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Thermal Conductivity of Poly(ethylene glycols) and Their Binary Mixtures

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A transient hot-wire method was used to measure the thermal conductivities of the first six members of the poly(ethylene glycol) series at 1 atm and in the temperature range 295–480 K. The method was used on a relative basis, and the accuracy of the measurements is estimated to be ±2%. The thermal conductivities of the binary systems ethylene glycol + tri(ethylene glycol), di(ethylene glycol) + tri(ethylene glycol), and di(ethylene glycol) + hexa(ethylene glycol) were also measured over the whole composition range at 1 atm and 295–480 K. The pure liquid data were correlated with use of a carbon number approach, and a linear mixing rule was used to extend the correlation to mixtures of poly(ethylene glycols). Agreement between prediction and experiment was within experimental error.

1. Introduction

The poly(ethylene glycols) ($HOCH_2CH_2(OCH_2CH_2)_nOH$) are industrially important chemicals that have found widespread application in the manufacture of explosives and as antifreezes, solvents, hygroscopic agents, plasticizers, lubricants, and conditioning agents. The large variety of uses for these glycols makes it necessary to have reliable physical-properties data for these substances. Vapor pressures of the glycols up to tetra(ethylene glycol) and molecular weights up to hepta(ethylene glycol) have been reported by Gallaugher and Hilbert (*1*, *2*). Densities, viscosities, and thermal conductivities of the lower glycols have been studied by a number of workers (*3*–*5*). The present work reports thermal conductivities of six glycols and three binary mixtures of glycols. The thermal conductivity of the pure liquids is interesting in that it exhibits a maximum when plotted against the temperature. Additionally, the glycols are known to be thermally unstable at the higher temperatures studied in this work (*1*). This provides an interesting challenge in the development of correlations for the thermal conductivity.

2. Apparatus and Procedure

The transient hot-wire apparatus employed in this work is shown in Figure 1 and consisted of a Wheatstone bridge, a power supply, and a data acquisition system. The Wheatstone bridge consisted of two 100 ± 0.01 Ω precision resistors, a General Radio (Model 1433 U) decade box with a range of 0–111.11 Ω, and a hot-wire cell. The hot-wire cell was fabricated from 0.01-m-diameter Pyrex (7740) tubing with a 0.002 m inside diameter glass sleeve at each end. The sleeves act to center two 0.001-m-diameter platinum lead wires. The lead wires protruded beyond the sleeves on the inside and outside of the cell and were sealed into place by melting the ends of the sleeves. A platinum filament 0.000025 m in diameter (Englehard; 99.9%) was spot-welded between the leads inside the cell. The length of the filament was 0.09995 ± 0.0005 m as determined with a cathetometer. On the outside of the cell, silver wires were soldered to each lead and, in turn, connected to an amphenol plug. The plug was directly attached to the bridge. The cell itself was attached to a 0.125-in. thermocouple to ensure that it remained vertical. The cell and thermocouple were then inserted into a stainless steel enclosure as shown in Figure 2.

The bridge was powered by a Hewlett-Packard (Model 6213A) power supply used as a constant voltage source. The supply was used to both balance the bridge and provide the voltage for heating. A Fluke multimeter (Model 8840A) was used to indicate a balanced condition in the bridge. A data acquisition system consisting of an IBM PC XT with a Strawberry Tree analog-to-digital (A/D) converter card (ACPC-16) was used to read both the offset voltage and the applied voltage.

The test fluid was loaded into the glass cell mounted in a stainless steel housing. The whole assembly was then placed in a Techne fluidized sand bath (Model SBL-2D) that maintained the temperature to ±0.1 K. A type K thermocouple, calibrated against a PRT, was used to determine the stability of the bath and the sample temperatures. After temperature equilibrium had been achieved, the air flow to the sand bath was stopped to prevent any vibration of the cell during measurement.

The procedure for each measurement was as follows. The bridge was first balanced and the computer program started. The program initiated a step input to the bridge with a relay (Magnecraft W172DIP-1). The relay settled in less than 0.3 ms. The program sampled the offset voltage on one channel and then switched channels to sample the applied voltage to ensure its constancy. The time between any two samples was 0.0084 s and that between two successive readings of the same channel was 0.0168 s. The delay between the closing of the relay and the first sampling was found experimentally to be 0.0132 s. There were 50 points measured during each run, and the experiment lasted about 0.9 s. From a previous calibration of the temperature versus resistance, the temperature of the wire was found. A plot of ΔT versus ln t was made, and the slope in the time interval from 0.1 to 0.8 s was calculated by

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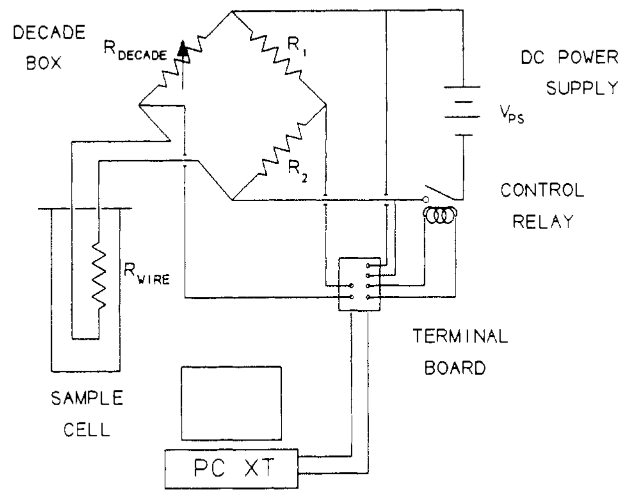

Figure 1. Schematic diagram of the apparatus.

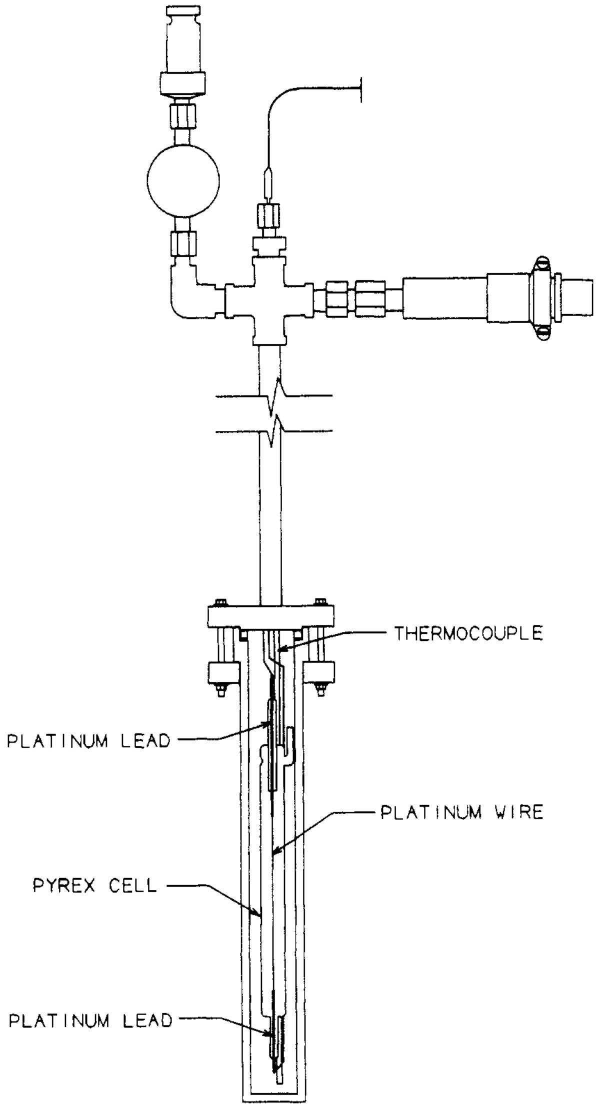

Figure 2. Hot-wire cell and pressure assembly.

a least-squares fit. The applied voltage was varied from about 2.5 to 3.5 V so that a more or less constant temperature rise in the wire of about 2.5 K was achieved. This resulted in offset voltages on the order of 5 mV. The A/D card has 16-bit resolution, and the ±25 mV range was used. Thus, the card is capable of 0.8-μV resolution.

At least five runs were made at each temperature.

3. Analysis

The ideal temperature rise of the wire was calculated from an expression derived by Carslaw and Jaegar (*6*) and Healy et al. (*7*) for $t \gg a^2/5\kappa$, which is satisfied for 10 ms < t < 100 ms. The expression is

$$\Delta T_{id} = \frac{q}{4\pi\lambda} \ln\left(\frac{4\lambda t}{a^2 \rho C_p C}\right) \tag{1}$$

where q is the heat dissipation per unit length, λ is the thermal conductivity, ρ is the density, C_p is the heat capacity, a is the radius of the filament, and C is equal to exp(γ) where γ is Euler's constant. If it is assumed that all physical properties (including the thermal diffusivity $\lambda/\rho C_p$) are independent of temperature over the small range of temperature considered (ca. 2.0 K), then

$$\lambda = \frac{q}{4\pi\left(\frac{\mathrm{d}\Delta T_{id}}{\mathrm{d}\ln t}\right)} \tag{2}$$

where $\mathrm{d}\Delta T_{id}/(\mathrm{d}\ln t)$ is found experimentally from a plot of ΔT_{id} vs ln t.

Healy et al. (*7*) also derived several corrections for the deviation of the model from reality. These may be written as

$$\Delta T_{id} = \Delta T\omega(t) + \sum_i \delta T_i \tag{3}$$

δT_1 accounts for the finite physical properties of the wire and is given by (*7*)

$$\delta T_1 = \frac{a^2[(\rho C_p)_\omega - (\rho C_p)]}{2\lambda t} \Delta T_{id} - \frac{q}{4\pi\lambda}\frac{a^2}{4\kappa t}\left(2 - \frac{\kappa}{\kappa_\omega}\right) \tag{4}$$

where $(\rho C_p)_\omega$ is the volumetric heat capacity of the wire and κ and κ_ω are the thermal diffusivity of the fluid and platinum, respectively.

The correction due to the finite extent of the fluid is given by (*7*)

$$\delta T_2 = \frac{q}{4\pi\lambda}\left(\ln \frac{4\kappa t}{b^2 C} + \sum_{\nu=1}^{\infty} \exp^{-g_\nu^2 \kappa t/b^2}[\pi Y_0(g_\nu)]^2\right) \tag{5}$$

where b is the inside diameter of the cell, Y_0 is the zero-order Bessel function of the second kind, and g_ν are the roots of J_0, the zero-order Bessel function of the first kind. Although the first several roots are readily available, the higher roots can be found to sufficient accuracy from (*8*)

$$g_\nu = (\pi\nu - \pi/4) + \frac{1}{8(\pi\nu - \pi/4)} - \frac{31}{385(\pi\nu - \pi/4)^3} + \frac{3779}{15366(\pi\nu - \pi/4)^5} \tag{6}$$

Values of Y_0 were calculated with the polynomial approximation given by Abramowitz and Stegun (*9*).

Radiation by the fluid can be accounted for with an analytical expression for the temperature rise of the wire given by Wakeham et al. (*10*):

$$\Delta T = \frac{q}{4\pi\lambda}\left(1 + \frac{Ba^2}{4\kappa}\right)\ln\frac{4\kappa t}{a^2 C} + \frac{Bqa^2}{16\pi\kappa\lambda} - \frac{Bqt}{4\pi\lambda} \tag{7}$$

where B is the radiation parameter and is a measure of the contribution of radiant emission by the fluid to the heat-transfer process. From eq 7, Wakeham et al. (*10*) derived the following expression for the correction to the observed temperature rise:

$$\delta T_{\mathrm{rad}} = \frac{-qB}{4\pi\lambda}\left(\frac{a^2}{4\kappa}\ln\frac{4\kappa t}{a^2C} + \frac{a^2}{4\kappa} - t\right) \qquad (8)$$

They used eq 7 to show that emission from a fluid causes the ΔT vs ln t slope to exhibit a slight curvature, concave to the ln t axis.

As suggested by Wakeham et al. (*10*), ΔT, after being corrected for the other effects mentioned, can be fit to eq 7 to obtain B. Then, eq 8 can be used to calculate δT_{rad}. If there is no radiation contribution, B = zero, and thus, there is no danger of biasing the data.

Two additional sources of error must be considered, the slackening of the wire and end effects. Ordinarily, a spring is used to compensate for the expansion of the platinum filament with temperature. Our cell does not incorporate such a feature and, because the glass expands to a small extent with temperature (0.054% (*11*) over 200 K vs 0.096% (*12*) for platinum), wire slackening must be accounted for in the calculations. However, no analytical solution exists for this source of error. End effects result from conduction of heat away from the wire along the thicker leads upon heating the filament. No analytical correction exists for this source of error either. It is generally accounted for experimentally with either potential leads or two wires (a long and a short wire) (*13*). Only a single wire was used in this work. However, since these two sources of errors would only be weak functions of the fluid in the cell, a relative method can be used to compensate for their effects, that is, to calculate a "cell constant" that allows for these and other effects. A relative method using a standard reference fluid was used in our work. The obvious choice for the reference fluid is toluene for which accurate values of the thermal conductivity are available. However, the thermal conductivity of toluene has not been satisfactorily determined over the whole range of temperatures used in this work. IUPAC (*14*) has suggested the use of dimethyl phthalate as a reference fluid over the range 273–493 K. This was therefore chosen as the reference fluid in the present work.

Thermal conductivities, corrected for the above effects, are reported in the results section. The temperatures reported are the average temperatures of the fluid during the heating process. That is,

$$T_{\mathrm{R}} = T_0 + \frac{\Delta T(t_{\mathrm{I}}) + \Delta T(t_{\mathrm{F}})}{2} \qquad (9)$$

where T_0 is the temperature of the fluid at the start of a measurement and t_{I} and t_{F} refer to the initial and final times of the data used to find the slope of ΔT vs ln t.

In order to apply the temperature corrections, various physical properties were required. The thermal diffusivity and conductivity of platinum were taken from the values recommended by the Thermophysical Properties Research Center (*15, 16*). The values for specific heat were taken from Kendall et al. (*17*). For dimethyl phthalate, the density was taken from Sverbely et al. (*18*). For toluene, the density and heat capacity were taken from Shulga et al. (*19*). For the glycols, all densities were calculated with the correlation of Tawfik and Teja (*3*). The heat capacity of ethylene glycol was taken from the values recommended by the Thermophysical Properties Research Center (*20*). The heat capacities of di(ethylene glycol) and tri(ethylene glycol) were taken from the ACS Monograph on glycols (*21*). For the higher poly(ethylene glycols), the heat capacities were estimated with the measured values of thermal diffusivity. As the corrections affect the thermal conductivity by less than 0.5%, the increase in error due to errors in the physical properties is small. The radiation parameter B for all fluids measured here was found to be negligible (less than 0.0007). Nevertheless, the correction was uniformly applied for consistency.

Table I. Calibration Constants Obtained from the Thermal Conductivity of Dimethyl Phthalate

T, K	λ_S, mW/(m K)	λ_E,[b] mW/(m K)	λ_S/λ_E
299.9	147.1	148.7	0.98924
333.3	143.0	145.2	0.98485
379.1	136.4	139.9	0.97498
410.8	131.4	135.3	0.97118
452.0	124.1	129.3	0.95978
479.3	118.9	124.2	0.95733

[a] From IUPAC (*14*). [b] This work, uncorrected.

Table II. Comparison of the Thermal Conductivity of Toluene

	λ, mW/(m K)		
T, K	IUPAC (*14*)	this work	% dev
298.8	130.9	131.5	0.46
324.4	123.2	123.8	0.49
343.2	117.6	117.5	0.09
363.0	111.6	112.2	0.54

4. Source and Purity of Materials

Pure fluids were used as received. Di(ethylene glycol) (99%), tetra(ethylene glycol) (99%), penta(ethylene glycol) (95%), hexa(ethylene glycol) (98%), and dimethyl phthalate (99+%) were obtained from the Aldrich Chemical Co. Toluene (HPLC Grade), ethylene glycol (99.8%), and tri(ethylene glycol) (99.5%) were obtained from Fisher Scientific Co. The binary mixtures were prepared by weight.

5. Calibration

Table I shows the cell constant calculated from the uncorrected thermal conductivities measured in the present study and the values recommended by IUPAC (*14*). At each temperature, the ratio of the IUPAC value (λ_S) to our uncorrected value of thermal conductivity (λ_E) was calculated and fit to the linear relationship

$$\lambda_S/\lambda_E = 1.046925 - (1.88761 \times 10^{-4})T \qquad (10)$$

where T is in kelvin. The average percent deviation for the fit was 0.1%, and the maximum value was 0.2%. The cell constant obtained in this way was then used to obtain the thermal conductivity of other liquids. In order to validate the (relative) method, values of the thermal conductivity of toluene were obtained with the above method and compared with values recommended by IUPAC (*14*). The results are listed in Table II where it can be seen that the maximum deviation between the two sets of values was 0.54% at T = 363 K, which is well within the accuracy of the experiment.

6. Results

All data points presented here represent the average value of five experimental runs. The maximum deviation from the average value never exceeded 0.24%. Thus, the precision of the data is 0.24%. We estimate the accuracy to be ±2.0%, based on the comparisons discussed below.

Table III shows the thermal conductivities of the first six poly(ethylene glycols) measured in this work. The thermal conductivity–temperature behavior is nonlinear and exhibits a maximum in the thermal conductivity. Also, the thermal conductivity decreases with the size of the glycol. Since the glycols have been reported to be thermally unstable at the temperatures studied in this work (*1*), it was initially believed that the nonlinear behavior with temperature was caused by thermal decomposition. However, the behavior could be reproduced with both increasing and decreasing temperature. It may therefore be concluded that nonlinear behavior is typical of glycols and not a result of decomposition. Data for ethylene

Table III. Experimental Thermal Conductivity of the Glycols

glycol	T, K	λ, mW/ (m K)	glycol	T, K	λ, mW/ (m K)
ethylene glycol	298.6	254.1	tetra(ethylene glycol)	296.5	188.2
	333.7	256.9		335.5	187.1
	378.0	257.4		379.0	184.2
	412.9	257.3		410.6	181.6
	424.3	257.0		451.6	175.6
	452.1	250.5	penta(ethylene glycol)	296.6	186.9
	471.3	244.4		334.8	185.3
di(ethylene glycol)	299.1	201.2		384.0	182.0
	334.8	203.7		410.2	177.9
	379.3	203.1	hexa(ethylene glycol)	300.3	186.0
	410.4	202.3		335.1	184.2
	444.5	197.5		379.3	181.4
	480.6	189.9		411.3	178.3
tri(ethylene glycol)	299.8	193.2		451.5	172.9
	335.6	193.2		480.6	167.4
	371.2	191.1			
	404.3	188.2			
	444.0	183.4			
	476.2	177.9			

Table IV. Experimental Thermal Conductivity of Ethylene Glycol (1) + Tri(ethylene glycol) (2) Mixtures

mole fraction of 1	T, K	λ, mW/ (m K)	mole fraction of 1	T, K	λ, mW/ (m K)
0.2053	296.5	199.8	0.7997	297.0	228.7
	336.2	198.4		335.6	230.2
	379.0	197.2		377.0	230.0
	407.6	195.2		410.3	229.4
	451.5	190.2		447.9	224.3
	480.7	184.8		474.7	215.7
0.4109	296.3	205.0	0.9007	296.7	239.1
	333.2	205.3		336.2	241.2
	376.6	203.9		378.4	241.7
	411.1	203.0		407.1	241.5
0.6020	297.0	215.3		450.9	235.9
	337.4	214.6		478.3	226.9
	377.4	213.8			
	407.8	212.9			
	451.9	205.4			
	478.0	203.5			

Table V. Experimental Thermal Conductivity of Di(ethylene glycol) (1) + Tri(ethylene glycol) (2) Mixtures

mole fraction of 1	T, K	λ, mW/ (m K)	mole fraction of 1	T, K	λ, mW/ (m K)
0.2008	297.5	194.9	0.6004	296.2	196.8
	335.6	195.2		333.1	198.0
	376.7	193.2		377.6	196.2
	410.7	191.2		409.7	194.3
	450.2	186.7		446.6	188.7
	479.9	180.2		474.9	184.9
0.4005	295.8	196.0	0.7992	294.7	199.1
	335.2	196.3		334.4	201.2
	375.1	194.7		375.0	200.1
	406.1	192.6		411.8	198.4
	452.7	186.7		443.3	194.7
	482.1	181.7		479.7	189.1

glycol, di(ethylene glycol), and tri(ethylene glycol) are compared with data obtained by Fischer with a concentric cylinder apparatus (*4*) in Figure 3. Fischer claimed that his data are of calibration quality. The maximum deviation between his data and the data obtained in this work was found to be less than ±2%. Therefore, we believe that the data in the present work are accurate to ±2%.

Tables IV–VI show results for the binary mixtures ethylene glycol + tri(ethylene glycol), di(ethylene glycol) + tri(ethylene glycol), and di(ethylene glycol) + hexa(ethylene glycol). The di(ethylene glycol) + tri(ethylene glycol) data are also shown in Figure 4. The regularity in behavior that is typical of mixtures of homologues is clearly evident from Tables IV–VI and Figure 4.

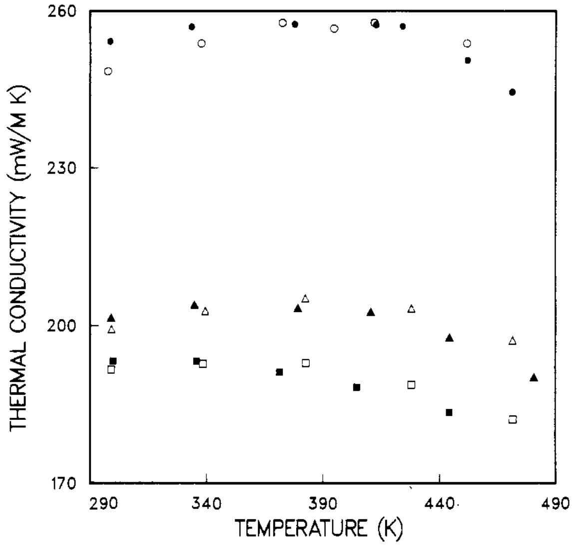

Figure 3. Comparison of the thermal conductivity of first three glycols with the measurements of Fischer (*4*). Ethylene glycol: (●) this work, (○) Fischer. Di(ethylene glycol): (▲) this work, (△) Fischer. Tri(ethylene glycol): (■) this work, (□) Fischer.

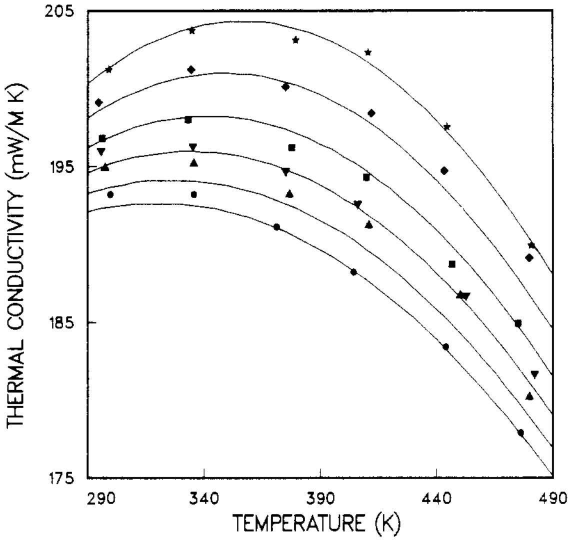

Figure 4. Thermal conductivity of binary mixtures of di(ethylene glycol) and tri(ethylene glycol): (★) 100%, (♦) 80%, (■) 60%, (▼) 40%, (▲) 20%, and (●) 0 mol % di(ethylene glycol). Solid lines represent predictions for mixtures.

Table VI. Experimental Thermal Conductivity of Di(ethylene glycol) (1) + Hexa(ethylene glycol) (2) Mixtures

mole fraction of 1	T, K	λ, mW/ (m K)	mole fraction of 1	T, K	λ, mW/ (m K)
0.2511	299.4	187.2	0.4958 (cont.)	451.6	179.5
	333.3	185.8		480.6	174.6
	378.6	183.5	0.7478	299.2	194.1
	412.4	180.6		334.0	192.8
	451.4	175.3		379.7	192.3
0.4958	297.4	189.7		412.2	190.7
	334.7	188.6		451.6	185.9
	379.5	186.9		481.4	175.0
	412.9	184.3			

7. Correlation

The number of carbon atoms has been used as a parameter for correlating physical properties of homologous series for

Table VII. Constants for the Carbon Number Correlation (Equation 13)

$a_1 = 400.687$
$a_2 = -236.599$
$b_1 = 2.48906$
$b_2 = 0.180542$
$c_1 = 2.90878 \times 10^{-3}$
$c_2 = -3.27281 \times 10^{-3}$
$d = 2.40286$

many years. For example, Wada et al. (*23*) recently correlated the thermal conductivities of the n-alkanes as follows:

$$\lambda \times 10^3 = (100.9 + 4.806n - 0.189n^2) - (0.1469 + 0.6124n + 2.974/n^2)t \quad (11)$$

where λ is in W/(m K) and t is in °C. n is the number of carbon atoms in the n-alkane. Teja and Tardieu (*24*) recently showed that this correlation could be used to predict the thermal conductivity of higher alkanes not included in the original correlation. They also extended the method to mixtures of alkanes.

In a similar vein, the thermal conductivity of the homologous series of glycols was correlated as follows:

$$\lambda = A(n) + B(n)T + C(n)T^2 \quad (12)$$

with

$$A(n) = a_1(1 - \exp(-n/d)) + a_2$$

$$B(n) = b_1 \exp(-n/d) + b_2$$

$$C(n) = c_1(1 - \exp(-n/d)) + c_2$$

where λ is in mW/(M K) and T is in K. The values of the constants a_1, a_2, a_3, b_1, b_2, c_1, c_2, and d were obtained by regression of the data and are given in Table VII. The first member of the series, ethylene glycol, was excluded from the regression because of anomalous behavior typical of first members of homologous series. The average absolute deviation between correlation and experiment was found to be 0.2% for 27 data points, and the maximum deviation was found to be 0.4%. On the basis of the work of Teja and Tardieu (*24*) on the alkanes, it is believed that the correlation for the glycols can be extrapolated to the higher members of the poly(ethylene glycol) series.

The correlation developed for pure substances can be extended to mixtures by the principle of congruence proposed by BrØnsted and Koefoed (*25*). This principle states that a mixture of chain molecules from a given homologous series may be characterized by an index τ such that mixtures with the same index have the same values of certain properties at a given temperature and pressure. For mixtures of n-alkanes, the index τ has usually been defined with the average number of carbon atoms n_m given by

$$n_m = \sum x_i n_i \quad (13)$$

where x_i is the mole fraction of component i in the mixture and n_i is the number of carbon atoms in a molecule of i.

In this work, the average number of carbon atoms n_m was used in eq 12 to *predict* the properties of glycol mixtures. The results are presented in Table VIII. The calculated and experimental results are in excellent agreement, in spite of the simplicity of the "mixing rule" (eq 13).

8. Conclusions

A relative hot-wire method was used to obtain the thermal conductivities of the first six poly(ethylene glycols) in the temperature range 295–480 K. Thermal conductivities of mixtures of ethylene + tri(ethylene glycol), di(ethylene glycol) + tri(ethylene glycol), and di(ethylene glycol) + hexa(ethylene glycol) over the entire composition range were also measured.

Table VIII. Comparison of Experimental and Predicted Thermal Conductivities of Glycol Mixtures

binary system	points	AAD%	MAD%
di(ethylene glycol) (1) + tri(ethylene glycol) (2)	24	0.4	1.3
di(ethylene glycol) (1) + hexa(ethylene glycol) (2)	17	1.1	2.0

A correlation for the thermal conductivity of the homologous series of glycols as a function of carbon number was derived. The first member of the series, ethylene glycol, was not included in the correlation as it exhibited the usual anomalous behavior for the first member of a homologous series. It was shown that the correlation can be extended to mixtures of glycols using the principle of congruence.

Registry No. Dimethyl phthalate, 131-11-3; toluene, 108-88-3; tri(ethylene glycol), 112-27-6; di(ethylene glycol), 111-46-6; ethylene glycol, 107-21-1; tetra(ethylene glycol), 112-60-7; penta(ethylene glycol), 4792-15-8; hexa(ethylene glycol), 2615-15-8.

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