe selections and literature values as a function of temperature.

Table 15. Percent accuracy as a function of the combination of standard reference materials at each temperature

temperature	GW	GC	WC	GWC
25	8.50 ^A	8.02 ^B	4.80 ^D	6.00 ^C
35	7.14 ^A	6.94 ^A	5.50 ^C	6.31 ^B
45	9.23 ^A	9.10 ^A	6.96 ^C	7.88 ^B
55	6.00 ^A	5.32 ^B	1.81 ^D	3.09 ^C
65	8.60 ^A	8.20 ^A	3.61 ^C	4.96 ^B
75	4.85 ^B	2.77 ^C	4.85 ^B	5.39 ^A
85	15.1 ^A	16.6 ^A	6.34 ^B	5.69 ^C
95	0.417 ^A	1.88 ^A	4.82 ^B	3.58 ^B
105	5.85 ^A	5.12 ^A	4.06 ^A	5.20 ^A
115	6.82 ^A	6.48 ^A	6.03 ^A	6.48 ^A
125	1.26 ^B	1.84 ^B	3.66 ^A	1.77 ^B

^{A, B, C, D} indicate significant differences at the 0.05 level between values for a given temperature

overall accuracy was achieved with a two-point water-castor oil standardization. If any two-point combination were to be found more accurate than the full three-point, it was expected that the glycerin-water pair would prove best. This is due to the fact that both water and glycerin are chemically pure molecules obtained in a purified state. Castor oil, however, is a mixture of fatty acids approximately 87% ricinoleic, 7% oleic, 3% linoleic, 2% palmitic, 1% stearic, and trace amounts of dihydroxystearic (Merck, 1983). Exact composition may vary due to variety, geographic location, or season of cold-pressing the seeds to extract the oil. It is this variability of composition that made castor oil a tentative choice as a standard reference material. It was, therefore, surprising to find that water-castor oil yielded the best results. The glycerin-castor oil pair was evaluated, but this combination would not be practical because the narrow range of thermal conductivities encompassed by them excludes almost all high moisture and liquid food products.

D. Method Evaluation

Results of the thermal conductivity measurements for skim milk, whole milk, half-and-half, and cream are presented in Figures 23, 24, 25, and 26, respectively. Data used to generate these figures is presented in

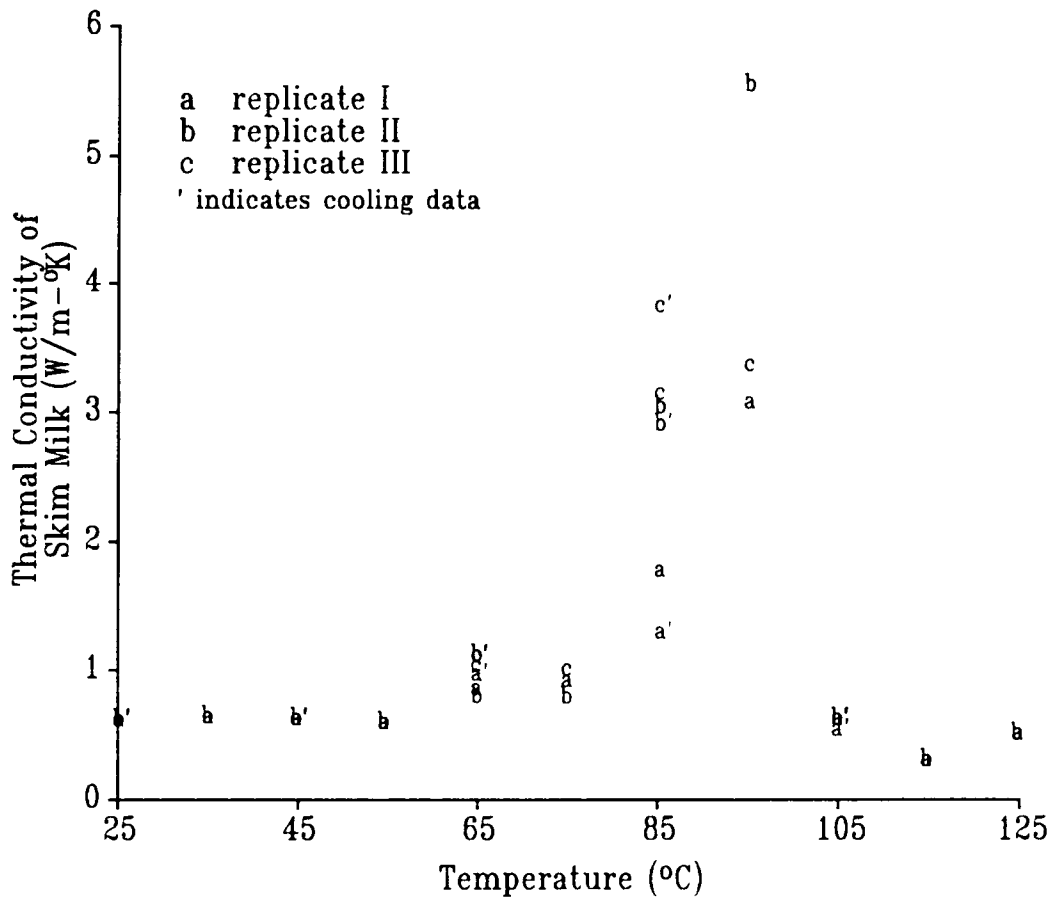


Figure 23. Measured thermal conductivity of skim milk for all three replicates.

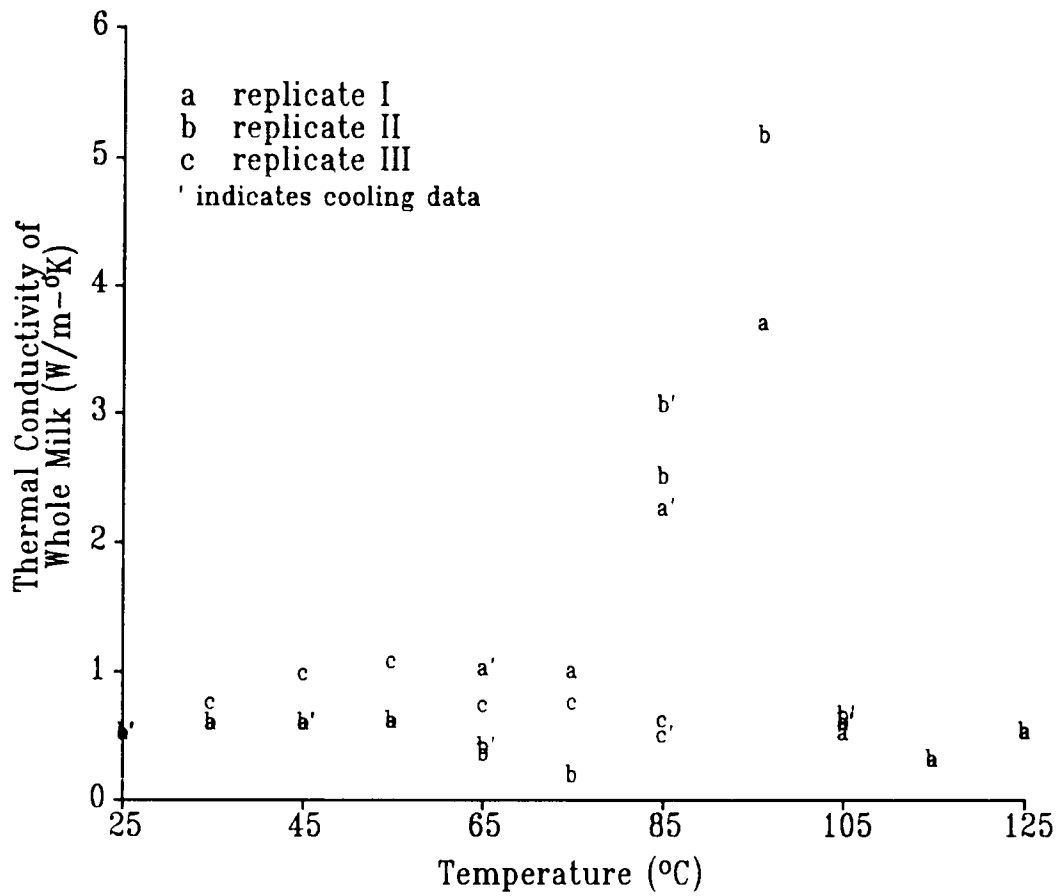


Figure 24. Measured thermal conductivity of whole milk for all three replicates.

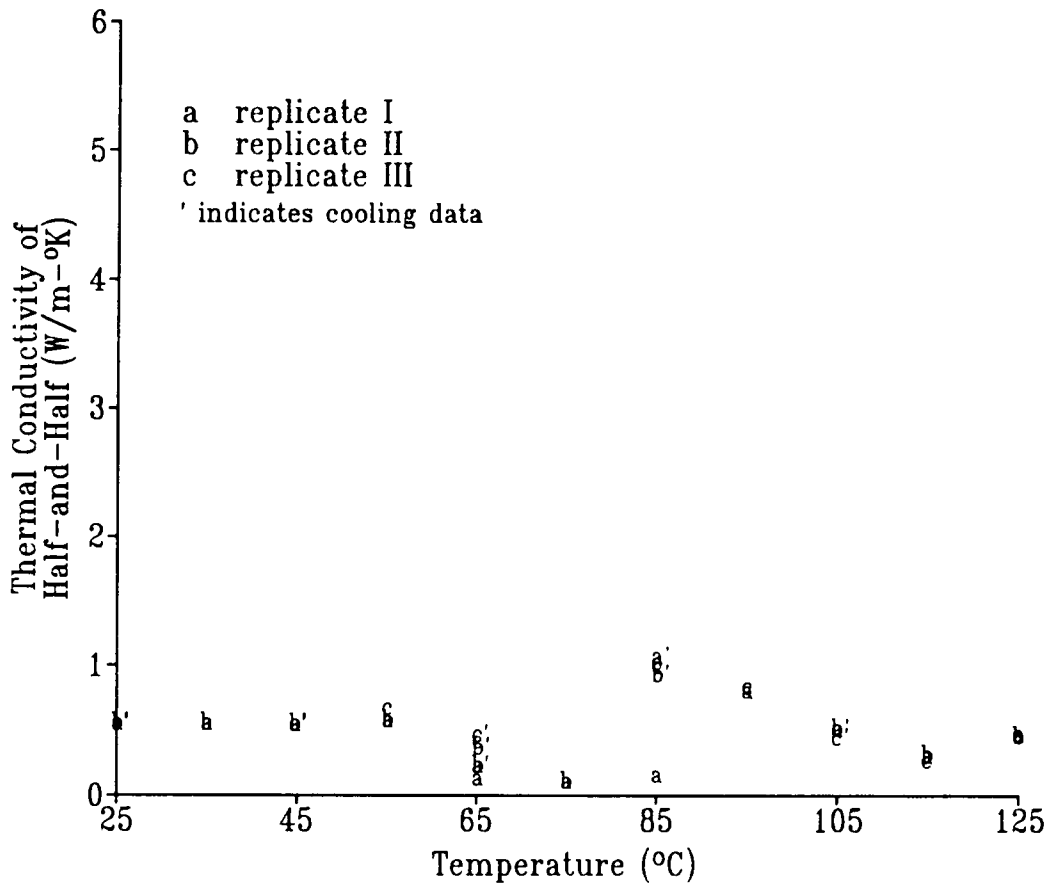


Figure 25. Measured thermal conductivity of half-and-half for all three replicates.

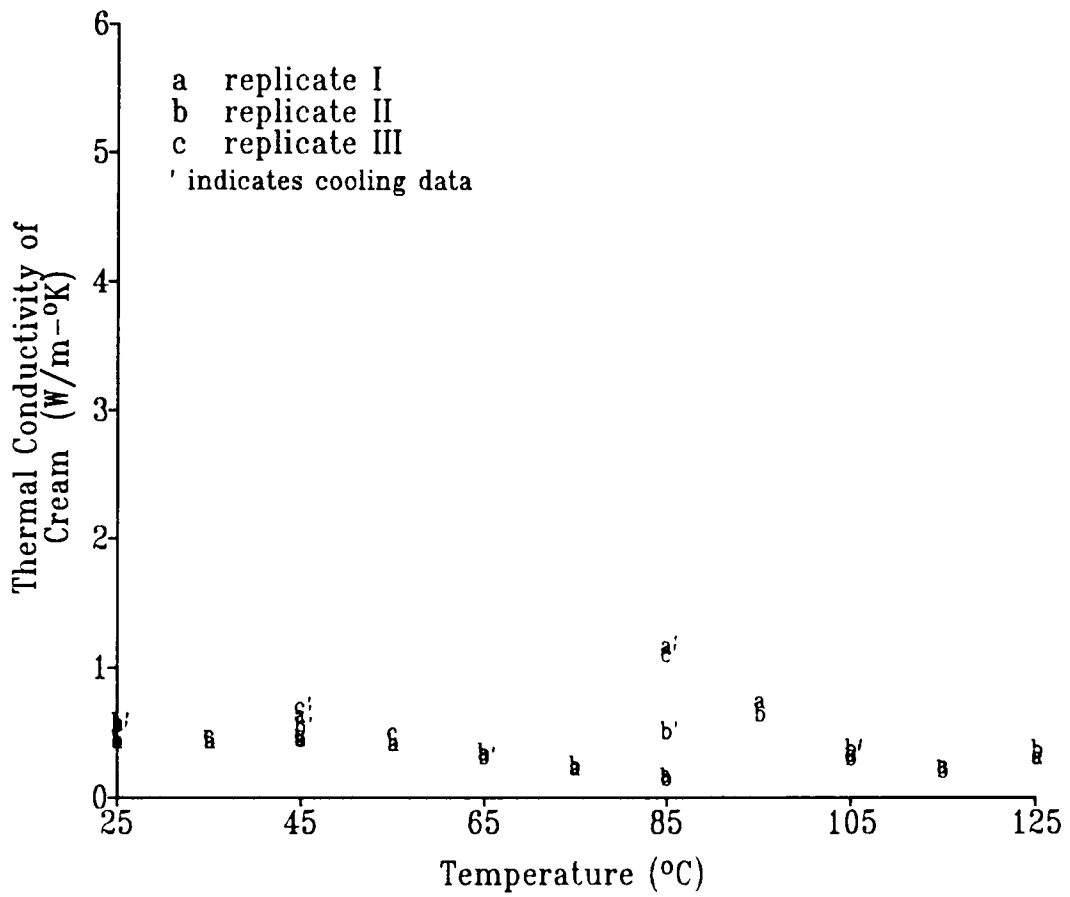


Figure 26. Measured thermal conductivity of cream for all three replicates.

Appendix A4. Heating and cooling values for the three replicates of each sample are plotted against temperature. Percent fat, moisture content, and solids-non-fat data for the products before and after cooking are presented in Table 16.

A series of problems arose during this portion of the study. The first skim milk sample was completed as expected, although the data collection with the 10 kOhm and 20 kOhm probes seemed uncharacteristically unstable. It was often necessary to restart the data collection program to obtain an accurate initial temperature reading from the probe. Analysis of the data the following day showed unusually high values for thermal conductivity of skim milk. During the second replicate run on skim milk, probe 20k1 failed, no stable initial temperatures or power outputs were obtainable. Probe 10k2 was used for 95°C, and the cooling data was collected. High temperature data was not collected at that time. This was the first indication of the thermal runaway problem described in the section III.D.. Probe 20k2 was recalibrated to check for damage to the probe due to thermal runaway and was found to have been affected. New calibration coefficients (and standard errors) for probe 20k2 were: $a_0 = 5.00E-4$ ($1.64E-4$), $a_1 = 3.31E-4$ ($3.56E-5$), $a_2 = -4.99E-7$ ($2.45E-7$). A 10 kOhm trimpot was added to the circuit as the resistor

Table 16. Percent fat, moisture (MC), and solids-non-fat (SNF) for each replicate of each milk product before (B) and after (A) cooking.

Product replicate	%fat		%MC		%SNF	
	B	A	B	A	B	A
Skim Milk						
I	0.06	0.035	90.02	91.81	9.92	8.155
II	0.06	0.045	90.02	89.775	9.92	10.18
III	0.06	0.03	90.02	91.895	9.92	8.075
Whole Milk						
I	2.65	0.06	87.705	91.825	9.645	7.575
II	2.65	2.0	87.705	90.25	9.645	7.75
III	2.65	2.0	87.705	89.355	9.645	8.645
Half-and-Half						
I	18.6	2.3	73.14	88.315	8.26	9.385
II	18.6	2.9	73.14	88.22	8.26	8.88
III	18.6	2.3	73.14	87.625	8.26	10.075
Cream						
I	31.0	31.0	62.95	65.425	6.05	3.575
II	31.0	28.5	62.95	65.60	6.05	5.90
III	31.0	32.5	62.95	66.255	6.05	1.245

immediately after the inverter (Figure 3) to reduce the power output reaching the probes. For the third skim milk replicate Probe 10k2 was again used for 95°C, and the trimpot was only inserted for the 10 and 20 kOhm probes. This test yielded unusually high results similar to that for the first replicate.

The first whole milk replicate again showed some instability with the probe 10k2, and probe 20k2 failed again. The trimpot seemed to have no affect in limiting the thermal runaway problem. The second and third replicates were only run through 95°C, with high temperature data to be collected at a later date. However, samples were still heated to 125°C so that fat, moisture, and solid-non-fat analyses would still reflect the full temperature treatment.

Upon completion of the whole milk samples, probe 20k3 was tried and found to be not hermetically sealed. Probe 20k4 was standardized at 125°C. A 47 Ohm resistor replaced the 10 kOhm trimpot immediately after the inverter in the circuit for standardization of probe 20k4 at 105°C and 115°C. Probe 10k1 replaced 10k2 for the middle temperature range. The 47 Ohm resistor was used at 65, 75, and 85°C during heating, but only at 65°C during cooling. After cooking, the power drop across the probe at 85°C was low enough to not require further reduction. Reasons for

different power levels at 85°C for heating and cooling may be due to an increase in the thermal conductivity of the samples, a result of separated fat rising to the sample surface leaving a higher water concentration around the probe. This would affect the power dissipation characteristics of the probe.

Half-and-half data still showed some variability with probe 10k1, but much less variation than was observed before the 47 ohm resistor was added in response to the thermal runaway problem. As can be seen from Figure 25, in some cases the probe gave usable data, and in others, the same probe at the same temperature gave no analyzable data. However, no additional 10 kOhm probes were available. Probe 20k4 gave good results at 105°C and 125°C. At 115°C, thermal conductivity values, while reproducible, seemed low relative to the temperatures above and below it. The most likely cause of low values at 115°C is an error in standardization of probe 20k4 at this temperature. This probe was used to complete the high temperature studies on skim and whole milk after earlier 20 kOhm probes failed.

At the start of the cream samples, another problem arose. Switches A, B, and C (Figure 3) no longer responded correctly leading to an inability of the data collection program to function properly. In an effort to complete the study, the switches were removed from the circuit and the

necessary switching was accomplished manually.

The inconsistency of probe 10k1 continued through the first two replicates, and by the third replicate, the probe failed to yield any analyzable data. Probe 20k4 continued to give good results at 105°C and 125°C, and lower values at 115°C.

As a result of these difficulties, the means of controlling the method, though not the method itself, was reevaluated. The present control system is not adequate to handle high temperature studies. The inability of the system to precisely control power output to the probes within the specifications for the probes at high temperatures is the primary cause of thermal runaway. This is substantiated by two observations. First, data collected at 25-55°C, where thermal runaway was not a concern, showed good reproducibility and excellent agreement with accepted literature values. Secondly, after addition of the 47 ohm resistor to decrease the voltage supplied to the probes for half-and-half and cream samples, measured thermal conductivity values showed marked improvement in reproducibility. Accuracy shows apparent improvement, but, since the probes were damaged by thermal runaway, the accuracy of these values is suspect. When considering the second observation it must be remembered that the 10 kOhm probes were also switched at this point.

However, probe 10k1, used after addition of the 47 ohm resistor, displayed greater variability than probe 10k2 in the ethylene glycol work presented in section IV.C.5. Decreased variability exhibited with the more variable probe further emphasizes the thermal runaway problem and its potential solution. As in work done by Valvano (1981), Woodbury (1984), and Dougherty (1987), a controllable constant current source should be employed as the source of power for running the bead thermistor method.

Another factor affecting power dissipation by the thermistor probes is resistance of the probes themselves. Resistance of the bead at the desired temperature should be as close to 1000 Ohms as possible to maximize the sensitivity and accuracy of the method. A large initial resistance requires a large voltage drop across the bead to maintain a given power level. The present method control system is not capable of supplying the necessary power. This is why one high resistance probe could not be used to study the full temperature range.

1. Physical Observations

Physical observation of skim milk samples showed extensive caramelization. The milk had turned a rich brown color. There was also significant protein denaturation. Normally white glass wool was brown and matted. There was

also considerable protein precipitate on the bottom of the chamber. At first it was thought this "solid" mass around the probe was responsible for the lower thermal conductivity values. However, this offered no explanation as to why conductivity returned to the same value at 25°C after cooking as the initial 25°C measurement. Apparently, this loss of sugar and protein in the samples had no detectable effect on thermal conductivity of skim milk at 25°C.

Examination of whole milk revealed slightly less caramelization as evident by the color being a slightly lighter shade of brown. Also, there was less protein denaturation as demonstrated by minimal precipitate in the chamber, and little matting in the glass wool. This may be due to fat cushioning the proteins to some extent. Also, while samples were heated to 125°C, they were not held at that temperature as long as if they had undergone a full temperature treatment because the failure of the 20 kOhm probe prevented data collection at the highest temperatures.

Observation of half-and-half showed cooking had less effect on the physical appearance of the samples than on previous milk products. The color was a light shade of brown, and there was no protein precipitate in the chamber. The glass wool rinsed clean and showed no signs of matting.

Some fat had separated out and was floating on top of the sample. Since these samples had undergone the full temperature treatment, the proteins may have been protected from denaturation by the high fat content. Low values for percent fat after treatment are due sampling error, the separated fat not being adequately mixed into the sample prior to fat analysis. The Babcock test for fat utilizes the heat released by acid hydrolysis of protein to liquify and separate out fat. A reduction in protein content during thermal conductivity measurements could cause an artificially low fat measurement using the Babcock test (Bishop, 1988). However, since the temperature treatment exceeded the temperature achieved by acid hydrolysis, this explanation may not account for all of the missing fat in the "after" samples of skim milk, whole milk, and half-and-half.

Inspection of the cream samples showed a very light brown coloration indicating minimal caramelization. A large amount of fat was found separated and floating on top of the sample. As with the half-and-half samples, the glass wool rinsed clean with no matting. High fat content, 67% greater than half-and-half, again cushioned the proteins from the high temperatures. This is corroborated by fat analysis data which showed no artificially low fat content that might have been due to protein loss.

2. Thermal Conductivity Values

Data collected with the 10 kOhm probes at 65-95°C were not included in the following discussion because of the thermal runaway problem during the skim and whole milk samples. Data for half-and-half and cream were not used because, despite the correction of thermal runaway by inclusion of the 47 ohm resistor, the probes had already been affected and the accuracy of these results are, therefore, questionable.

The thermal conductivity of skim milk at 25-55°C compared very well with the values for skim milk presented in Table 3, and with water, since skim milk is 90% water, listed in Appendix A2. These results help substantiate the accuracy of the method. Measured values were consistently higher than those predicted for skim milk using experimental values for fat and moisture content (Table 16) in the empirical model of Fernandez-Martin and Montes (1972) listed in Table 3. Measured values also listed in Table 3 are even farther below thermistor determined values. Thermistor measured thermal conductivities indicate that solids-non-fat made little, if any contribution to thermal conductivity at low temperatures because loss of these components due to caramelization and denaturation had no detectable effect on these values. At 105 and 115°C, however, conductivity values were well below

that expected for pure water. At 125°C, the value increased above that at 115°C, but was still below that of 105°C. At these points, caramelization and protein denaturation served to reduce the thermal conductivity of the product. At 105°C during cooling, thermal conductivity recovered somewhat. When the product had been cooled to 45°C and 25°C, thermal conductivity had returned to pre-cook values. Physical evidence coupled with these results clearly show that some change is occurring in skim milk at thermal processing temperatures. These effects may be reversible with respect to thermal conductivity because the high water content would not show permanent changes in thermal conductivity with heating and cooling.

Initial conductivity of whole milk was below that of the skim milk. This is consistent with the slightly higher fat content. This value also agrees well with the thermal conductivity of a 4% fat product at 20°C as given in Table 3. However, as with skim milk, both the Fernandez-Martin and Montes model prediction values and those determined by Reidy (1968) (Table 3) are consistently lower than the thermistor measured values. Measured thermal conductivity showed the expected gradual increase with an increase in temperature through 45°C. At 55°C, however, conductivity decreased slightly. This decrease was further evident at 105 to 125°C. If protein denaturation is releasing fat

from the casein micelles, this fat, due to hydrophobic interactions, may be concentrating in the glass wool matrix and lowering the measured conductivity near the probe. Whole milk, as with skim milk, showed some recovery as a higher thermal conductivity upon the onset of cooling. By 25°C, conductivity had returned to a level slightly below that of the initial value. Again, possibly due to fat entrapment in the glass wool matrix.

The initial thermal conductivity of half-and-half showed about a 5% decrease from whole milk despite a 600% increase in fat content. The 25°C conductivity of the 18.6% fat half-and-half, 0.557 W/m-°K, was much higher than the 0.382 W/m-°K given for a 20% fat product at 20°C by Govorkov (1950) in Table 3. Slight differences in fat content and temperature are insufficient to explain this discrepancy. Therefore, the difference between these values must be a function of the methods, bead thermistor method and thermal conductivity probe, respectively, used to measure these values. The most probable cause of this variation is in the standardization of the respective methods. As stated in section II.C., the lack of reliable thermal standard reference materials is a serious concern. And whenever possible, thermal conductivity values taken from the literature should be chosen from either one reference or references using the same method of

determination to help minimize potential error associated with mixing data determined by different methods.

The thermal conductivity of half-and-half remained relatively constant from 25-45°C, followed by a slight increase at 55°C. As with the previous two products, the thermal conductivity of half-and-half decreased at 105°C and 115°C, while increasing slightly at 125°C. This may again be due to release of fat from casein micelles. And, as above, conductivity returned to precook values at 45°C, though the 25°C value was slightly below its precook level.

As with half-and-half, thermal conductivity of cream stays relatively constant up to 55°C where it decreases slightly. This decrease continues through 125°C. As with the other three products, thermal conductivity recovers as the cream is cooled, reaching a maximum at 45°C before dropping at 25°C. Post-cook conductivity at 25°C is 29% larger than the precook value because of the release of low thermal conductivity fat seen floating on the surface after the temperature treatment, and a corresponding increase in the higher conductivity water content around the probe below the surface. It is speculated that fat entrapment did not occur with the cream because the high fat content cushioned the proteins from coagulation trapping the fat so the large amount of fat was sufficient to float free of the glass wool matrix.

With the few above noted exceptions, thermal conductivity measurements correlated well with observed physical changes in the products.

The final aspect of this study was to try and fit prediction equations to measured thermal conductivity values. Temperature and powers of temperature were the only independent variables used because a separate equation was derived for each product. Best fit equations were achieved using only data collected at 25-55°C and 105-125°C. Since the middle temperature thermal conductivity values were in error due to thermal runaway and probe damage, no prediction equations for cooling data could be determined because of insufficient data. Therefore, best fit equations were only attempted for heating data. First, second, and third order equations in temperature were fit to the data, and the significant coefficients of the best fit equations are presented below with the standard errors for the coefficients and the coefficients of determination.

skim milk ($r^2 = 0.3546$)

$$k = 0.699$$

Coefficient standard error is 0.0849.

whole milk ($r^2 = 0.7707$)

$$k = 3.91E-2*T - 5.44E-4*T^2 + 2.14E-6*T^3$$

Coefficient standard errors are 2.264E-2, 3.383E-4, and 1.542E-6, respectively.

half-and-half ($r^2 = 0.5006$)

$$k = 0.636$$

Coefficient standard error is 0.0637.

cream ($r^2 = 0.7755$)

$$k = 0.508 + 1.82E-3*T$$

Coefficient standard errors are 0.0359 and 4.390E-4, respectively.

None of the above equations fit the data very well. The most likely explanation for this is simply that the model, integer powers of temperature, is not the correct one. Examination of Figures 23-26 show that milk thermal conductivity data varied little with temperature. Therefore, a reasonable approximation of milk product conductivity can be made by assuming a constant value over the measured temperature range as was done by Swartzel (1982). Physical changes occurring in the milk products at thermal processing temperatures appear to counter the rise in thermal conductivity expected due to the temperature dependence of conductivity of the water and fat components. A detailed study of how physical changes in a product during processing affect thermal properties, and, therefore, sterilization rates, would yield valuable information for product and process development.

V. Conclusions

The following conclusions summarize the bead thermistor method as a means of determining thermal conductivity of food materials through thermal processing temperatures:

- The method is practical. Accuracy and percent coefficient of variation determinations showed that the method compares favorably with existing methods of thermal conductivity measurement. Data collection proceeds for 20 seconds. This data can be converted to a thermal conductivity value in less than 1 minute using a program for simple linear regression.

- The procedure is useable through thermal processing temperatures. Accuracy data showed that the method is most accurate at 125°C. Accuracy and percent coefficient of variation results were consistent with that predicted by the error analysis. The degree of correlation between physical changes in the milk products and measured thermal conductivities strengthens this conclusion.

- Food particulates with a diameter of 5 mm or larger can be tested. Minimum particle size experiments showed that the infinite boundary condition assumption necessary to

solve the heat transfer equation is maintained when the minimum dimension of the material being measured is ≥ 5 mm. Beyond this restriction, the determination of thermal conductivity is independent of the shape of the material being tested.

- Standardization of the probes is of critical importance to the accuracy of the method. New and better thermal conductivity reference materials need to be developed. The temperature dependence of the thermal conductivity of reference materials also requires a thorough examination.

- A two-point water-castor oil standardization is sufficient for an accurate method. Omitting glycerin as a reference material gave a more accurate procedure than when all three materials were used.

- There are differences between probes. It is evident that the magnitude of the standard deviations on the temperature-resistance calibration coefficients is not a good criterion for choosing a probe. Several probes should be evaluated for accuracy and reproducibility before selecting one for use in the procedure.

- High temperature-long time cooking of milk products has severe physical effects on those products. These effects resulted in no appreciable change in the thermal conductivity values as measured in this study. An examination of how these physical changes affect thermal conductivity would provide useful data for food processors.

VI. Recommendations

The bead thermistor method for determination of thermal conductivity as delineated in this study is a fast, accurate, and relatively simple technique. However, certain aspects of the procedure warrant continued evaluation and refinement.

Power output to the probes needs to be more precisely controlled. This is best accomplished with a programmable constant current source. Power applied to the probe during the test at a given temperature should not exceed its power dissipation constant, leading to degradation of the bead material and probe failure.

The ability of the thermistor method to determine thermal diffusivity and thermal conductivity simultaneously should be developed.

The control circuit should be miniaturized to a single integrated circuit. This would further reduce the time necessary to obtain a final conductivity value, and improve the reproducibility of the method. The system has the potential to be reduced to a simple, possibly hand-held instrument that would display both thermal conductivity and thermal diffusivity directly in a matter of seconds.

New thermal reference materials need to be developed, and their temperature dependence evaluated. Given better

standards, the accuracy of the method can be improved.

Sensitivity of the thermistor method should be determined for how small of a change in thermal conductivity can be accurately detected. This would prove useful when examining how physical changes in a product affect thermal conductivity. It may also provide a means for using a bead thermistor probe as a way to monitor these changes.

The minimum particle size determination should be reevaluated as a function of temperature. To meet the needs for aseptic processing of food particulates, the full capabilities of the bead thermistor method to accurately and precisely measure thermal properties of small food particulates through thermal processing temperatures should be developed.

VII. Appendix

Appendix A1. Computer program used for method control and data acquisition. Probe coefficients and set resistances are for a 1 kOhm probe at 25°C.

```
900 CLEAR ,58368! ' for interprative basic hex = &HE400
910 IBINIT1 = 58368! ' for interprative basic
920 IBINIT2 = IBINIT1 + 3 ' for interprative basic
930 BLOAD "BIB.M",IBINIT1 ' for interprative basic
940 CALL BINIT1(IBFIND,IBSTOP,IBTRG,IBCLR,IBPCT,IBSIC,
IBLOC,IBPPC,IBBNA,IBONL,IBRSC,IBSRE,IBRSV,IBPAD,
IBSAD,IBIST,IBDMA,IBEOS,IBTMO,IBEOT) 'interprative
950 CALL IBINIT2(IBGTS,IBCAC,IBWAIT,IBPOKE,IBWRTF,IBWRTA,
IBWRT,IBCMDA,IBCMD,IBRDE,IBRDA,IBRD,IBRPP,IBRSP,IBDIAG,
IBXTRC,IBSTA%,IBERR%,IBCNT%) 'interprative
960 '
970 '-----
980 ' USING COMPILED BASIC
990 '-----
991 '
992 ' THE ABOVE STATEMENTS ARE USED WHEN INTERPRATIVE BASIC
IS BEING USED.
993 ' FOR COMPILED BASIC THE FOLLOWING STATEMENT IS ALL
THAT IS NEEDED.
994 '
995 ' COMMON IBSTA%, IBERR%, IBCNT%
996 '
1000 DEF SEG = 8192 : PCT = 0! ' 2000 Hex
1010 BLOAD "PCTHERM",PCT
1050 '
1060 ' Created: 20 Nov 86, Program struture
1065 ' Revised: 26 Nov 86, Added GPIB control
1070 '
1080 ' Copyright (C) Virginia Tech - Department of Food
1090 ' Science and Technology (1986)
1100 '
1110 ' This program is intended to be used with the DT2805
1120 ' data aquisition board made by Data Translation.
1130 ' The program will control the experimental
requirements
1140 ' associated with the HEATED THERMISTOR BEAD - thermal
1150 ' diffusivity and conductivity measurements.
1160 '
1170 ' The structure of the program is as follows:
1190 ' 1. the resistance of the thermistor is measured and
1200 ' the temperature of the thermistor is calculated.
1210 ' 2. the resistance needed for the thermistor + 4 deg
C is
```

```

1220 '   calculated and then the possible resistance the
1230 '   equipment is set to handle is determined.
1240 ' 3. after setting the desired set-point resistance
    the
1260 '   resistance is measured using an outside Ohm
    meter.
1270 ' 4. the user inputs both the resistance of the
    thermistor and
1280 '   the measured resistance of the set-point.
1290 ' 5. the experiment is then run and the voltage is
    measured
1300 '   for the duration of the test.
1310 ' 6. the necessary end results are then calculated
    from the
1320 '   collected information.
1330 '
1710 '   DEFINT J-N,X
1720 '
1800 ' -----
1810 ' SET-UP COMPUTER FOR DATA COLLECTION
1820 ' -----
1825 ON ERROR GOTO 9800           'ON ERROR GO TO ERROR ROUTINE
1830 DIM R!(4),PRES!(16)
1850 DIM V!(2000),T!(2000)      'ARRAY FOR STORAGE OF VOLTS
1855 GOSUB 9100                 'SET PROGRAM DEFAULT VALUES
1860 GOSUB 9500                 'SET DEFAULT D/A VALUES
1870 GOSUB 10000                'SET DT2805 OFFSET VALUES
1875 GOSUB 11000               'SET GPIB DEFAULT VALUES
1880 I% = 1 : CALL XSB(I%)      'SELECT DT-BOARD NUMBER 1
2010 ECW% = &HO                 'Shut off PCLAB error report
2020 CALL XSECW(ECW%)          'SET.ERROR.CONTROL.WORD
2040 CALL XSAR(DT.VL,DT.VH)    'CALL SET.ADC.RANGE
2050 GOSUB 9000                'CHECK FOR ERROR
2060 IOP% = 1 : CALL XEFO(IOP%) 'SELECT I/O PORT 1
2070 GOSUB 9000                'CHECK FOR ERROR
2080 MASK%=&HFF:HILOW%=&HFF     'SET ALL I/O PORTS TO OFF
2082 CALL XODV(IOP%,MASK%,HILOW%)
2084 GOSUB 9000                'CHECK FOR ERROR
2085 DEF SEG
2086 CALL IBFIND(DEV$,DVM%)     'SELECT GPIB DEVICE
2088 CALL IBCLR(DVM%)          'CLEAR GPIB DEVICE
2090 GOSUB 8900                'CHECK FOR ERROR ON GPIB
2100 '
2110 ' -----
2120 ' START SET-UP OF PROGRAM
2130 ' -----
2140 '
3000 CLS
3010 LOCATE 1,21:PRINT "HEATED THERMISTOR BEAD CONTROL
    PROGRAM"

```

```

3020 LOCATE 3,1 :PRINT "System set to measure thermistor
      resistance"
3022 LOCATE 4,1 :INPUT "Set Switch and Press RETURN";A$
3024 DEF SEG = 8192
3025 HILOW% = (G3% OR G1%) XOR MASK% :
      CALL XODV(IOP%,MASK%,HILOW%)
3026 WRT$ = "F2R3S3T3WOG1POY"+LF$+"X" : DEF SEG
3027 CALL IBWRT(DVM%,WRT$) `SET GPIB FOR AUTO RANGE-OHMS
3028 FOR I = 1 TO 300 : NEXT `WAIT A BIT FOR THE OHM METER
3029 CALL IBTRG(DVM%):CALL IBRD(DVM%,RD$) `MEASURE
      THERMISTOR RESISTANCE
3030 RESO! = VAL(RD$) `CONVERT TO REAL NUMBER
3032 WRT$="T4X" : CALL IBWRT(DVM%,WRT$)
3035 LOCATE 3,1 :PRINT " " "
3036 LOCATE 4,1 :PRINT " " "
3037 RES! = RESO! : GOSUB 9300 : TEMPO! = TEMP!
3040 LOCATE 3,1 :PRINT USING "Initial Thermistor resistance
      ###,###.##,";RESO!
3045 LOCATE 3,44 :PRINT USING "Temperature ###.###";TEMPO!
3050 GOSUB 9400 `CALCULATE RSET VALUE AND RMASK%
3060 LOCATE 4,1 :PRINT "RSET estimate = ";RSETR! :
      DEF SEG = 8192
3070 LOCATE 5,1 :INPUT "Set Switch and Press RETURN";A$
3075 HILOW% = (G4% OR G1% OR RMASK%) XOR MASK% :
      CALL XODV(IOP%,MASK%,HILOW%)
3080 FOR I = 1 TO 3000 : NEXT : DEF SEG `WAIT A BIT
      FOR THE OHM METER
3081 WRT$="T3X" : CALL IBWRT(DVM%,WRT$)
3082 CALL IBTRG(DVM%):CALL IBRD(DVM%,RD$) `MEASURE
      THERMISTOR RESISTANCE
3083 RSETR! = VAL(RD$) `CONVERT TO REAL NUMBER
3084 WRT$="T4X" : CALL IBWRT(DVM%,WRT$)
3085 LOCATE 4,1 :PRINT " " "
3090 LOCATE 4,1 :PRINT USING "RSET resistance of
      ###,###.##,";RSETR!
3092 RES! = RSETR! : GOSUB 9300
3095 LOCATE 4,44 :PRINT USING "Temperature ###.###";TEMP!
3100 LOCATE 5,1 :PRINT " " "
3110 `
3120 ` -----
3130 ` START COLLECTION OF DATA
3140 ` -----
3150 `
3160 LOCATE 6,22 :PRINT "SYSTEM READY FOR COLLECTION OF
      DATA"
3170 LOCATE 8,1 :INPUT "Enter FILENAME to be used for
      storage of data ";FLN$
3180 OPEN "O",#1,FLN$
3190 LOCATE 9,1 :INPUT "Enter JOB description ";JOBDS$
3191 PRINT #1,CHR$(34);JOBDS$;CHR$(34) `PRINT JOB
      DESCRIPTION

```

```

3195 PRINT #1,CHR$(34);"Initial Thermistor Temperature =
";CHR$(34);TEMPO!
3196 PRINT #1,CHR$(34);"Initial Thermistor Resistance =
";CHR$(34);RESO!
3197 PRINT #1,CHR$(34);"Rset Resistance =";CHR$(34);
RSETR!;" ";CHR$(34);"Rset Temp. =";CHR$(34);TEMP!
3198 DELTAT! = TEMP! - TEMPO!
3199 PRINT #1,CHR$(34);"Delta T = ";CHR$(34);DELTAT!
3205 PRINT #1,CHR$(34);DATE$;CHR$(34)           `PRINT DATE
3210 PRINT #1,CHR$(34);TIME$;CHR$(34)           `PRINT TIME
3220 LOCATE 10,1 :INPUT"Enter Duration of Experiment
(secs)";TDUR%
3230 ON TIMER(TDUR%) GOSUB 4000                   `AT END OF
EXPERIMENT GOTO 4000
3231 DEF SEG : WRT$ = "FOR4S3T3X"
3232 CALL IBWRT(DVM%,WRT$) : CALL IBTRG(DVM%)     `SET
GPIB FOR DCVOLTS
3240 LOCATE 11,1 :INPUT"Press RETURN to Start
Experiment";A$
3245 `-----
3246 ` START EXPERIMENT
3247 `-----
3260 LOCATE 13,4 : PRINT "TIMER                VOLTS" :
DEF SEG = 8192
3300 HILOW% = (G3% OR G4% OR RMASK%) XOR MASK% :
CALL XODV(IOP%,MASK%,HILOW%)
3301 DEF SEG
3304   I% = 0
3305   TIMER ON                               `START TIMER FOR TEST
3310   CALL IBTRG(DVM%)                       `TRIGGER GPIB
3311   CALL IBRD(DVM%,RD$)                     `READ VOLTAGE
3312   VOLTS! = VAL(RD$)                       `CONVERT TO REAL NUMBER
3314   I% = I% + 1                             `INCREMENT COUNTER
3315   V!(I%) = VOLTS!                         `RECORD VOLTAGE
3316   T!(I%) = TIMER                          `RECORD TIME
3320 ` PRINT #1,TIMER;" ";VOLTS!             `STORE RESULTS
3330 LOCATE 14,1 :PRINT USING "#####.###";TIMER `PRINT
TIME
3340 LOCATE 14,17:PRINT USING "###.####";VOLTS! `PRINT
VOLTS
3350 GOTO 3310                               `CONTINUE TILL TIME UP
3390 `
4000 `-----
4010 ` START ANALYSIS OF COLLECTED DATA
4020 `-----
4030 TIO = T!(1)
4040 FOR J=1 TO I%
4050   VOLTS! = V!(J)*V!(J)
4060   TI = T!(J) - TIO
4070   PRINT #1,TI;" ";VOLTS!
4080 NEXT J

```

```

4090 '
5990 DEF SEG = 8192 : CALL XODV(IOP%,MASK%,MASK%)
5991 DEF SEG : WRT$ = "FOR4S3T4X" : CALL IBWRT(DVM%,WRT$)
5992 I% = 0
5993 CALL IBLOC(DVM%)
5994 CALL IBONL(DVM%,I%)
6000 END
8890 ' *****
8900 ' -----
8910 ' CHECK FOR ERROR WITH GPIB
8920 ' -----
8930 IF IBSTA% < 0 THEN 8950 'CHECK STATUS VALUE
8940 RETURN
8950 LOCATE 22,1 : PRINT "ERROR HAS OCCURRED WITH GPIB"
8960 LOCATE 23,1 : PRINT "STATUS WORD = ";IBSTA%
8970 LOCATE 24,1 : PRINT "ERROR CONDICTION = ";IBERR%
8980 GOTO 5990 'ABORT ON ERROR
9000 ' -----
9010 ' CHECK FOR DT2805 ERROR AND PRINT MESSAGE
9020 ' -----
9030 IER% = 0 'CHECK FOR ERROR MESSAGE
9040 CALL XGEC(IER%) 'GET.ERROR.CODE
9050 IF(IER% = 0) THEN RETURN
9060 LOCATE 22,1 : PRINT " Unexpected error # ";IER%;" Has
Occurred WITH DT2805 "
9070 GOTO 5990
9100 ' -----
9110 ' SET PROGRAM DEFUALTS
9120 ' -----
9130 G1%=&H1:G2%=&H2:G3%=&H4:G4%=&H8 'SET
I/O-1 PORT VALUES
9140 G5%=&H10:G6%=&H20:G7%=&H40:G8%=&H80 'G?% =
PORT NUMBER
9150 S1%=&H100:S2%=&H200:S3%=&H400:S4%=&H800 'SET
I/O-0 PORT VALUES
9160 S5%=&H1000:S6%=&H2000:S7%=&H4000:S8%=&H8000 'S?% =
PORT NUMBER
9200 R!(0)=753!:R!(4)=15.6:R!(3)=31.3 'DEFINE
POSSIBLE RESISTANCE
9210 R!(2)=62.2:R!(1)=124! 'VALUES FOR RSET.
9215 TRISE!=4! 'SET DESIRED TEMP. RISE
9216 RTHERO!=1.381647E-03 : RTHER1!=2.907325E-04 'SET
THERMISTOR EQUATION
9217 RTHER3!=1.423007E-08 'COEFFICIENTS
9220 RETURN
9300 ' -----
9310 ' CALCULATION OF THERMISTOR TEMPERATURE
9320 ' -----
9325 RLN! = LOG(RES!)
9330 TEMP! =1!/(RTHERO!+RTHER1!*RLN!+
RTHER3!*RLN!*RLN!*RLN!) - 273.15

```

```

9340 RETURN
9400 ' -----
9410 ' CALCULATION OF RSET AND RMASK
9415 ' -----
9420 PRES!(1) = R!(0) ' MINIMUM RESISTANCE
9421 PRES!(2) = R!(0)+ R!(4) ' PRES!
CONTAINS EACH
9422 PRES!(3) = R!(0)+ R!(3) ' OF THE
POSSIBLE RESISTANCES
9423 PRES!(4) = R!(0)+ R!(3)+R!(4)
9424 PRES!(5) = R!(0)+ R!(2)
9425 PRES!(6) = R!(0)+ R!(2)+ R!(4)
9426 PRES!(7) = R!(0)+ R!(2)+R!(3)
9427 PRES!(8) = R!(0)+ R!(2)+R!(3)+R!(4)
9428 PRES!(9) = R!(0)+R!(1)
9429 PRES!(10) = R!(0)+R!(1)+ R!(4)
9430 PRES!(11) = R!(0)+R!(1)+ R!(3)
9431 PRES!(12) = R!(0)+R!(1)+ R!(3)+R!(4)
9432 PRES!(13) = R!(0)+R!(1)+R!(2)
9433 PRES!(14) = R!(0)+R!(1)+R!(2)+ R!(4)
9434 PRES!(15) = R!(0)+R!(1)+R!(2)+R!(3)
9435 PRES!(16) = R!(0)+R!(1)+R!(2)+R!(3)+R!(4)
9446 FOR I = 1 TO 16 'FIND RESISTANCE
NEEDED FOR
9447 RES! = PRES!(I) : GOSUB 9300 'DESIRED
TEMPERATURE RISE
9450 IF TEMP! <= TEMPO!+TRISE! THEN GOTO 9460
9455 NEXT I
9456 LOCATE 10,1
9457 PRINT "**** ERROR **** TEMPERATURE OUT OF RESISTANCE
RANGE - ABORTING"
9458 GOTO 5990
9460 RMASK% = (16 - I) * 16 'SET RESISTANCE
MASK FOR SWITCHES
9470 RSETR! = PRES!(I)
9490 RETURN
9500 ' -----
9510 ' SUBROUTINE FOR DEFAULT D/A BOARD
9520 ' -----
9560 DT.VH = 10! : DT.VL = -10! ' DT2805 High and
low VOLTAGE values
9600 RETURN
9800 ' -----
9810 ' ERROR HANDLEING ROUTINE
9820 ' -----
9830 LOCATE 22,1 : PRINT "ERROR NUMBER : ";ERR
9840 LOCATE 23,1 : PRINT "HAS OCCURRED AT LINE NUMBER :
";ERL
9850 LOCATE 24,1 : PRINT "ABORTING PROGRAM"
9860 GOTO 5990
9990 ' -----

```



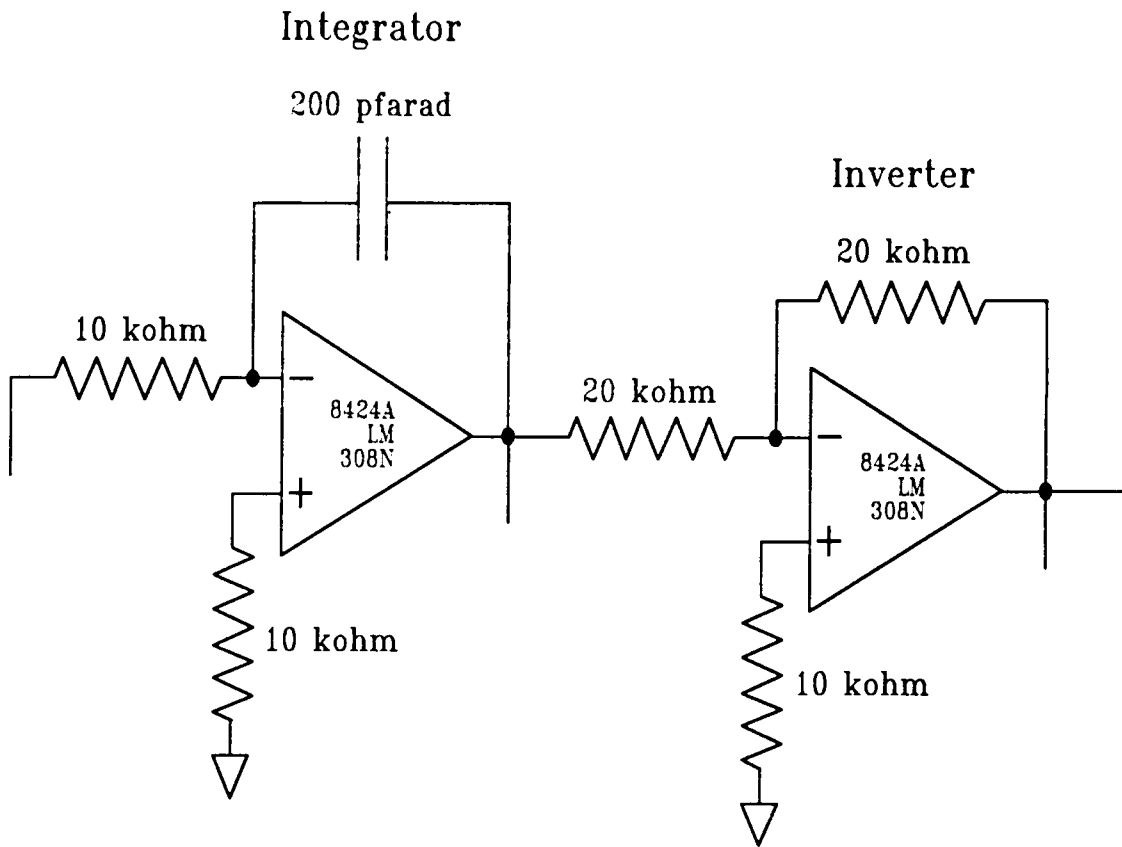
```

10000 ' SUBROUTINE FOR SETTING DEFAULT OFFSET VALUES
10010 ' -----
10240 XEFO = 60 + PCT      ' ENABLE.FOR.OUTPUT
10260 XODV = 66 + PCT      ' OUTPUT.DIGITAL.VALUE
10290 '
10300 XSECW = 75 + PCT     ' SET.ERROR.CONTROL.WORD
10310 XGEC = 78 + PCT     ' GET.ERROR.CODE
10320 '
10330 XSB = 81 + PCT       ' SELECT.BOARD
10360 XSAR = 90 + PCT     ' SET.ADC.RANGE
10370 XSAC = 93 + PCT     ' SET.ADC.CHANNELS
10380 XSDR = 96 + PCT     ' SET.DAC.RANGE
10400 XST = 105 + PCT     ' SET.TIMEOUT
10650 '
10700 RETURN
11000 ' -----
11010 ' GPIB Command Defaults
11020 ' -----
11950     REM
11960     REM Miscellaneous
11970     REM
11990     LE$ = CHR$(&HA)
12000     REM
12010     REM Application program variables passed to
12020     REM GPIB functions
12030     REM
12040     JOBD$ = SPACE$(255)      ' job description buffer
12050     RD$ = SPACE$(25)        ' read buffer
12060     WRT$ = SPACE$(255)      ' write buffer
12070     BDNAME$ = "GPIB0"      ' board/device name
12080     FLN$ = SPACE$(50)      ' file name
12085     DEV$ = "K1"           ' DEVICE NAME
12090     RETURN
^Z

```

Appendix A2. Thermal conductivity of glycerin, water, and castor oil as a function of temperature. All values are from Kaye and Higgins (1928) except for water from 85-125°C which are from Touloukian (1970).

Temperature (°C)	glycerin (W/m-°K)	water (W/m-°K)	castor oil (W/m-°K)
25	0.2853	0.6107	0.1799
35	0.2868	0.6361	0.1795
45	0.2883	0.6434	0.1787
55	0.2900	0.6497	0.1779
65	0.2918	0.6575	0.1771
75	0.2930	0.6654	0.1762
85	0.2945	0.6730	0.1752
95	0.2960	0.6787	0.1742
105	0.2974	0.6829	0.1733
115	0.2989	0.6854	0.1724
125	0.3004	0.6863	0.1715



Appendix A3. Detailed blow-up of Integrator/Inverter portion of the control circuit

Appendix 4. Thermal conductivity data for the three replicates of the four milk products

Appendix A4.a.i. Skim milk thermal conductivity data for replicate I

replicate	temp (°C)	probe	mean	standard deviation	%COV
I	25	1k1	0.602	0.0061	1.013
	35		0.632	0.0052	0.8228
	45	2k1	0.643	0.0041	0.6376
	55		0.652	0.0050	0.7669
	65	10k2	0.837	0.0383	4.576
	75		0.926	0.0627	6.771
	85		1.78	0.2695	15.14
	95	20k1	3.08	0.7112	23.09
	105	20k4	0.644	0.0430	6.677
	115		0.313	0.0214	6.837
	125		0.514	0.0742	14.44
	105		0.532	0.0278	5.226
	85	10k2	1.29	0.2148	16.65
	65		0.982	0.0926	9.430
	45	2k1	0.660	0.0160	2.424
	25	1k1	0.597	0.0068	1.139

Appendix A4.a.ii. Skim milk thermal conductivity data for replicate II

replicate	temp (°C)	probe	mean	standard deviation	%COV
II	25	1k1	0.597	0.0063	1.055
	35		0.636	0.0062	0.9748
	45	2k1	0.654	0.0132	2.018
	55		0.648	0.0082	1.265
	65	10k2	0.787	0.0497	6.315
	75		0.780	0.0722	9.256
	85		3.04	0.6590	21.68
	95		5.55	1.905	34.32
	105	20k4	0.668	0.0326	4.880
	115		0.351	0.0367	10.46
	125		0.526	0.0408	7.757
	105		0.617	0.0350	5.673
	85	10k2	2.92	0.9954	34.14
	65		1.13	0.1257	11.12
	45	2k1	0.660	0.0046	0.6970
	25	1k1	0.600	0.0074	1.233

Appendix A4.a.iii. Skim milk thermal conductivity data for replicate III

replicate	temp (°C)	probe	mean	standard deviation	%COV
III	25	1k1	0.601	0.0072	1.198
	35		0.624	0.0110	1.763
	45	2k1	0.650	0.0070	1.077
	55		0.644	0.0047	0.7298
	65	10k2	1.03	0.0526	5.107
	75		1.02	0.1456	14.27
	85		3.14	0.9080	28.92
	95		3.37	1.171	34.75
	105	20k4	0.639	0.0372	5.822
	115		0.308	0.0164	5.325
	125		0.515	0.0374	7.262
	105		0.604	0.0272	4.503
	85	10k2	3.84	1.047	27.26
	65		1.11	0.1423	12.82
	45	2k1	0.636	0.0114	1.792
	25	1k1	0.610	0.0042	0.6885

Appendix A4.b.i. Whole milk thermal conductivity data for replicate I

replicate	temp (°C)	probe	mean	standard deviation	%COV
I	25	1k1	0.595	0.0026	0.4370
	35		0.608	0.0029	0.4770
	45	2k1	0.632	0.0073	1.155
	55		0.617	0.0041	0.6645
	65	10k2	1.028	0.0659	6.410
	75		0.988	0.1012	10.24
	85		2.25	0.5824	25.88
	95		3.70	0.9186	24.83
	105	20k4	0.539	0.0339	6.289
	115		0.310	0.0068	2.194
	125		0.527	0.0369	7.002
	105		0.576	0.0250	4.340
	85	10k2	2.26	0.5627	24.90
	65		1.02	0.1124	11.02
	45	2k1	0.608	0.0082	1.349
	25	1k1	0.570	0.0021	0.3684

Appendix A4.b.ii. Whole milk thermal conductivity data for replicate II

replicate	temp (°C)	probe	mean	standard deviation	%COV
II	25	1k1	0.581	0.0034	0.5852
	35		0.595	0.0042	0.7059
	45	2k1	0.621	0.0045	0.7246
	55		0.614	0.0060	0.9772
	65	10k2	0.370	0.0207	5.594
	75		0.191	0.0178	9.319
	85		2.48	0.3175	12.80
	95		5.16	1.054	20.43
	105	20k4	0.591	0.0428	7.242
	115		0.334	0.0248	7.425
	125		0.552	0.0226	4.094
	105		0.636	0.0845	13.29
	85	10k2	3.07	0.7036	22.92
	65		0.401	0.0172	4.289
	45	2k1	0.616	0.0029	0.4708
	25	1k1	0.574	0.0042	0.7317

Appendix A4.b.iii. Whole milk thermal conductivity data for replicate III

replicate	temp (°C)	probe	mean	standard deviation	%COV
III	25	1k1	0.593	0.0022	0.3710
	35		0.756	0.0444	5.873
	45	2k1	0.984	0.0662	6.728
	55		1.08	0.0694	6.426
	65	10k2	0.735	0.0380	5.170
	75		0.752	0.0623	8.284
	85		0.618	0.0934	15.11
	95		---	---	---
	105	20k4	0.581	0.0400	6.885
	115		0.321	0.0122	3.801
	125		0.507	0.0524	10.34
	105		0.602	0.0498	8.272
	85	10k2	0.494	0.0663	13.42
	65		---	---	---
	45	2k1	0.605	0.0032	0.5289
	25	1k1	0.573	0.0083	1.448

Appendix A4.c.i. Half-and-Half thermal conductivity data
for replicate I

replicate	temp (°C)	probe	mean	standard deviation	%COV
I	25	1k1	0.561	0.0108	1.925
	35		0.561	0.0028	0.4991
	45	2k1	0.555	0.0030	0.5405
	55		0.572	0.0043	0.7517
	65	10k1	0.123	0.0018	1.466
	75		0.099	0.0106	10.69
	85		0.155	0.0059	3.828
	95		0.797	0.1515	19.01
	105	20k4	0.535	0.0114	2.135
	115		0.309	0.0304	9.844
	125		0.469	0.0282	6.012
	105		0.543	0.0130	2.398
	85	10k1	1.06	0.1552	14.64
	65		0.238	0.0102	4.298
	45	2k1	0.561	0.0027	0.4813
	25	1k1	0.536	0.0022	0.4104

Appendix A4.c.ii. Half-and-Half thermal conductivity data
for replicate II

replicate	temp (°C)	probe	mean	standard deviation	%COV
II	25	1k1	0.554	0.0065	1.173
	35		0.559	0.0025	0.4472
	45	2k1	0.560	0.0050	0.8928
	55		0.578	0.0039	0.6747
	65	10k1	0.236	0.0045	1.892
	75		0.098	0.0079	8.115
	85		---	---	---
	95		---	---	---
	105	20k4	0.532	0.0152	2.847
	115		0.321	0.0216	6.727
	125		0.460	0.0301	6.543
	105		0.488	0.0207	4.242
	85	10k1	0.926	0.1167	12.60
	65		0.347	0.0168	4.829
	45	2k1	0.566	0.0022	0.3887
	25	1k1	0.532	0.0031	0.5827

Appendix A4.c.iii. Half-and-Half thermal conductivity data
for replicate III

replicate	temp (°C)	probe	mean	standard deviation	%COV
III	25	1k1	0.556	0.0091	1.637
	35		0.559	0.0028	0.5009
	45	2k1	0.562	0.0024	0.4270
	55		0.675	0.0058	0.8592
	65	10k1	0.401	0.0169	4.209
	75		---	---	---
	85		0.977	0.0896	9.175
	95		0.835	0.1379	16.52
	105	20k4	0.490	0.0239	4.876
	115		0.270	0.0163	6.052
	125		0.474	0.0500	10.55
	105		0.439	0.0312	7.105
	85	10k1	0.995	0.1433	14.40
	65		0.459	0.0363	7.904
	45	2k1	0.559	0.0021	0.3757
	25	1k1	0.526	0.0022	0.4182

Appendix A4.d.i. Cream thermal conductivity data for replicate I

replicate	temp (°C)	probe	mean	standard deviation	%COV
I	25	1k1	0.429	0.0023	0.5361
	35		0.430	0.0026	0.6046
	45	2k1	0.454	0.0015	0.3304
	55		0.409	0.0110	2.689
	65	10k1	0.334	0.0061	1.837
	75		0.222	0.0100	4.518
	85		0.163	0.0063	3.866
	95		0.738	0.0929	12.59
	105	20k4	0.322	0.0102	3.161
	115		0.233	0.0262	11.24
	125		0.307	0.0132	4.300
	105		0.337	0.0107	3.178
	85	10k1	1.16	0.1191	10.27
	65		---	---	---
	45	2k1	0.625	0.0029	0.4640
	25	1k1	0.586	0.0016	0.2730

Appendix A4.d.ii. Cream thermal conductivity data for replicate II

replicate	temp (°C)	probe	mean	standard deviation	%COV
II	25	1k1	0.430	0.0042	0.9769
	35		0.429	0.0025	0.5828
	45	2k1	0.426	0.0017	0.3991
	55		0.406	0.0023	0.5665
	65	10k1	0.330	0.0133	4.036
	75		0.236	0.0248	10.53
	85		0.135	0.0043	3.154
	95		0.625	0.0981	15.70
	105	20k4	0.290	0.0112	3.865
	115		0.205	0.0146	7.125
	125		0.376	0.0160	4.255
	105		0.374	0.0452	12.09
	85	10k1	0.505	0.1289	25.52
	65		0.311	0.0238	7.646
	45	2k1	0.532	0.0507	9.530
	25	1k1	0.579	0.0062	1.071

Appendix A4.d.iii. Cream thermal conductivity data for replicate III

replicate	temp (°C)	probe	mean	standard deviation	%COV
III	25	1k1	0.434	0.0047	1.083
	35		0.465	0.0186	4.000
	45	2k1	0.481	0.0347	7.214
	55		0.491	0.0257	5.234
	65	10k1	---	---	---
	75		---	---	---
	85		---	---	---
	95		---	---	---
	105	20k4	0.334	0.0104	3.107
	115		0.247	0.0176	7.133
	125		0.309	0.0272	8.802
	105		0.322	0.0221	6.854
	85	10k1	1.10	0.2943	26.75
	65		---	---	---
	45	2k1	0.688	0.0339	4.927
	25	1k1	0.508	0.0069	1.358

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