

AUTOMATED LINE-HEAT SOURCE SYSTEM FOR THE MEASUREMENT OF THERMAL CONDUCTIVITY AND DIFFUSIVITY

Douglas S. McGinnis

Engineering and Statistical Research Centre, Research Branch, Agriculture Canada, Ottawa, Ontario K1A 0C6

Contribution no. I-875, received 16 September 1986, accepted 9 March 1987

McGinnis, Douglas S. 1987. Automated line-heat source system for the measurement of thermal conductivity and diffusivity. *Can. Agric. Eng.* **29**: 201-207.

A system was developed for the rapid, automatic, and simultaneous measurement of thermal conductivity and diffusivity of food materials and, with knowledge of the food density, for determination of its specific heat. The system utilizes the line-heat source method for conductivity and diffusivity determination, and operates under the control of a microcomputer which is interfaced with a data logger for temperature and voltage measurements, and with external electronics linked to a line-heat source apparatus. Experimental measurements are made automatically after the user has entered the necessary experimental conditions and desired experimental precision to the microcomputer. The system was found to be extremely simple to use, and capable of providing accurate measurements, together with statistical precision statements, in 6 min or less for agar-water gel. The system software was programmed in IBM-BASIC (version 3.0). The software was written to provide maximum user flexibility, and contains an optional algorithm (subroutine) for automatic calibration of the diffusivity measurement apparatus.

INTRODUCTION

Thermal diffusivity values of foods are used in a variety of food engineering applications involving heat and mass transfer calculations for freezing, cooling, or other thermal processing operations. The thermal diffusivity (α) of a food material depends upon its composition and physical properties and is related to conductivity (k), specific heat (c), and density (ρ) by the relationship

$$\alpha = k/(\rho \cdot c)$$

Thermal property values are reported in the literature for a number of selected food materials, but for an ever increasing inventory of processed and/or manufactured food products, thermal property data are not available or are simply not reported.

For foods undergoing thermal sterilization or pasteurization processes, the formulation of the product and the thermal properties of its components comprise a set of critical factors which determine the thermal response characteristics of the product under the applied heating conditions. Singh (1982) and others have discussed the importance of using the component thermal diffusivity values in numerical or analytical solutions to heat transfer in composite foods, as opposed to the potentially erroneous use of "effective" or "apparent" thermal diffusivity values. It is therefore important that the thermal processing specialist know the thermal properties of each component when applying that knowledge in the design of processes to ensure food safety and optimum quality. This is particularly true in the practical development of thermal processes for heat-sterilizable foods wherein the slowest heating food compo-

nent must be identified on the basis of largest size and/or lowest thermal diffusivity.

A number of methods for thermal diffusivity determination have been reported in the literature. These methods generally require solving equations describing a transient temperature field resulting from an applied constant or controlled heat source or sink. Most of the methods are indirect in that the thermal conductivity, specific heat, and density are measured separately, sometimes under different experimental conditions, and α is calculated. A popular method for food materials involves using a thermal conductivity probe (line heat source) in conjunction with one or more temperature sensing probes at fixed radial distance(s) from it, as described by Mohsenin (1980) and others. This technique has the advantages of reasonably short test duration, small temperature change of the product, and the ability to obtain the conductivity and specific heat under the same test conditions. The technique is well suited to biological materials, especially those which are significantly altered by changes in temperature.

Despite the short test times for this method, extensive computations for the determination of the thermal diffusivity must be carried out using a trial and error method on a digital computer, and transferring data to the computer can be time consuming. It was recognized that a significant time saving could be realized if control of the experimental apparatus, gathering of the data, and subsequent data analysis could all be performed by a single desktop computer. The objective of the work presented here was to develop a sys-

tem with these attributes and to demonstrate its usefulness in making precise measurements of thermal conductivity and diffusivity automatically and rapidly.

THEORY

The history and development of a mathematical basis for the line-heat source method of conductivity and diffusivity determination is presented extensively in the literature (Chang et al. 1980; Mohsenin 1980; Nix et al. 1967). The essential elements of this theory are given here for discussion purposes.

Considering only radial conduction heat flux in an infinite cylinder, the temperature-rise at any point within that cylinder following a suddenly initiated, constant heating rate, line-heat source is given by the following equation (Lowan 1940):

$$T(\theta) = \frac{Q}{2\pi k} \left[\frac{-E}{2} - \ln\beta - \sum_{n=1}^{\infty} \frac{(-1)^n \beta^{2n}}{(2n)(n!)} \right] \quad (1)$$

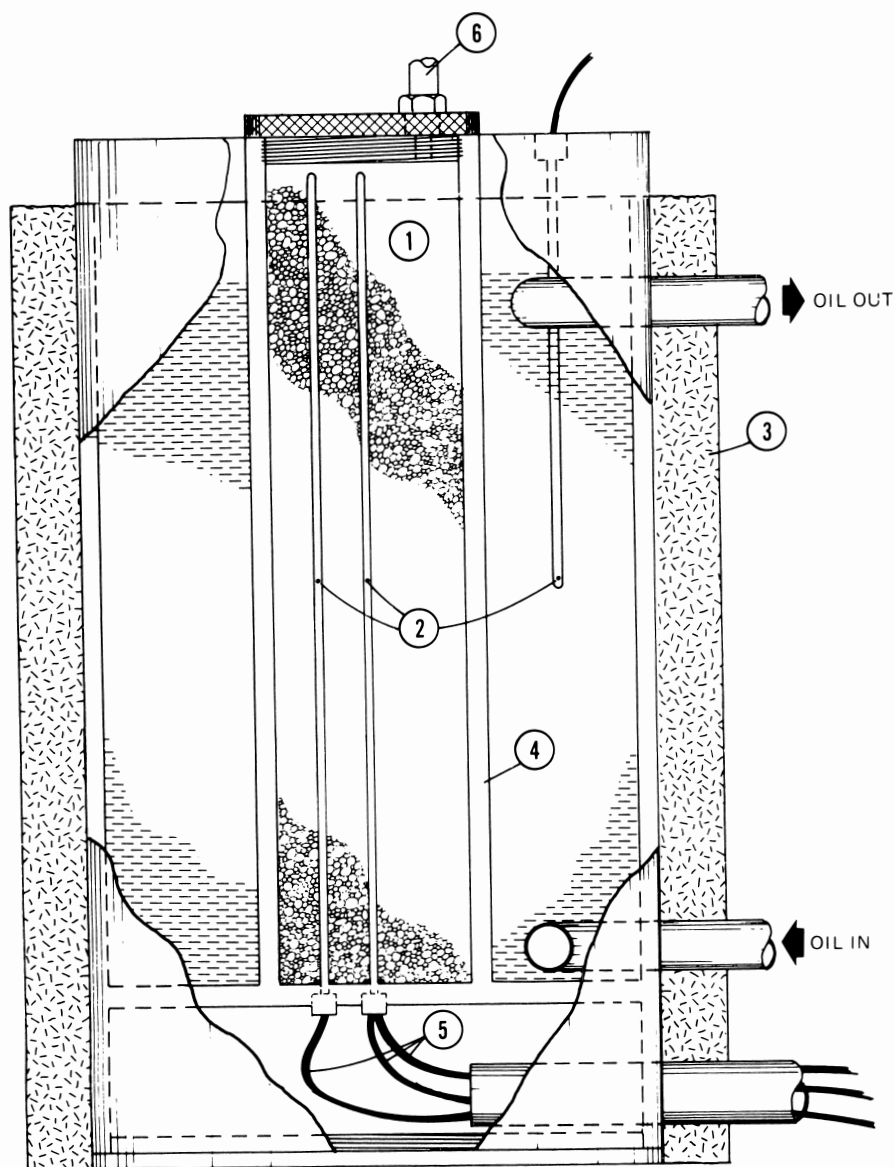
For points close to the line-heat source such that $\beta \leq 0.16$, it has been found that Eq. 1 can be expressed to better than 1% error by using the first two terms (Hooper and Lepper 1950), hence:

$$T(\theta) = \frac{Q}{2\pi k} \left[\frac{-E}{2} - \ln\beta \right] \quad (2)$$

The change in temperature for this point between times θ_1 and θ_2 is, therefore:

$$T(\theta_1) - T(\theta_2) = (Q/(4\pi k)) \ln(\theta_1/\theta_2) \quad (3)$$

The conductivity may then be determined from Eq. 3 knowing the heat source strength and the temperature versus time relationship. Use of a time correction factor has been widely accepted as a means of compensating for the finite diameter and



LEGEND:
 1. HEATING PROBE
 2. THERMOCOUPLE LOCATIONS
 3. THERMAL INSULATION
 4. SAMPLE CYLINDER
 5. LEAD WIRES
 6. OVERPRESSURE PORT (OPTIONAL)

Figure 1. Temperature-controlled line-heat source apparatus for the measurement of thermal conductivity and diffusivity.

unique thermal properties of the line-heat source. The time correction factor (θ_0) is subtracted from each time value and the slope (S) of the time-corrected temperature rise versus $\ln(\theta - \theta_0)$ plot can be used in the calculation of k , giving:

$$k = Q/(4\pi S) \quad (4)$$

where

$$S = \frac{T(\theta_1) - T(\theta_2)}{\ln\left(\frac{\theta_1 - \theta_0}{\theta_2 - \theta_0}\right)}$$

To determine the thermal diffusivity, and hence specific heat, the temperature-rise versus time values for a point a known distance from the line heat source are used

in combination with the known conductivity and density in Eq. 1. To do this, β is found by nonlinear regression analysis of terms in that expression (e.g. 50 terms).

MATERIALS AND METHODS

A thermal diffusivity measurement apparatus, described by McGinnis (1986), was modified and used for the purposes of this work. This apparatus (Fig. 1) consists of two sealed concentric cylinders. The outer cylinder was designed to allow a constant temperature fluid (e.g. oil) to circulate in a spiral fashion around the inner sample cylinder. The sample cylinder contains two parallel (1.7 mm o.d.) probes

1 cm apart: a heating probe (probe no. 1) and a passive probe (probe no. 2). Both probes contain a grounded thermocouple, with its sensing junction at the mid-length location. A constantan heating element is packed inside the hollow probe together with a thermal joint compound (Type 120, Wakefield) to permit rapid heat transfer from the heating element to the surrounding food sample. Because pressure is an important variable in many thermal processes for foods, such as retort processes for foods packaged in flexible retortable containers, the sample cylinder was fitted with a port for controlled application of an overpressure during diffusivity measurements. A third thermocouple is installed in the outer cylinder to monitor the temperature of the circulating fluid.

A miniature thermal diffusivity probe (i.e. pair of probes), similar in construction to the miniature thermal conductivity probe described by Timbers and Robertson (1977), would be well suited for the measurement of small and/or solid particulate food materials.

The system configuration (Fig. 2) provides for the measurement of probe temperatures and DC voltage across the probe heating element. A microcomputer (PC/XT, IBM) connected to an intelligent analog/digital signal converter (Isaac-911, Cyborg) is used for control. A digital data logger (Digistrip-4C, Kaye Instruments) is used for logging, conversion, and scaling of the thermocouple and voltage signals.

In this configuration, digital information is taken directly from the logger through an asynchronous communications port (RS-232) on the microcomputer, at intervals specified by the user. Communication software is combined with a timer routine to request reports from the monitor and to download each report to the microcomputer. These reports are stored as raw data in array form, and broken down into time, temperature, and voltage arrays following each test for subsequent analysis. The data acquisition interval of the system is limited to a minimum of 2 sec by the intrinsic sampling rate of the data logger.

During preliminary work, it was demonstrated that a faster logging rate is possible in a configuration in which the analog temperature signals are supplied by a thermocouple signal conditioner connected directly to an A/D converter which itself is connected to the microcomputer. Scaling and interpretation of the analog signals can then be accomplished after the test period, resulting in an increased potential for high rate data acquisition (i.e. up to a possible 10 000 readings per second per channel for four channels). Such a system

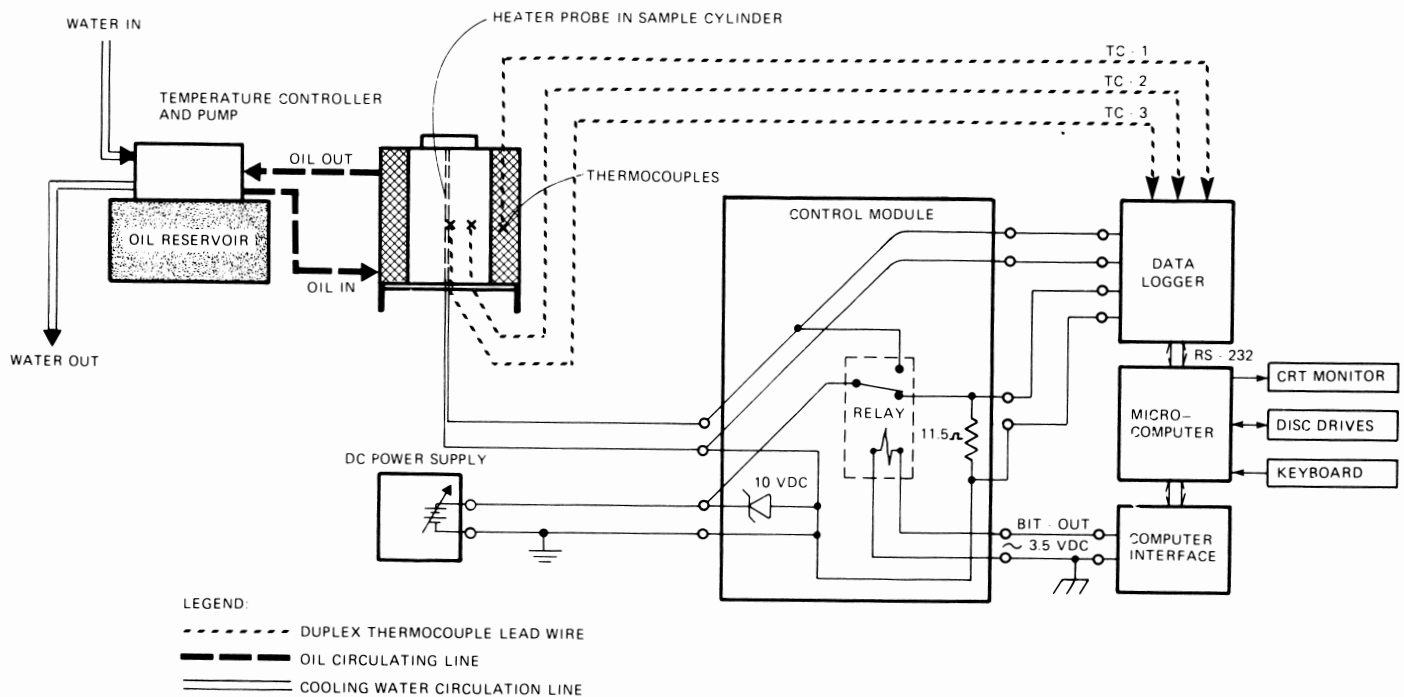


Figure 2. Schematic layout of automated system for conductivity and diffusivity measurement.

can give accurate thermal conductivity measurements in the shortest possible time (e.g. ≤ 10 sec), with the advantage of minimizing temperature changes in the product near the heating probe, yielding a closer estimate of the k value associated with the mean sample temperature during the test. A shorter test time may also be advantageous for some products which undergo irreversible changes in thermal properties at the prescribed test temperature. However, to obtain a sufficient temperature rise at the passive probe location in such a short time for accurate thermal diffusivity determinations, considering temperature measurement precision (resolution $\pm 0.1^\circ\text{C}$) and error, would require that the probes be located very close together (e.g. $r \leq 1$ cm), and/or that the heating power be increased substantially (e.g. $Q \gg 30$ W/m). The finite diameter of the two probes (e.g. 1.7 mm), and their combined influence on heat flow in the vicinity of the probes imposes a limitation on the probe spacing. In addition to this, the probe spacing is limited by the particle size and uniformity of the product. For these reasons, the potentially high rate of temperature data acquisition possible using this configuration was considered to yield no significant advantage for this application.

Heating of the conductivity probe is initiated by the user from the computer keyboard. This operates by way of a LABSOFT command to the A/D, D/A converter producing a latched binary out-

put to a relay (SPDT, R10S-E1Y1-J10K, Potter and Brumfield) which closes the heating circuit. The circuit and software were designed to permit setting of the probe voltage prior to probe energization.

A simplified logic flow chart of the computer program is presented in Fig. 3. Initially the user is prompted to input the desired experimental conditions. The program next monitors temperature conditions until a state of thermal equilibrium, as defined by the user, is achieved between the two probes and between the probes and the constant temperature fluid. Once equilibrium is attained, the user is prompted to initiate probe heating through key entry. The heating probe element is energized, and data are logged until the user-specified test duration is completed, at which time the probe is de-energized. The raw data are then transferred to temperature, time, and voltage arrays. The resistance of the heating probe is calculated for each probe temperature and used with the known voltage to calculate the average probe heat strength (Q) for the test (the constantan heating element resistance varies slightly with temperature, in a linear fashion). The slope of the probe temperature rise versus $\ln(\theta - \theta_0)$ curve is determined by linear least squares regression, and the thermal conductivity is calculated (Eq. 4). Since heating probe temperature rise values during the first few seconds following the start of probe energization can be dramatically affected by the initial temperature response of the probe, the range of time-

temperature pairs used in the regression may be specified by the user to better estimate the straight-line portion of the curve. In the final phase of the program, the user is requested to provide values of parameters by which the diffusivity can be determined by nonlinear least squares regression, fitting Eq. 1 to the observed passive probe temperature rise values. The sample density, number of regression data points to use, the acceptable resolution (maximum estimation bracket size) of the best fit solution (α^1), and a starting estimate of the specific heat for the iterative solution, are supplied by the user. The "Golden Section method" as described by Himmelblau (1972) is used for the unidimensional search algorithm for α^1 .

Confidence limits for the experimental curve fit of Eq. 1 are developed by a second Golden Section search algorithm. This algorithm finds a value of α such that:

$$\sigma(\alpha) = \sigma(\alpha^1) \cdot \left(1 + \frac{1}{m-1} (F(1, m-1, 1-\gamma)) \right) \quad (5)$$

where

$$\sigma(\alpha) = \sum_{i=1}^m (T_i - f(\theta_i, \alpha))^2,$$

and

α^1 = value of α such that σ is minimized.

The probability level (γ) in Eq. 5 is only approximate where f is nonlinear (Draper and Smith 1981). However, Eq. 1 was found to be highly linear (linear $R^2 = 0.95 - 0.99$) for extended segments of the

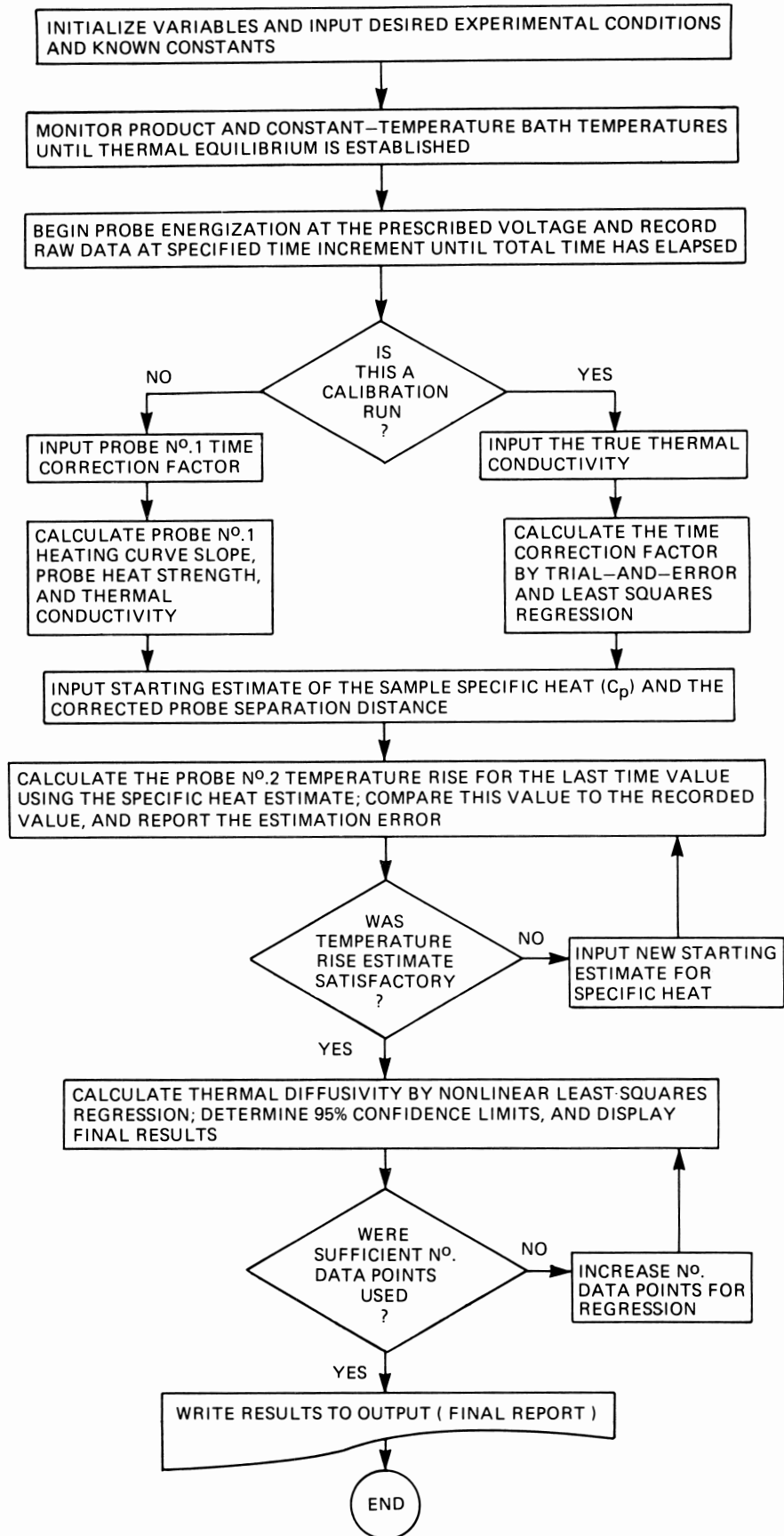


Figure 3. Logic flow diagram of program used to control the thermal property measurement system and to analyze the experimental results.

TABLE I. NOMENCLATURE

Variable	Definition	Units
<i>E</i>	Euler's constant = 0.5772157	—
<i>F</i>	Frequency statistic of the <i>F</i> -distribution	—
<i>f</i>	Predicted temperature-rise using Eq. 1	°C
<i>k</i>	Thermal conductivity	W/(m°C)
<i>m</i>	Sample size	—
<i>n</i>	Positive integers	—
<i>Q</i>	Heat source strength	(W/m)
<i>r</i>	Distance from line heat source	m
<i>R</i> ²	Coefficient of determination (0-1)	—
<i>S</i>	Slope of temperature-rise vs. ln (<i>θ</i> - <i>θ</i> ₀) for heating probe	°C
<i>T</i>	Temperature rise	°C
<i>T</i> _{<i>i</i>}	Observed (<i>i</i> th) temperature-rise	°C
\bar{T}	Sample arithmetic mean temperature-rise	°C
α	Thermal diffusivity	m ² /s
α^1	Best fit solution of thermal diffusivity using Eq. 1	m ² /s
β	$r/(2(\alpha\theta)^{1/2})$	—
γ	Probability level for null hypothesis (0.05 for 95% confidence limits)	—
θ	Time from initiation of line heating	s
θ_0	Time correction factor	s
σ	Sum of squares	°C ²

TABLE II. PROGRAM OUTPUT FILE PRODUCED BY THERMAL CONDUCTIVITY/DIFFUSIVITY MEASUREMENT SYSTEM

Test sample:		THERMAL PROPERTY REPORT			DATE: 86-08-20		
Datafile:		0.5% AGAR			Calibration run		
Applied pressure (kPa)		A:TEST1.DAT					
RAW DATA:		= 0.00					
Data set No.	Time (sec)	Voltage (V)	Heat (W/m)	Bath (°C)	Heating probe 1 (°C)	Passive probe 2 (°C)	T-Rise (°C)
0	0.	0.000	0.000	28.1	28.2	28.2	0.0
6	30.	9.990	28.920	28.1	40.7	28.2	0.0
12	60.	9.980	28.868	28.1	42.9	28.3	0.1
18	90.	9.970	28.813	28.1	44.3	28.4	0.2
24	120.	9.960	28.758	28.1	45.2	28.6	0.4
30	150.	9.950	28.702	28.1	45.9	28.9	0.7
36	180.	9.950	28.703	28.1	46.4	29.1	0.9
42	210.	9.940	28.646	28.1	46.9	29.4	1.2
48	240.	9.930	28.590	28.1	47.3	29.6	1.4
54	270.	9.930	28.591	28.1	47.7	29.8	1.6
60	300.	9.920	28.534	28.1	48.0	30.0	1.8
66	330.	9.920	28.535	28.1	48.3	30.2	2.0
Averages		9.949	28.695	28.1			
SUMMARY:							
Conductivity (W/mC)		= 0.6115					
Density (kg/m ³)		= 996.49					
Specific heat (J/(kg °C))		= 4161.84					
(4137.80 - 4186.17) 95% CL*							
Diffusivity (m ² /sec)		= 0.1474E-06					
(0.1466E-06 - 0.1483E-06) 95% CL*							
* CL = Confidence limits							
Conductivity parameters							
Slope of <i>T</i> vs. log (<i>t</i>) curve (°C)		= 3.73					
Temperature intercept (°C)		= 27.1					
Time correction factor (s)		= -9.0417					
First and last points used		5 & 26					
Coefficient of determination		= 0.99845					
Diffusivity parameters							
Total test duration (s)		= 360.0					
Distance between probes (m)		= .009998					
No. data points used		= 25					
No. terms used in series solution		= 45					
Temperature rise error sum of squares		= 0.207E-01					
Coefficient of determination		= 0.98795					

curve following the first 60–120 sec for the selected range of experimental design conditions (i.e. probe spacing, heat source strength, etc.).

The confidence limits so developed provide the user with a measure of the uncertainty in the results owing mainly to the imprecision of the temperature measurements. In order for the user to resolve and correct for systematic errors, however, the user must first calibrate the apparatus using a substance of known thermal conductivity and diffusivity. In this regard an optional part of the computer program allows the user to automatically determine the time correction factor for the probe apparatus. In this operation the user provides the true (known) thermal conductivity of a test material such as 0.5% agar-water gel and the program finds, by trial and error, the time correction factor for the probes that permits a match, to a user-specified precision (0.01%, say) of the measured and true thermal conductivity.

Equation 1 is highly sensitive to probe separation distance, and it will be necessary to either correct or check for minor disturbances from true parallel alignment of the probes, or for off-center position of the thermocouples within each probe. The corrected probe separation distance can be obtained by trial-and-error calibration using a homogeneous substance of known thermal diffusivity. Systematic or random errors which cannot be corrected directly through calibration must be estimated by the usual experimental replication and subsequent calculation of a mean and standard error for α and *k*.

As a means of assessing the relative degree to which the experimental passive probe data fits Eq. 1 (for α), and the degree to which the heating probe data fits Eq. 4 (for *k*), a coefficient of determination (*R*²) value is calculated for the nonlinear and linear regressions. Of the eight alternative and uniquely different expressions for *R*² identified by Kvalseth (1985), the following recommended form was selected for both regressions:

$$R^2 = 1 - \frac{\sum_{i=1}^m (f_i - T_i)^2}{\sum_{i=1}^m (T_i - \bar{T})^2} \quad (6)$$

RESULTS AND DISCUSSION

Typical program output is presented in Table II. Using 0.5% agar-water gel as a test substance, it was found that a sufficient passive probe temperature rise (e.g. 2°C) could be obtained to enable accurate thermal diffusivity measurements in from 5 to 6 min using a heating strength of 26 W/m and 1 cm probe separation.

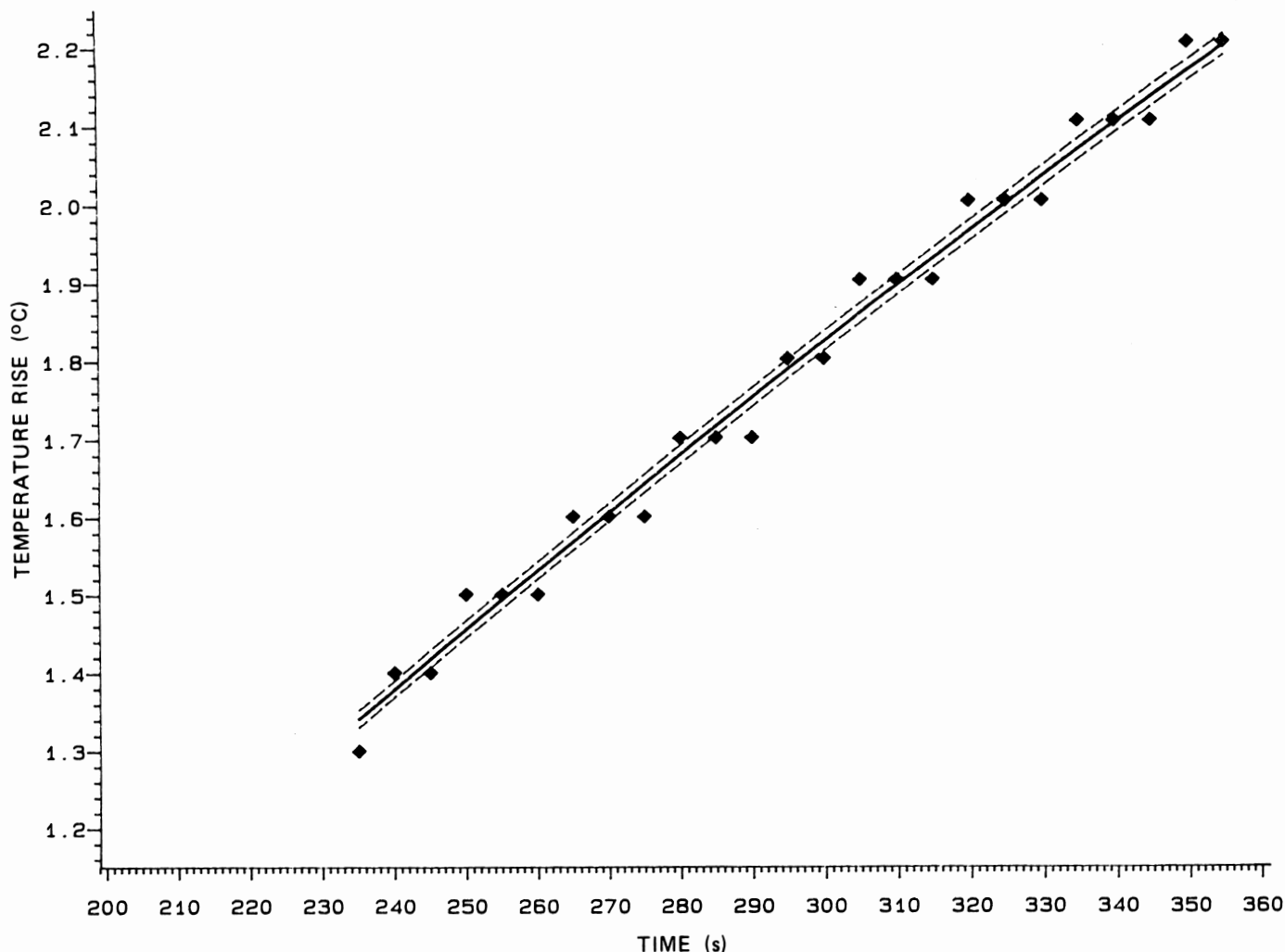


Figure 4. Experimental and predicted temperature rise of the passive probe as a function of heating time.

However, the optimum heating time could vary significantly depending upon the probes used, their separation distance, the heating power applied, and the substance being measured. Table II reports all relevant information pertaining to the calculation of the thermal conductivity and diffusivity respectively. The R^2 value for the heating probe temperature versus \log_e (time) curve was found to be no less than 0.998 for the tests on 0.5% agar water gel using 20 data points. Because the temperature rise for the passive probe is low compared to that of the heating probe, and because of the limited precision of the temperature measurements ($\pm 0.1^\circ\text{C}$), the number of data points required for accurate diffusivity determination was found to increase with decreasing test time or with decreasing overall temperature rise of the passive probe. Table II provides the residual sum of squares value and 95% confidence limits for this regression. Figure 4 shows the last 25 measured temperature rise values from the data presented in Table II, together with the

predicted temperature rise curve. Figure 4 shows the two temperature rise profiles (dashed lines) for the upper and lower 95% confidence limits of α . These curves are not to be confused with 95% confidence contours for the temperature-rise versus time data. The approximately uniform pattern of the temperature-rise error residual (measured-predicted) indicates that Eq. 1 provides a good fit to the data, and provides evidence of good equipment performance.

It was found that approximately 12 iterations are required to obtain α to within 0.1% of the "best-fit" solution using 50 terms of Eq. 1 and 20 temperature-rise vs. time data pairs.

SUMMARY

An automatic system for the measurement of thermal conductivity and diffusivity has been successfully developed. The development system reported here could be further refined and redesigned towards production of a more compact commercial system by combining the fun-

damental electronic components into a single package, with the attendant advantages of lower system cost and portability. However, the system can be easily assembled from components which may already exist in many well-equipped thermal process development laboratories.

ACKNOWLEDGMENT

The author is grateful for technical support provided by Dave Dow of the Engineering and Statistical Research Centre (E.S.R.C.), and for assistance provided by associates and staff of E.S.R.C. on this project.

REFERENCES

- CHANG, C. S., F. S. LAI, and B. S. MILLER. 1980. Thermal conductivity and specific heat of grain dust. *Trans ASAE (Am. Soc. Agric. Eng.)* 23: 1303-1306, 1312.
- DRAPER, N. R. and H. SMITH. 1981. *Applied regression analysis*. 2nd ed. John Wiley and Sons, Inc., Toronto, Ont.
- HIMMELBLAU, D. M. 1972. *Applied nonlinear programming*. McGraw-Hill Book Co., Toronto, Ont.
- HOOPER, F. S. and A. R. LEPPER. 1950.

- Transient heat flow apparatus for the determination of thermal conductivity. Am. Soc. Heat. Vent. Eng. Trans. 56: 309–324.
- KVALSETH, T. O. 1985. Cautionary note about R^2 . The American Statistician 39(4): 279–285.
- LOWAN, A. N. 1940. Tables of sine, cosine and exponential integrals. Vol. 2. New York Math Project, National Bureau of Standards, Washington, D.C.
- MOHSENIN, N. N. 1980. Thermal properties of foods and agricultural materials. Gordon and Breach Science Publishers, New York.
- McGINNIS, D. S. 1986. Prediction of transient conduction heat transfer in foods packaged in flexible retort pouches. Can. Inst. Food Sci. Technol. J. 19(4): 148–157.
- NIX, G. H., G. W. LOWERY, R. I. VACHON, and G. E. TANGER. 1967. Direct determination of thermal diffusivity and conductivity with a refined line-source technique. Progress in aeronautics and astronautics. Thermophysics of Spacecraft and Planetary Bodies 20: 865–878, Academic Press, New York.
- SINGH, R. P. 1982. Thermal diffusivity in food processing. Food Technol. 36(2): 87–91.
- TIMBERS, G. E. and G. D. ROBERTSON. 1977. An inexpensive thermal conductivity probe for *in-situ* measurements with foods. Report No. 7404-614 of the Engineering and Statistical Research Centre, Research Branch, Agriculture Canada, Ottawa, Ont.