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A Computer-Controlled Transient Needle-Probe Thermal Conductivity Instrument for Liquids[1]

G. B. Asher,[2] E. D. Sloan,[2] and M. S. Graboski[3]

A computerized system utilizing the transient needle-probe technique has been developed for thermal conductivity measurements on solids and liquids. Thermal conductivities are determined to an accuracy of better than 5%. The instrument is unique in that it uses "off the shelf" components such as a personal computer and analog-to-digital conversion devices, together with software developed in our laboratory. The initial expense and time required to begin measurements are less than 20% of those for normal transient hot-wire measurements. Typical results are presented for liquid tertiary butyl alcohol, 1-methylnaphthalene, and glycerin.

KEY WORDS: Butyl alcohol; glycerin; 1-methylnapthalene; needle probe; thermal conductivity; transient method.

1. INTRODUCTION

A computerized system utilizing the transient needle-probe technique has been developed for thermal conductivity measurements on solids and liquids. Thermal conductivities are determined to an accuracy of better than 5%. The instrument is unique in that it uses "off the shelf" components such as a personal computer and analog-to-digital conversion devices, together with software developed in our laboratory. The initial expense and time required to begin measurements are less than 20% of those for normal transient hot-wire measurements.

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[1] Paper presented at the Ninth Symposium on Thermophysical Properties, June 24–27, 1985, Boulder, Colorado, U.S.A.

[2] Chemical Engineering and Petroleum Refining Department, Colorado School of Mines, Golden, Colorado 80401, U.S.A.

[3] SynGas Incorporated, Golden, Colorado 80401, U.S.A.

It is not intended that this thermal conductivity system replace the hot-wire technique but, rather, that it fill vacancies left by the hot wire. Excellent candidate situations for the needle-probe technique are (i) when the hot wire is difficult to employ due to physical constraints and (ii) when the accuracy of the hot wire (1%) is not necessary.

Previous workers have employed the needle probe on a variety of compounds, such as core samples, soils, deep-sea sediments, and laboratory specimens of solids and saturated solids [1–6]. These researchers determined thermal conductivities on a relative basis. The present work extends the application of the needle probe to include liquid compounds and absolute thermal conductivity measurements.

2. THERMAL CONDUCTIVITY NEEDLE PROBE

Thermal conductivities of solids and liquids are conveniently measured via the transient needle-probe technique. Time requirements are generally of the order of minutes or seconds for the measurement of solids or liquids, respectively. The probe is initially inserted into the media and thermal equilibrium is obtained. At time zero a power step is supplied to the probe, and the temperature rise of the probe is monitored with respect to time. A linear plot of the probe temperature rise versus the natural logarithm of time yields a slope which is inversely proportional to the media thermal conductivity.

2.1. Needle-Probe Design

The basic needle-probe design consists of a wire loop for heating and a thermistor placed at probe midlength for temperature measurement (Fig. 1). The heater wire and thermistor are mounted by epoxy cement inside a 20-gauge hypodermic needle. The probe length is approximately

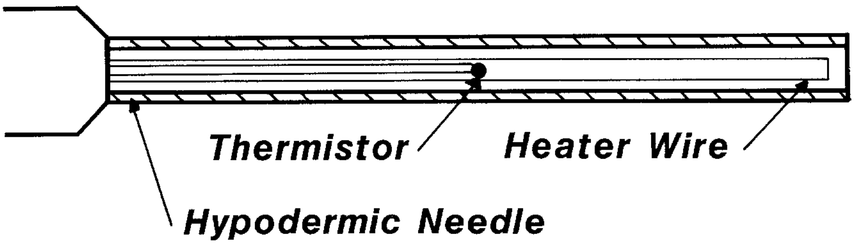

Fig. 1. Thermal conductivity needle probe.

35 mm and the probe diameter is 0.9 mm. The probes used in this work are of the same design as originally presented by Von Herzen and Maxwell [5] and are purchased from Fluid Dynamics Corporation, Boulder, Colorado.

The thermal conductivity system is comprised of hardware wherein experimental control, data acquisition, and data analysis are accomplished using a microcomputer under software control. The developmental computerized apparatus for conductivity measurements is presented in Fig. 2. A Keithley Series 500 is interfaced through an IBM Personal Computer. The Keithley Series 500 is a state-of-the-art analog/digital interfacing machine, and the IBM Personal Computer was selected because it is an industry standard.

2.2. Theory

The models used for the transient line source method of determining thermal conductivities are well established in the literature [7–10]. Past researchers used a model with several simplifying assumptions. These are the following: (i) the probe is infinitely long, (ii) the probe has an infinite thermal conductivity, (iii) all physical properties are constant, (iv) the medium is infinite in extent, and (v) energy is transferred by one-dimensional radial conduction, and there is no influence due to convection or radiation. With the above model simplifications, the thermal conductivity is calculated from the infinite-line source solution for long times [8].

$$T = (Q/4\pi k)\ln(4\alpha t/Ca^2) \tag{1}$$

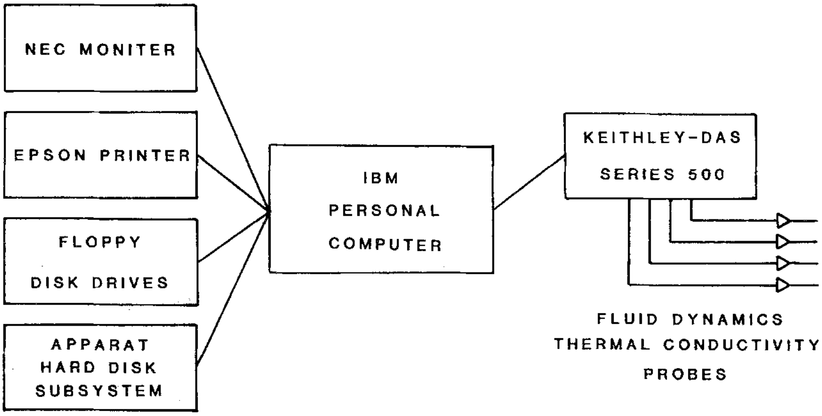

Fig. 2. Functional diagram of the thermal conductivity measuring system.

where T is the temperature rise at the probe–medium interface, Q is the power supplied per unit probe length, $\pi = 3.14159$, k is the medium thermal conductivity, α is the medium thermal diffusivity, t is the time from the start of heat input, $C = 1.7811$ (exponential of Euler's constant), and a is the radius of the probe.

The assumption that the probe exhibits only radial heat flow is incorrect because the probe is of finite length. By making the probe sufficiently long and measuring the temperature at probe midlength, the error caused by assuming radial heat flow can be made neglible within experimental accuracy. A minimum length-to-diameter ratio of 20 to 30 is given by Blackwell [7]. Through the theory presented by Kierkus et al. [10], it was determined that the long-time infinite line source was accurate to within 2% for the probe used in this work.

To fit further the model of Eq. (1), it must be determined when a sample finite in size resembles an infinite sample. From the thermal wave solution presented by Carslaw and Jaeger [8], the following was obtained:

$$r/a \gg 3(\alpha t/a^2)^{\frac{1}{2}} \tag{2}$$

where r is the radius of the sample and t is the longest measurement time encountered. This equation assumes that the temperature at the sample boundary is less than 1% of the probe surface temperature for all times. For the needle probe and samples used in this work, the radius of the sample is required to be much greater than 30 times the probe radius.

Convection in the media could occur before long times are reached. If this occurs, then corrections based on the media and probe volumetric heat capacity are necessary. At shorter times the model becomes [8].

$$T = (Q/4\pi k)\{\ln(4\alpha t/Ca^2) + a^2/2\alpha t[1 + (\omega - 2)/\omega \ln(4\alpha t/Ca^2)]\} \tag{3}$$

where ω is twice the ratio of the volumetric heat capacity of the media to the probe.

Pantaloni et al. [11] gave the following correlation for the onset of convection:

$$(\alpha t/a^2)^{\frac{2}{3}} \ln(4\alpha t/Ca^2) = (380\ C_p \mu \alpha^2)/(g a^3 Q \beta) \tag{4}$$

where t is the time for onset of convection, C_p is the liquid heat capacity, μ is the liquid viscosity, g is the acceleration of gravity, and β is the volume expansivity of the liquid. If the viscosity is large, such as in glycerin, then viscosity is the controlling factor. For smaller viscosity liquids, such as tertiary butyl alcohol and 1-methylnapthalene, it is apparent that other factors, such as thermal diffusivity, will also control convection. In this paper only viscosity is studied, because of doubt in the claim that only liquids as viscous as glicerin can be measured [4].

2.3. Data Reduction

Past needle-probe researchers overcame the difficulties of probe usage by performing a calibration experiment on a compound of well-defined properties. In this work, absolute thermal conductivities were determined directly from Eq. (1), with an accuracy within 5%.

The method used to analyze the data rests on predictions of the behavior of the probe temperature rise. A typical experimental response of the transient needle probe is given in Fig. 3. The curvature in the early time portion of the experimental response is caused by heat capacity effects. The curvature in the latter time portion is caused by axial end effects and/or convection. After time zero, the slope of the experimental temperature response increases with time until axial effects cause a decrease in slope at the time t_m shown in Fig. 3. Just prior to this time the experimental response is at its closest approximation to the long-time infinite-line source solution. After this time, the slope continually decreases until steady state is reached and the slope is zero. Maximum accuracy in the measurement is obtained by analyzing the data prior to time t_m.

Apparent thermal conductivities are calculated at several points along the experimental response curve, by employing Eq. (1). The thermal conductivities are termed apparent because it is assumed that the long-time infinite-line source solution is valid. From this set of apparent thermal con-

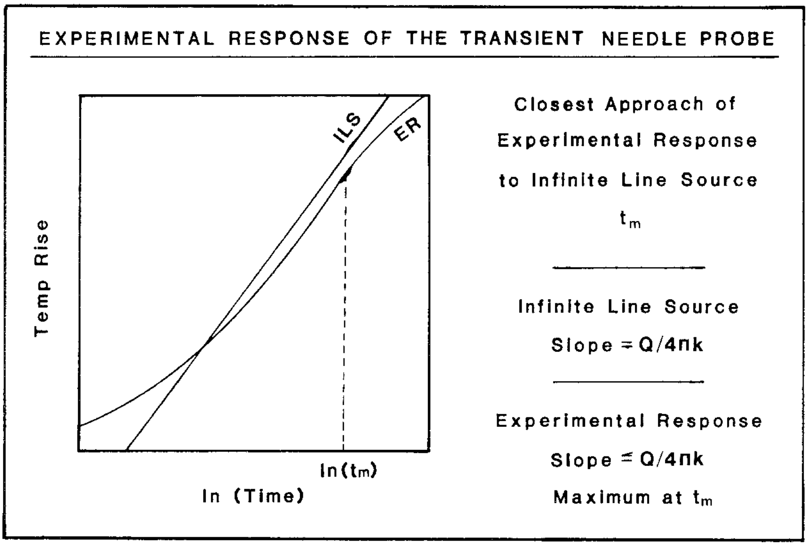

Fig. 3. Experimental response of the transient needle probe.

ductivities, the minimum apparent thermal conductivity and the corresponding time are determined. This minimum conductivity is designated the measurement conductivity, and this time is the time of closest approach to the long-time solution. The model and experimental points show that this $k_{\min}$ is the "most correct" value of k.

Figure 4 gives an example of an actual experiment on 1-methylnaphthalene. The temperature rise and apparent conductivity versus natural log of time are shown in Figs. 4a and b, respectively.

3. EXPERIMENTATION ON LIQUIDS

Three liquids, tertiary butyl alcohol, 1-methylnapthalene, and glycerin, were analyzed at room temperature using the thermal conductivity needle probe. No elaborate safeguards were taken to minimize the effect of liquid convection. A technique for building cells to minimize convection for the transient hot-wire technique is described by Graham [12] and Perkins [13]. A cell to minimize convection effects could also be built for the needle probe.

Table I summarizes the results of all liquid experiments. In this table k is the best estimate of the thermal conductivity, σ is the confidence in the data for that particular run, and N is the number of experiments performed. All thermal conductivities are reported at the temperature corresponding to half the experimental temperature increase (typically 1 to 2 K).

For the compounds presented, the corrections due to heat capacity effects (short time solution) are within the stated experimental accuracy. This result was determined by using the actual fluid properties and the theory for all times presented by Carslaw and Jaeger [8].

3.1. Tertiary Butyl Alcohol

Experiments were conducted on 99.9% pure liquid tertiary butyl alcohol (viscosity, 6 cP) at room temperature. A thermal conductivity of $0.110 \pm 0.006\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ at 301 K was determined, which is in excellent agreement with that of $0.109\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ reported by Perkins [13].

At first consideration of these raw data, it appears that the measured thermal conductivity varies with the heat supplied to the medium. Further analysis showed that this effect was due to convection instabilities. The occurrence of this phenomenon is delayed by decreasing the supplied heat. Therefore, the maximum confidence in the measured thermal conductivity occurs at the minimum heat input.

Heat inputs lower than those shown in Table I were not feasible. As

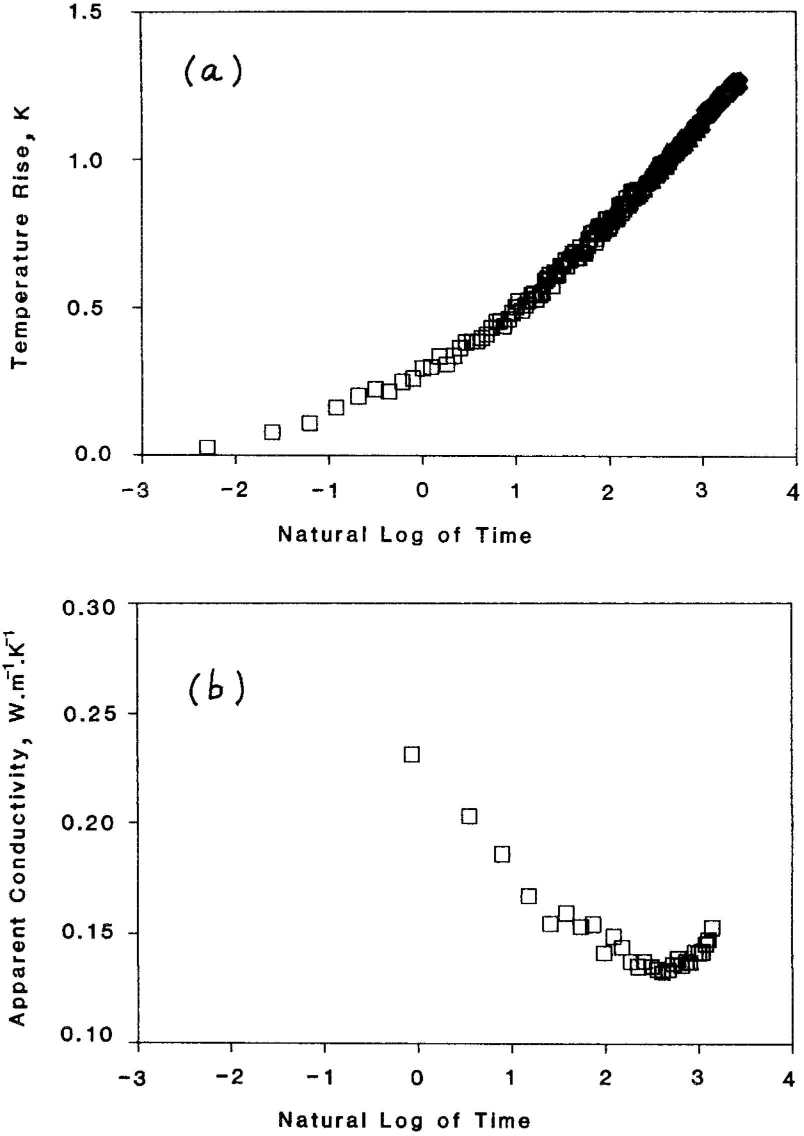

Fig. 4. Experimental temperature response and data analysis for 1-methylnaphthalene at 296 K and $Q = 0.6\ \mathrm{W \cdot m^{-1}}$. (a) Experimental temperature rise vs natural log of time and (b) apparent thermal conductivity vs natural log of time, where time is in seconds.

Table I. Thermal Conductivities of Liquids

	T(K)	N	k($\mathrm{W \cdot m^{-1} \cdot K^{-1}}$)	σ($\mathrm{W \cdot m^{-1} \cdot K^{-1}}$)	Q($\mathrm{W \cdot m^{-1}}$)
Tertiary butyl alcohol	300	7	0.122	0.006	0.8
	300	5	0.119	0.005	0.6
	300	3	0.113	0.005	0.5
	301	4	0.110	0.006	0.4
1-Methyl-naphthalene	297	4	0.135	0.002	1.0
	297	4	0.134	0.002	0.9
	297	2	0.136	0.001	0.8
	296	4	0.133	0.002	0.7
	296	4	0.136	0.003	0.6
Glycerin	301	2	0.292	0.005	1.1
	298	4	0.296	0.004	1.0
	298	4	0.287	0.005	0.9
	297	3	0.289	0.004	0.8

the heat input decreases, the temperature rise of the probe decreases. The relative discretization error in the temperature measurement then increases. This causes a large error associated with fitting the temperature rise versus log of time plot.

Both liquid convection and probe finite length have the same effect on the experimental temperature response in that both cause a temperature rollover. Experiments on solid tertiary butyl alcohol were performed in order to distinguish between convection effects and axial effects. For solid tertiary butyl alcohol a thermal conductivity of $0.122 \pm 0.006\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ was determined, which is in excellent agreement with a literature value of $0.123\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ [13].

As theoretically predicted by Kierkus et al. [10] and experimentally observed, the time when axial effects dominate is not related to the heat input to the probe. In liquids the onset of convection is directly related to the probe power input. Minimizing the power input lengthens the time period until convection, then the long-time infinite-line source solution yields a more accurate fit.

3.2. 1-Methylnaphthalene

Experiments were conducted on 1-methylnaphthalene (viscosity, 3 cP) at room temperature. A thermal conductivity of $0.135 \pm 0.002\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ at 296 K was determined, which is in excellent agreement with that of $0.134\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ reported by Graham [12]. Convection did not appear significant in any of the measurements.

3.3. Glycerin

Experiments were conducted on pure liquid glycerin (viscosity, 3800 cP) at room temperature. A thermal conductivity of $0.292 \pm 0.004\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ at 298 K was determined, which is in agreement with a literature value of $0.288\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ for pure glycerin [14].

Previous research [4] recommended using needle probes on liquids only as viscous as glycerin. In this research no convective instabilities occurred in glycerin for any level of power input. Experimental temperature increases were observed in excess of 2 K without causing convection. Temperature increases of this magnitude cause convection in tertiary butyl alcohol and 1-methylnaphthalene. Through accurate data acquisition and analysis it becomes possible to use the probe on liquids far less viscous than glycerin.

4. CONCLUSION

The needle probe can measure absolute thermal conductivities with an accuracy of better than 5%. For liquids of a viscosity greater than 3 cP at room temperature, it is possible to measure thermal conductivities without convective instabilities. The range of materials and temperatures the needle probe can test is expandable. Examples of such expansions are as follows.

(i) A cell to control convection would allow for liquids less viscous than 3 cP.

(ii) The upper limit of conductivity measurements could be increased. beyond that presented in this paper.

(iii) The development of a shorter-time solution is another alternative to augment the needle probe. Equation (1) can be expanded to include shorter times [8]. These expansions account for the early curvature in the experimental temperature rise versus natural log of time (Fig. 3).

Work is currently underway in the laboratory to effect the above improvements.

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