

# Thermal Conductivity of Electrically Conducting Liquids by the Transient Hot-Wire Method

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A relative transient hot-wire apparatus for the measurement of the thermal conductivity of electrically conducting liquids is described. The instrument consists of a single glass capillary filled with mercury to act as an insulated hot wire. The resistance change in the wire with respect to time is used to obtain the thermal conductivity of the liquid surrounding the wire with an estimated accuracy of  $\pm 2\%$ . The most significant advantage of the liquid metal filled glass capillary is its increased temperature limit compared to other types of transient hot-wire cells. The use of the new method at temperatures up to 493 K is shown for several aqueous systems, and new data are reported for propionic acid + water mixtures.

## Introduction

In recent years, the transient hot-wire technique has become the leading method for the measurement of the thermal conductivity of liquids. The use of this method for electrically conducting liquids, however, has revealed several limitations. One of these is the low-temperature limit of most of the methods developed for these liquids. A method is presented here which allows the thermal conductivity of electrically conducting liquids to be accurately measured at temperatures that are considerably higher than those practical with other methods.

The transient hot-wire method typically employs a bare metal wire immersed in the liquid of interest. The wire is subjected to a step voltage and allowed to heat, resulting in an increase in its resistance caused by the rising temperature. The thermal conductivity of the surrounding liquid is determined from this rise in resistance, using a model based on an infinitely long line source of heat in an infinite liquid medium. Corrections are applied to the model to account for the nonidealities of the apparatus. In addition, radiative heat transfer is kept small due to the nature of the apparatus, and convective heat transfer is avoided by monitoring any abnormalities in the temperature trends as the wire is heated.

When a bare metal wire is used in electrically conducting liquids, leakage of current and polarization of the fluid at the surface of the wire occurs. The resulting current is not easily determined, and thus the actual heat dissipation from the wire is not well known. In order for the transient hot-wire technique to be used for electrically conducting liquids, the wire must be insulated. A number of methods have been employed to insulate the wire. Nagasaka and Nagashima (17) used a platinum wire with a polyester coating to make measurements with a claimed accuracy of  $\pm 0.5\%$ . The coating, however, decomposed at temperatures above 150 °C.

Alloush *et al.* (2) used a tantalum wire anodized to have an electrically insulating tantalum oxide layer. To date, this has been the most widely used method to insulate the transient hot wire. Kawamata *et al.* (12) used the tantalum oxide coated wire, but noted that the electric potential of the wire must be maintained at a level above the ground level to protect the insulation layer from breakdown. They

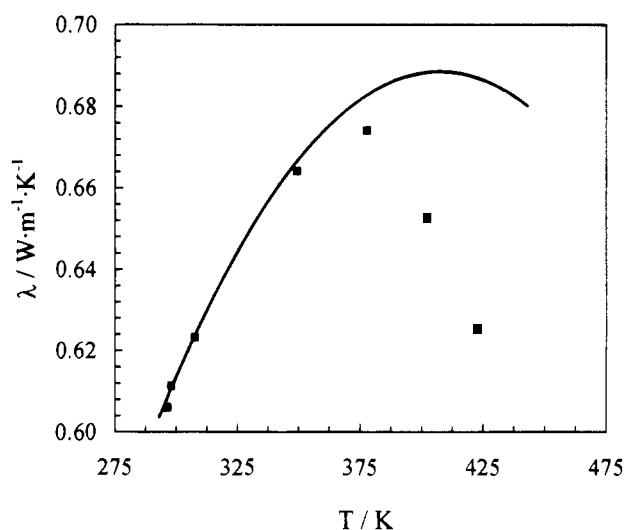


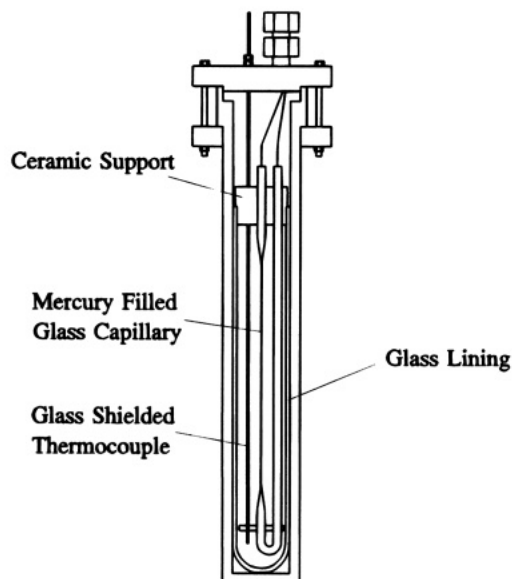
Figure 1. Measurement of the thermal conductivity of water using a tantalum oxide coated wire: (■) DiGuilio *et al.* (6), (—) Kestin and Whitelaw (14).

also noted that the oxide coating failed to properly insulate the wire above 100 °C. DiGuilio *et al.* (6) confirmed this temperature limitation while attempting to measure the thermal conductivity of water above 100 °C (Figure 1). Peterson (20) measured the thermal conductivity of acetic acid + water mixtures at temperatures up to 150 °C using the tantalum oxide coated wire. His data, however, differ by as much as 30% from data measured by other researchers (4), leading to the conclusion that his oxide layer was probably cracked at the higher temperatures due to differences in thermal expansion between the tantalum oxide layer and the metal wire. More recently, researchers (21, 22) have continued to successfully use the tantalum wire below the temperature limit to measure the thermal conductivity of electrically conducting liquids with a claimed accuracy of  $\pm 0.5\%$ .

In 1989, an aluminum oxide coated platinum wire was used to measure the thermal conductivity of molten salts with a claimed accuracy of  $\pm 3\%$  (15). However, although the aluminum oxide coating works at higher temperatures, it is difficult to prepare.

A thin glass capillary filled with a liquid metal such as mercury or gallium has also been employed as the hot wire

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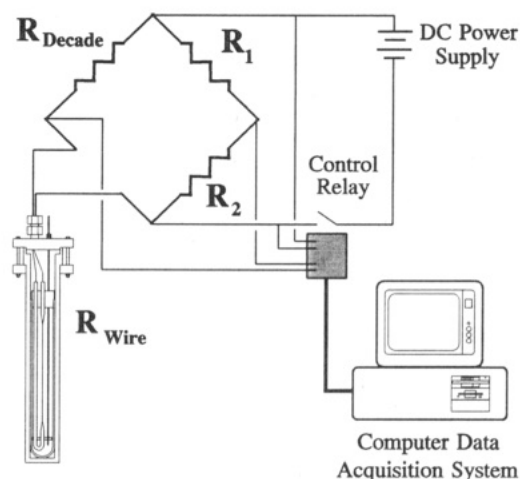
**Figure 2.** Transient hot-wire cell employing a mercury filled Pyrex capillary and pressure vessel.

for the measurement of the thermal conductivity of molten salts and aqueous electrolyte solutions by several researchers (3, 4, 6, 7, 10, 19). Two methods have been used to prepare the capillary. The first method was developed by Omotani *et al.* (19) who heated and stretched a section of glass tubing to form the capillary. The second method was introduced by DiGuilio and Teja (6, 7). They sealed a quartz capillary into a larger quartz support tube using high-temperature silicone rubber cement or quartz adhesive. Both methods present drawbacks in the design of the apparatus. The former method results in a capillary with nonuniform cross sectional area and capillary diameters that are difficult to reproduce. The seals used in the second method can limit the temperature range of the apparatus and can break the capillary due to differences in expansion with changing temperature. The glass capillary, however, allows measurements to be made at higher temperatures than is possible using a polyester layer or tantalum oxide coating and is easier to prepare than the aluminum oxide coating.

Our goal was to design an apparatus allowing measurements on electrically conducting liquids to be made at temperatures well above the 100–150 °C limit of the previous methods. We have chosen to use the transient hot-wire technique with a mercury-filled borosilicate glass capillary formed by the stretching of a larger piece of glass tubing. This apparatus allows measurements to be performed at elevated temperatures and is more readily fabricated than either the oxide coated platinum wire or the glass capillary sealed into a support.

### Experimental Apparatus

Figure 2 illustrates the liquid metal transient hot-wire cell used in this work. The cell was constructed by forming Pyrex tubing (4 mm o.d. × 2 mm i.d.) into a U tube and then heating and stretching one leg of the tube to form the capillary. The diameter of the resulting capillary was measured with a microscope. The average outer diameter was 75 μm, and the average inner diameter was 38 μm. The glass tubing was cemented into a ceramic support, and a vacuum pump was used to draw mercury through the capillary and fill the tubing. The assembled cell was placed in a glass-lined stainless steel pressure vessel which held the liquid to be measured. The temperature of the fluid was monitored by a 1.6 mm diameter glass-shielded Type



**Figure 3.** Wheatstone bridge circuit for the transient hot-wire cell.

E thermocouple calibrated against a standard platinum resistance thermometer (Leeds and Northrup SN No. 709892). Tungsten wire was used for the electrical leads to the mercury. To suppress boiling at elevated temperatures, nitrogen gas was used to pressurize the vessel. The temperature was controlled to ±0.1 K using a Techne constant temperature fluidized sand bath (model SBL-2D).

The other parts of the experimental apparatus are shown in Figure 3. The hot-wire cell was placed in one leg of a Wheatstone bridge with a General Radio (model 1433 U) decade resistor box adjustable to ±0.01 Ω and two equal ((100 ± 0.01) Ω) precision resistors in the remaining legs of the bridge. The resistance balance in the bridge was determined with a Fluke multimeter (model 8840A). A Hewlett-Packard (model 6213A) constant voltage supply was used to power the bridge for heating and balancing the bridge. A data acquisition system consisting of an IBM PC XT with a Strawberry Tree analog to digital converter card (ACPC-16) was used to control a relay (Magnecraft W172DIP-1) and to read both applied voltage and voltage offset of the bridge.

The following procedure was followed for each measurement. The Wheatstone bridge was balanced, and a BASIC program was run to operate the data acquisition system. The program initiated a step voltage to the control relay which closed the circuit to the bridge, thus applying a voltage from the power supply across the bridge. The resistance of the liquid metal filament changed because of heating, thereby producing a voltage offset across the bridge. The computer data acquisition system recorded the voltage offset on one channel and then switched to a second channel and recorded the voltage applied to the bridge by the constant voltage power supply. There was a 0.0084 s time delay between the sampling of each channel, giving 0.0168 s between two samplings of the same channel. Each run lasted about 3.4 s, and the computer recorded 200 sets of voltage readings in this time. Points measured between 0.84 and 2.52 s were used in determining the thermal conductivity. The temperature rise in the wire was determined from the resistance,  $R_w$ , of the wire calculated as a function of time as follows:

$$R_w(t) = \frac{V_a R_d (R_1 + R_2)}{R_1 V_a - V_o(t)(R_1 + R_2)} - R_d \quad (1)$$

where  $V_a$  is the applied voltage,  $V_o(t)$  is the voltage offset of the bridge at time  $t$  from the start of the run,  $R_d$  is the resistance of the decade box, and  $R_1$  and  $R_2$  are the values of the precision resistors.

The resistance of the wire is converted to a temperature rise as follows:

$$\Delta T_w(t) = \frac{R_w(t) - R_w(0)}{\kappa R_w(0)} = \frac{R_w(t) - R_d}{\kappa R_d}$$

$$\kappa = \frac{1}{\rho} \frac{d\rho}{dt} \quad (2)$$

where  $\kappa$  is the temperature coefficient of resistivity for mercury and  $\rho$  is the resistivity of mercury reported by Williams (30). The heat dissipated,  $q$ , per unit length of wire was determined by

$$q = \frac{V_a^2 R_{w,av}}{(R_d + R_{w,av})^2 L} \quad (3)$$

where  $L$  is the length of the wire. It should be noted that using the average wire resistance to determine the heat dissipation introduces no significant error. The typical resistance increase over the entire heating period is about 0.15  $\Omega$  or 0.2%. This relates to a change in  $q$  of under 0.00004%. The thermal conductivity of the fluid is determined from these measurements as described below.

### Mathematical Analysis of the Transient Hot-Wire Technique

In the transient hot-wire technique, the wire is modeled as an infinite line source of heat in an infinite fluid medium, using an expression for the temperature rise derived by Carslaw and Jaeger (5):

$$\Delta T_{id} = \frac{q}{4\pi\lambda} \ln(4\lambda t/r_w^2 \rho C_p C) \quad (4)$$

where  $\Delta T_{id}$  is the ideal temperature rise of the wire,  $q$  is the heat dissipation per unit length,  $\lambda$  is the thermal conductivity of the fluid,  $t$  is the time from the start of the heating,  $r_w$  is the radius of the wire,  $\rho$  and  $C_p$  are the density and heat capacity of the fluid, and  $C$  is the exponent of Euler's constant. If the physical properties are assumed to be constant over the temperature range, the thermal conductivity can be determined from the slope of the line  $\Delta T_{id}$  versus  $\ln(t)$  in eq 4. The temperature rise is typically less than 2 K, so the assumption of constant physical properties is valid. Equation 4 is valid when  $t \gg r_w^2/4\alpha$  where  $\alpha$  is the thermal diffusivity of the fluid (8). The inequality is satisfied within 0.1 s from the start of heating for the glass capillary in liquid water.

For eq 4 to be used, corrections must be made to the measured temperature rise in order to account for nonidealities of the experimental apparatus. Healy *et al.* (8) have expressed these corrections as follows:

$$\Delta T_{id} = \Delta T_w(t) + \sum_i \delta T_i \quad (5)$$

where  $\Delta T_w$  is the measured temperature rise of the wire and  $\delta T_i$  are corrections to the temperature rise.

The first temperature correction accounts for the finite physical properties of the wire and the temperature correction due to the insulating layer. Nagasaka and Nagashima (17) derived the following expression for this correction:

$$\delta T_1 = \frac{-q}{4\pi\lambda} \left[ \ln\left(\frac{r_w}{r_1}\right)^2 + \frac{2\lambda}{\lambda_1} \ln \frac{r_1}{r_w} + \frac{\lambda}{\lambda_1} + \frac{1}{t} \left( C_1 + C_2 + C_3 \ln\left(\frac{4\alpha t}{r_1^2 C}\right) \right) \right]$$

$$C_1 = \frac{r_w^2}{8} \left[ \left( \frac{\lambda - \lambda_1}{\lambda_w} \right) \left( \frac{1}{\alpha_w} - \frac{1}{\alpha_1} \right) + \frac{4}{\alpha_1} - \frac{2}{\alpha_w} \right] \quad (6)$$

$$C_2 = \frac{r_1^2}{2} \left( \frac{1}{\alpha} - \frac{1}{\alpha_1} \right) + \frac{r_w^2}{\lambda_1} \left( \frac{\lambda_1}{\alpha_1} - \frac{\lambda_w}{\alpha_w} \right) \ln\left(\frac{r_1}{r_w}\right)$$

$$C_3 = \frac{r_w^2}{2\lambda} \left( \frac{\lambda_1}{\alpha_1} - \frac{\lambda_w}{\alpha_w} \right) + \frac{r_1^2}{2\lambda} \left( \frac{\lambda}{\alpha} - \frac{\lambda_w}{\alpha_w} \right)$$

The subscript w refers to the properties of the wire (mercury thread), and the subscript l refers to the properties of the insulating layer (Pyrex). Properties with no subscripts refer to the fluid being studied. The heat capacity and density of mercury were obtained from the CRC Handbook (30), and thermal conductivity was obtained from Ho *et al.* (9). The heat capacity and thermal conductivity of borosilicate glass were obtained from the compilation by the Thermophysical Properties Research Center (24, 25).

To account for the finite extent of the fluid, Healy *et al.* (8) derived

$$\delta T_2 = \frac{q}{4\pi\lambda} \left( \ln\left(\frac{4\alpha t}{b^2 C}\right) + \sum_{\nu=1}^{\infty} \exp\left(\frac{-g_\nu^2 \alpha t}{b^2}\right) [\pi Y_0(g_\nu)]^2 \right) \quad (7)$$

where  $b$  is the inside diameter of the cell,  $Y_0$  is the zero-order Bessel function of the second kind,  $g_\nu$  are the roots of  $J_0$ , the zero-order Bessel function of the first kind. Values of  $Y_0$  can be calculated from the approximation given by Abramowitz and Stegun (1). The first several roots of  $J_0$  are available, and the higher roots can be determined with sufficient accuracy by the expression given by Watson (28).

The final temperature correction accounts for heat loss due to radiation. Nieto de Castro *et al.* (18) derived the following expression for radiation during the heating of the wire:

$$\Delta T = \frac{q}{4\pi\lambda} \left( 1 + \frac{Br_w^2}{4\alpha} \right) \ln \frac{4\alpha t}{r_w^2 C} + \frac{Bqr_w^2}{16\pi\alpha\lambda} - \frac{Bqt}{4\pi\lambda} \quad (8)$$

where  $B$  is a radiation parameter and is used in the following expression also derived by Nieto de Castro *et al.* (18):

$$\delta T_3 = \frac{-qB}{4\pi\lambda} \left( \frac{r_w^2}{4\alpha} \ln \frac{4\alpha t}{r_w^2 C} + \frac{r_w^2}{4\alpha} - t \right) \quad (9)$$

$B$  is determined using eq 8 after the first two temperature corrections are made to the measured temperature rise in the wire. Equation 9 can then be used to determine the final temperature correction.

Two other effects must be considered in determining thermal conductivity by the transient hot-wire technique. First, the model described above accounts for all the heat being dissipated radially from the wire, but ignores a small amount of heat conducted axially through the ends of the filament. No analytical correction is available to compensate for this heat loss. The second effect is caused by the

method of preparing the glass capillary (heating and stretching a piece of tubing) which results in a nonuniform cross sectional area of the wire.

Two methods have been used to experimentally eliminate the effects of axial heat transfer (8): (a) the potential lead method and (b) the two-wire method. In method a, the resistance change during heating is determined for the central portion of the heating wire via potential lead wires connected a short distance from the ends of the fine heating wire. However, it has been difficult to precisely determine how the lead wires affect the measurements. Also, it has not been possible to design this type of apparatus using a glass capillary filled with liquid metal. Method b employs a second short line source of heat that is wired into the Wheatstone bridge circuit in the leg with the decade resistor box. End effects are then present in two legs of the bridge and therefore offset each other. The apparatus is then modeled as though one wire were present with a length equal to the difference in length between the long and short wires. When using method b, it is important that heating be approximately equal in both wires so that axial heat transfer is similar in both. Thus, it is necessary for both wires to have similar radii. Kestin and Wakeham (13) proposed a method to mathematically account for two wires with slightly different radii. DiGuilio and Teja (7) successfully designed an apparatus using two liquid metal filled glass capillaries with a diameter tolerance of  $\pm 10\%$ . As stated earlier, the main drawback of this approach was the sealing of the capillaries. However, it is very difficult to heat and stretch glass tubing to result in a long capillary and a short capillary both with diameters of such close tolerance. Capillaries resulting from heating and stretching a larger diameter piece of glass are generally not used for the two-wire apparatus due to large differences in radii. Therefore, another method is used to account for axial heat transfer.

To account for both the end effects and the nonuniform cross sectional area of a single capillary, the cell is calibrated using a reference fluid. IUPAC (16) has suggested several reference fluids where the thermal conductivity is known as a function of temperature. The "effective" length of the liquid wire is backed out of the calculations by using the reference value of the thermal conductivity. For aqueous electrolyte solutions, water was chosen as the reference fluid. The effective length was determined at six temperatures to determine any change with respect to temperature. The average length at these temperatures was  $9.205 \text{ cm} \pm 0.19\%$ , so the length was assumed to be independent of temperature. It is important to note that the relative transient hot-wire method (single calibrated wire) results in an estimated accuracy at least twice as large as that obtained in the absolute method (two wires). The accuracy of the relative measurement is estimated to be  $\pm 2\%$  (1% for the calibration and 1% for the actual measurement).

## Results and Discussion

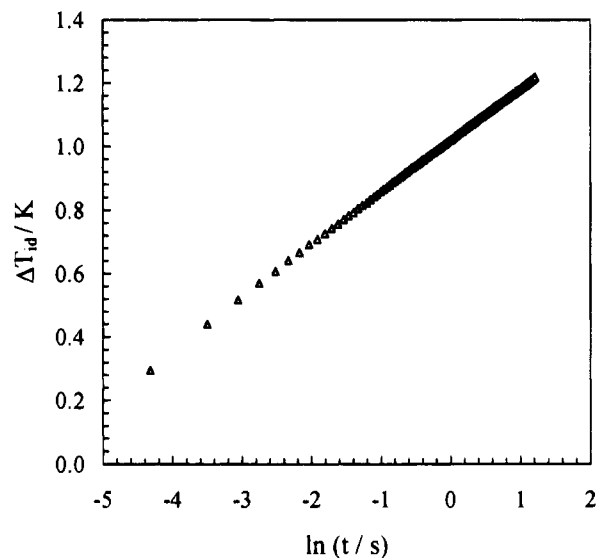
Table 1 contains the thermal conductivity of dimethyl phthalate at seven temperatures and comparisons between the measured values using the average wire length and the values as suggested by IUPAC. No temperature dependence is observed for the determination of the effective length of the wire. Figure 4 shows a typical  $\Delta T_{id}$  vs  $\ln(t)$  plot for the liquid metal wire. Figure 5 shows the deviation from the linear fit for the same  $\Delta T_{id}$  vs  $\ln(t)$ , indicating no bias.

The three temperature corrections to the physical model were evaluated to determine the significance of each

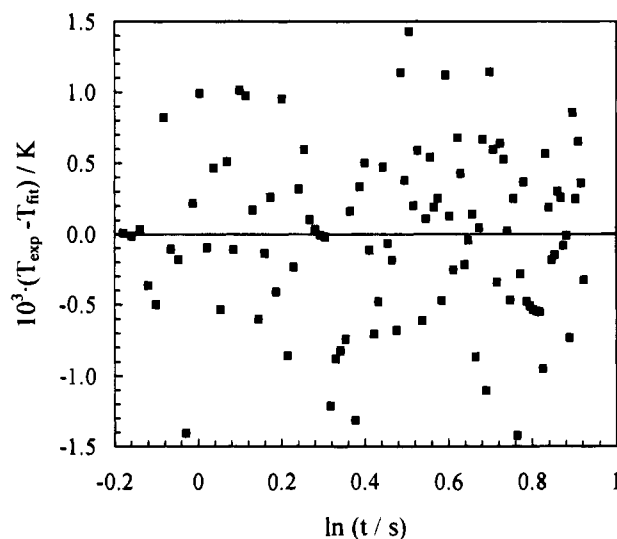
**Table 1. Thermal Conductivity of Dimethylphthalate Measured Using a Mercury Filled Pyrex Capillary**

$T/K$	$\lambda/(W \cdot m^{-1} \cdot K^{-1})$	$\lambda^a/(W \cdot m^{-1} \cdot K^{-1})$	dev/%
292.0	0.1468	0.1481	-0.855
323.9	0.1431	0.1442	-0.760
353.3	0.1396	0.1402	-0.436
383.2	0.1358	0.1358	0.053
412.6	0.1309	0.1309	0.045
443.4	0.1254	0.1254	-0.053
468.0	0.1215	0.1207	0.624

<sup>a</sup> IUPAC recommendation (16).

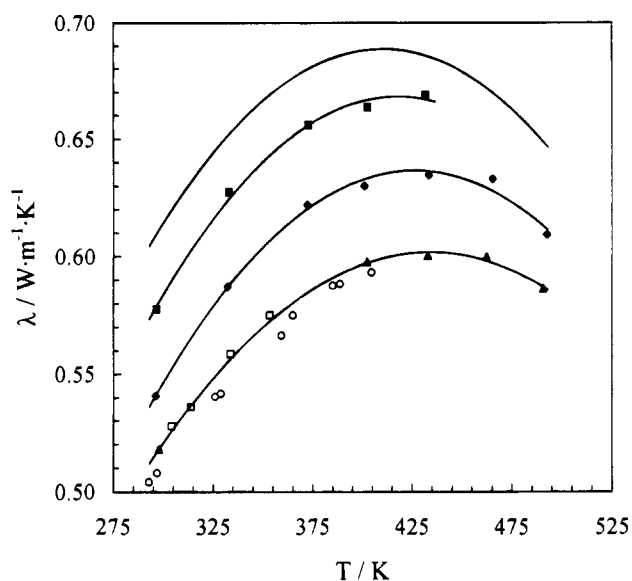


**Figure 4.** Ideal temperature rise as a function of time for the liquid metal filled glass capillary.



**Figure 5.** Deviation plot of the experimentally measured temperature rise relative to a linear fit of the data for  $t$  between 0.84 and 2.52 s.

correction. The correction for the finite physical properties of the wire and the glass insulation layer,  $\delta T_1$ , was determined to vary between 10% and 14% of the temperature rise in the wire due largely to the temperature drop across the insulation layer. This correction adds a significant offset to the temperature rise measured, but only slightly affects the slope of the  $\Delta T_{id}$  vs  $\ln(t)$  and thus the thermal conductivity. The corrections for the finite extent of the fluid,  $\delta T_2$ , and heat transfer due to radiation,  $\delta T_3$ , were determined to be less than 0.0002% and 0.003%, respectively. Though these corrections were insignificant



**Figure 6.** Measured thermal conductivity of aqueous LiBr solutions. Solid curves are from a least squares fit of the data: (■) 10.0, (◆) 20.4, and (▲) 29.8 mass % LiBr, Bleazard *et al.* (3); (○) 30.2 mass % LiBr, DiGuilio and Teja (7); (□) 30.3 mass % LiBr, Kawamata *et al.* (12); (—) water, Kestin and Whitelaw (14).

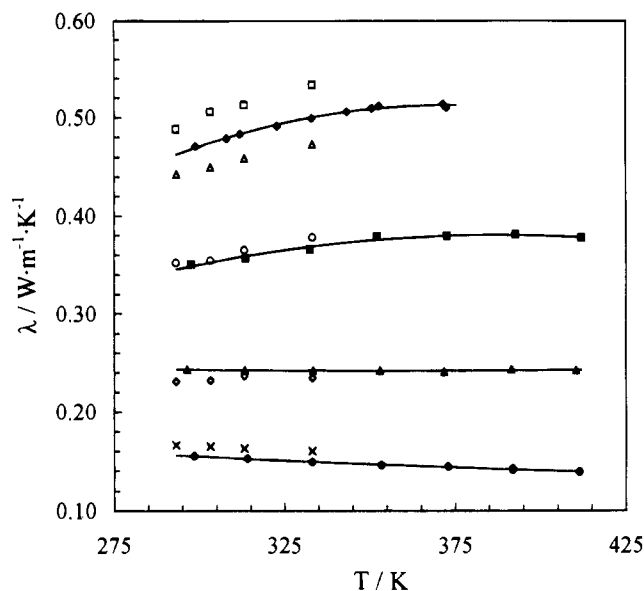
**Table 2. Thermal Conductivity of Propionic Acid + Water**

[acid]/ (mass %)	T/K	$\lambda/(W \cdot m^{-1} \cdot K^{-1})$	[acid]/ (mass %)	T/K	$\lambda/(W \cdot m^{-1} \cdot K^{-1})$
25	294.3	0.4557	75	294.0	0.2214
	294.3	0.4614		323.0	0.2225
	322.8	0.4862		342.2	0.2216
	342.4	0.4940		362.9	0.2201
	362.1	0.5032		365.3	0.2216
				405.2	0.2177
50	294.8	0.3285	100	294.7	0.1434
	322.6	0.3374		323.1	0.1387
	342.5	0.3424		343.3	0.1350
	362.3	0.3426		362.2	0.1327
	382.0	0.3496		387.4	0.1291
	405.8	0.3511			

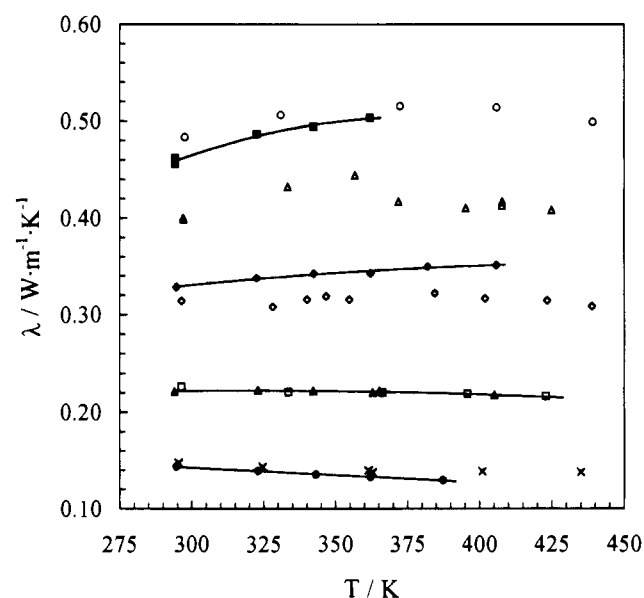
with respect to  $\delta T_1$ , they were still used in the determination of thermal conductivity for consistency.

The relative transient hot-wire technique was used to measure the thermal conductivity of LiBr solutions from 293 to 493 K. Solutions of 10.0, 20.4, and 29.8 mass % salt were measured and are reported elsewhere (3). Other researchers have reported values for thermal conductivity of LiBr–water solutions generally over 25 mass % salt and up to 100 °C (2, 6, 12, 23, 26). Figure 6 displays the data available for constant composition solutions of less than 31 mass % measured over a temperature range. This study agrees within experimental error with the other measurements and extends the available data to lower concentrations and higher temperature.

The thermal conductivity of acetic acid + water mixtures and propionic acid + water mixtures was also measured using the single glass capillary cell. Mixtures of 25, 50, 75, and 100 mass % acid were measured from 293 to 413 K. Values for acetic acid are presented elsewhere (4) while those for propionic acid are reported in Table 2. Peterson (20) and Usmanov (27) report the only other available data for acetic acid + water mixtures where thermal conductivity was measured over a temperature range. Though direct comparison of all the data is not possible due to measurements being reported at different acid compositions, our work generally agrees with that of Usmanov (27) within



**Figure 7.** Measured thermal conductivity of acetic acid + water mixtures and a least squares fit of the data: (◆) 25.0, (■) 50.0, (▲) 75.0, and (●) 100 mass % acetic acid, Bleazard and Teja (4); (□) 10.0, (△) 20.0, (○) 50.0, (◇) 80.0, and (×) 100 mass % acetic acid, Usmanov (27).



**Figure 8.** Measured thermal conductivity of propionic acid + water mixtures and a least squares fit of the data: (■) 25.0, (◆) 50.0, (▲) 75.0, and (●) 100 mass % propionic acid, this work; (○) 19.04, (△) 32.05, (◇) 50.89, (□) 73.46, and (×) 100 mass % propionic acid, Peterson (20).

experimental error (Figure 7). Differences of as much as 20% are seen with the data reported by Peterson (20). Data for the pure acids are reported by several researchers (11, 20, 27, 28). For pure acetic acid, our data differ by up to 7% with the data of Usmanov (27), while agreeing within experimental error with the data of Peterson (20). Peterson (20) is the only other researcher to report the thermal conductivity of propionic acid + water mixtures over a temperature range. Figure 8 shows a graph of the available data for the propionic acid + water systems. Our data differ by as much as 10% with the values reported by Peterson (29). However, our data appear to be more consistent with respect to temperature and composition trends than the data of Peterson.

## Conclusions

The transient hot-wire apparatus employing a single glass capillary filled with a liquid metal has been used for measuring the thermal conductivity of electrically conducting liquids at high temperatures. The apparatus has an estimated accuracy of  $\pm 2\%$  and has been used to measure thermal conductivity up to temperatures of 493 K. Similar cells incorporating quartz capillaries have been used to measure the thermal conductivity of molten salts to 590 K (7). Although other insulated hot-wire methods are capable of higher accuracy, the liquid metal filled glass capillary has been shown to be more versatile and can be used over a broader range of conditions.

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