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The Thermal Conductivity and Viscosity of Acetic Acid–Water Mixtures[1]

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The viscosity and thermal conductivity of acetic acid–water mixtures were measured over the entire composition range and at temperatures ranging from 293 to 453 K. Viscosity measurements were performed with a high-pressure viscometer and thermal conductivity was measured using a modified transient hot-wire technique. A mercury filled glass capillary was used as the insulated hot wire in the measurements. The viscosity data showed unusual trends with respect to composition. At a given temperature, the viscosity was seen to increase with increasing acid concentration, attain a maximum, and then decrease. The thermal conductivity, on the other hand, decreased monotonically with acid concentration. A generalized corresponding-states principle using water and acetic acid as the reference fluids was used to predict both viscosity and thermal conductivity with considerable succes.

KEY WORDS: acetic acid; aqueous mixtures; thermal conductivity; transient hot-wire technique; transport properties; viscosity.

1. INTRODUCTION

Acetic acid is found in many manufacturing processes as both a product and a precursor for such products as acetate plastics, acetic anhydride, ester solvents, and aspirin. Very often water is also present during the manufacturing process. A knowledge of the thermophysical properties of acid–water mixtures is therefore vital in the design of process equipment. Data for these systems are generally scarce and often contradictory. As an example, the two sources of thermal conductivity data over a range of concentrations and temperatures [1,2] differ by over 20% and also disagree in

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their temperature dependence. The purpose of the present work is to determine the viscosity and thermal conductivity of acetic acid–water mixtures over the entire composition range and an extended temperature range and resolve some of the contradictions in the literature data for this system.

2. EXPERIMENTS

Reagent-grade acetic acid from Fisher Scientific Company with a specified purity of 99.7 mol% was used in the experiments without further purification. Acetic acid–water mixtures were prepared gravimetrically using double-distilled water.

2.1. Viscosity

Viscosities were measured using a capillary viscometer placed in a pressure vessel designed for pressures up to 3 MPa and temperatures up to 473 K. According to Poiseuille's law, for a specific capillary viscometer, the kinematic viscosity ν can be related to the efflux time using

$$\nu = \frac{\eta}{\rho} = C_1 t - \frac{C_2}{t} \tag{1}$$

where η is the absolute viscosity, and ρ the density, C_1 and C_2 are viscometric constants, and t is the efflux time. Density was also measured in our laboratory and is presented elsewhere [3]. An appropiately sized viscometer was chosen, making it possible to neglect the kinetic energy correction C_2/t in Eq. (1). A size 1 Zeitfuchs cross-arm capillary viscometer (International Research Glassware) was used in our experiments. The high-pressure cell was equipped with glass view ports to allow visual observation of the liquid in the viscometer. An insulated air bath was used to establish the desired temperature. The mass of the heavy steel pressure cell helped to stabilize the temperature of the viscometer. Excess solution of the acetic acid–water mixture was placed in the pressure vessel outside the viscometer to assure no composition change of the mixture in the viscometer due to vaporization into the headspace. Temperature was measured inside the cell by a calibrated thermocouple with an accuracy of ±0.1 K. To suppress boiling, the cell was pressurized using helium. Further details of the apparatus and procedure can be found in Ref. 4. The error of the measured viscosity is estimated to be ±2% based on a measurement reproducibility of ±1% and a calibration fluid uncertainty of ±1%.

Errors introduced by the solubility of the pressurizing gas were determined to be negligible from a study of the viscosity of water in the presence

Table I. Comparison of the Viscosity of Water in the Presence of a Gas at 0.7 MPa with Literature Values for Pure Water [5]

Gas	T (K)	η_{exp} (cP)	η_{lit} (cP)	$\delta\eta/\eta_{\text{lit}}$ (%)
He	299.3	0.860	0.8670	−0.81
	313.2	0.653	0.6514	0.24
	326.1	0.519	0.5181	0.17
	339.1	0.421	0.4240	−0.71
	353.0	0.352	0.3512	0.23
	369.0	0.290	0.2914	−0.48
	383.4	0.249	0.2515	−0.99
	397.3	0.219	0.2218	−1.25
N_2	303.2	0.791	0.7965	−0.69
	318.4	0.596	0.5917	0.72
	333.0	0.459	0.4641	−1.10
	346.1	0.387	0.3842	0.72
	363.3	0.311	0.3103	0.21
	378.3	0.265	0.2643	0.25
	393.6	0.225	0.2290	−1.75

of two gases with different solubilities, helium and nitrogen. Table I contains a comparison between the viscosity of water reported in the literature [5] and the viscosity measured in the presence of each gas. All measured values are within the estimated ±2% experimental error and show no significant trends with respect to gas solubility.

2.2. Thermal Conductivity

The transient hot-wire method was used to measure the thermal conductivity. This technique typically employs a thin electrically heated metal wire. However, the wire must be insulated for electrically conducting fluids. A pyrex capillary filled with liquid mercury was used as the insulated metal wire in this work, and the technique was validated with aqueous LiBr solutions in earlier work in our laboratory [6,7]. The transient hot-wire cell was prepared by forming a U-shaped tube from pyrex (4-mm OD × 2-mm ID) and heating and stretching one of the legs of the U tube to form a thin capillary. The resulting capillary had an average outer diameter of 75 μm and an average inner diameter of 38 μm (±10% for both). A stainless-steel pressure vessel, pressurized with nitrogen gas to suppress boiling, was used to house the hot-wire cell during the experiments. The other components of the apparatus were a computer data acquisition system, a Wheatstone bridge, a constant-voltage DC power supply, and a constant-temperature bath. The four legs of the Wheatstone bridge consisted of the mercury thread, two precision resistors of equal value, and a decade resistance box adjustable to $\pm 0.01\ \Omega$.

At the beginning of the experiment, the cell and the pressure vessel were filled with the fluid being studied and the assembly was placed in the constant temperature bath. The temperature in the cell was constant within ±0.1 K and was monitored by a 1.6-mm-diameter Type E thermocouple calibrated against a standard platinium resistance thermometer. After the equilibrium temperature had been reached in the cell, the Wheatstone bridge was balanced by adjusting the decade resistor. The computer data acquisition system closed a relay which allowed the DC power supply to place a voltage across the bridge and heat the mercury filament. The resistance of the filament changes during heating and causes a voltage offset across the bridge. The computer data acquisition system recorded the voltage offset, as well as the voltage applied to the bridge by the constant-voltage power supply. About 200 voltage readings were recorded by the computer in about 3.4 s during each run. The relay was then opened and the hot-wire cell again allowed to return to the equilibrium temperature. Temperature rise in the wire was held under 2 K and was determined from the increased resistance of the wire. Further details of the experimental method are reported elsewhere [8].

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 Jaegar [9]:

$$\Delta T_{\mathrm{id}} = \frac{q}{4\pi\lambda} \ln\left(\frac{4\lambda t}{r_{\mathrm{w}}^2 \rho C_{\mathrm{p}} C}\right) \tag{2}$$

where ΔT_{id} is the ideal temperature rise of the wire, q is the heat dissipation per unit length, λ 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, ρ 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 ΔT_{id} versus $\ln(t)$ in Eq. (2). The temperature rise is typically less than 2 K, so the assumption of constant physical properties is valid. Equation (2) is a first-order approximation of a series expansion and is valid when $t \gg r_{\mathrm{w}}^2/4\alpha$, where α is the thermal diffusivity of the fluid [10]. Healy et al. [10] calculated the truncation error as follows:

$$\frac{\delta T}{\Delta T} = \frac{r_{\mathrm{w}}^2}{4\alpha t \ln(4\alpha t/r_{\mathrm{w}}^2 C)} \tag{3}$$

In the present work, the thermal conductivity was determined from the slope of the ΔT vs $\ln(t)$ line for times between 0.84 and 2.52 s from the start of heating. In the case of water, this corresponds to a ratio $\xi = r_w^2/4\alpha t$ of between 0.0028 and 0.00095, or a truncation error of between 0.054 and 0.015%. The truncation error compares favorably to recent measurements made on water and toluene reported by Ramires et al. [11]. The data in Ref. 11 are generally considered as reference quality data with an estimated accuracy of ±0.5%. Ramires et al. used a 25-μm-diameter wire and heated the wire for less than 1 s. The truncation error in that work is between 0.035 and 0.0035% for water and between 0.066 and 0.0064% for toluene.

For Eq. (2) to be used, corrections must be made to the measured temperature rise in order to account for nonidealities of the experimental apparatus. Expressions have been determined to account for the physical properties of the wire, the effect of the insulating glass layer, the finite extent of the fluid, and heat transfer due to radiation. Corrections for each of these are outlined in Ref. 8.

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 temperature correction is available to compensate for this heat transfer. 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. To account for these two effects, the cell was calibrated using the IUPAC suggested value for the thermal conductivity of water [12]. The "effective" length of the liquid wire was backed out of the calculations using the reference value of the thermal conductivity for water. Temperature effects were estimated by determing the "effective" length at 298 and 330 K. The average length at these two temperatures was 9.87 cm ±0.1%. To check the calibrated wire length, the thermal conductivity of a second IUPAC reference liquid, dimethylphthalate (DMP), was measured. DMP was chosen because of the large temperature range for which reference values of thermal conductivity are available and because the thermal conductivity differs greatly from that of the calibration fluid, water (the thermal conductivity of DMP is roughly one-fourth that of water). Table II contains the measured values and comparisons with the IUPAC reference values between 290 and 470 K. Each value presented in the table is an average of five replicates with a precision of ±1%. All measured values are within the estimated ±2% experimental error over the entire temperature range. This shows that the length of the wire is temperature independent.

Table II. The Thermal Conductivity of Dimethylphthalate

T (K)	λ_{exp} ($W \cdot m^{-1} \cdot K^{-1}$)	λ_{IUPAC} ($W \cdot m^{-1} \cdot K^{-1}$)	$\delta\lambda/\lambda_{IUPAC}$ (%)
292.0	0.1468	0.1481	−0.86
323.9	0.1431	0.1442	−0.76
353.3	0.1396	0.1402	−0.44
383.2	0.1358	0.1358	0.05
412.6	0.1309	0.1309	0.04
443.5	0.1254	0.1254	−0.05
468.0	0.1215	0.1207	0.62

The thermal conductivity of water was also measured while separately pressurizing the vessel with nitrogen and helium, as was done for viscosity. Table III shows the measured values compared with the reference values reported by IUPAC [12]. Again, errors are within the estimated ±2% experimental error and show no trends with respect to gas solubility or temperature.

3. RESULTS AND DISCUSSION

Table IV lists the experimental viscosities of acetic acid–water mixtures containing 12, 25, 50, 75, 84, 92, and 100 wt% acetic acid. The temperature range of the measurements varied from 293 to 460 K, and the

Table III. Comparison of the Thermal Conductivity of Water in the Presence of a Gas at 0.38 MPa with IUPAC Values for Pure Water [12]

Gas	T (K)	λ_{exp} ($W \cdot m^{-1} \cdot K^{-1}$)	λ_{IUPAC} ($W \cdot m^{-1} \cdot K^{-1}$)	$\delta\lambda/\lambda_{IUPAC}$ (%)
He	293.1	0.6005	0.5978	0.45
	312.8	0.6294	0.6290	0.07
	323.0	0.6418	0.6423	−0.08
	333.2	0.6538	0.6533	0.07
	343.2	0.6617	0.6623	−0.09
N_2	302.4	0.6093	0.6135	−0.68
	312.6	0.6353	0.6287	1.04
	322.2	0.6384	0.6413	−0.45
	333.0	0.6518	0.6532	−0.22
	372.7	0.6611	0.6619	−0.12
	352.1	0.6688	0.6686	0.02

Table IV. Experimental Viscosity of Acetic Acid–Water Mixtures

Wt% acid	T (K)	η (cP)
12	294.05	1.188
	308.30	0.879
	323.35	0.668
	338.25	0.517
	353.20	0.418
	368.05	0.347
	378.05	0.312
	383.15	0.291
	394.45	0.266
	405.25	0.241
	413.45	0.227
	424.15	0.207
	433.45	0.194
	444.35	0.181
	452.95	0.171
25	296.15	1.509
	303.75	1.247
	312.65	1.024
	322.35	0.852
	332.65	0.709
	342.60	0.613
	352.25	0.535
	362.15	0.464
	372.30	0.408
	393.35	0.317
	411.00	0.272
	430.00	0.231
	450.15	0.210
50	298.95	1.803
	307.35	1.446
	316.60	1.194
	325.40	0.995
	333.55	0.868
	342.50	0.752
	352.35	0.655
	362.15	0.574
	372.95	0.512
	385.95	0.440
	407.50	0.343
	426.75	0.285
	447.55	0.244

Table IV. *(Continued)*

Wt % acid	T (K)	η (cP)
75	297.60	2.341
	307.40	1.822
	316.80	1.496
	326.20	1.237
	334.60	1.059
	343.30	0.922
	353.30	0.800
	363.20	0.675
	372.95	0.595
	379.30	0.554
	386.00	0.479
	405.60	0.408
	425.40	0.317
	445.65	0.269
	459.95	0.241
84	295.25	2.381
	308.40	1.726
	323.40	1.276
	338.20	0.986
	353.15	0.792
	368.10	0.660
	378.35	0.581
	383.15	0.549
	396.55	0.460
	408.55	0.412
	420.75	0.366
	436.25	0.309
	449.45	0.277
92	295.35	1.991
	313.48	1.356
	328.40	1.042
	343.30	0.833
	358.25	0.686
	373.35	0.570
	379.35	0.518
	391.85	0.458
	401.95	0.412
	411.45	0.376
	420.65	0.345
	431.05	0.317
	439.70	0.298
	452.95	0.267

Table IV. *(Continued)*

Wt% acid	T (K)	η (cP)
100	296.20	1.136
	313.40	0.887
	328.30	0.744
	343.40	0.634
	358.15	0.549
	373.35	0.482
	377.25	0.457
	388.30	0.425
	402.83	0.370
	416.40	0.326
	431.39	0.289
	445.46	0.252
	452.45	0.239

pressure was maintained from 0.2 to 1 MPa in order to suppress boiling. The average of at least three measurements was taken to obtain each value reported in Table IV. The reproducibility of the viscosity was found to be ±1% and the error of the data was estimated to be ±2%. The viscosity of acetic acid–water solutions at each concentration can be described by the following equation:

$$\begin{aligned}
&\ln \eta = A_0 + A_1/T + A_2 \ln T + A_3 T \\
&A_0 = -95.9146 - 458.953x + 354.539x^2 + 10.0992x^3 - 4.28456x^4 \\
&A_1 = 5143.74 + 15714.8x - 14001.7x^2 \qquad (4) \\
&A_2 = 14.44 + 77.753x - 59.6015x^2 \\
&A_3 = -0.012456 - 0.105398x - 0.0706552x^2
\end{aligned}$$

where η is the viscosity (cP), T is the temperature (K), and x is the mole fraction of acetic acid. The absolute average deviation (AAD) was found to be 2.0% and the maximum absolute deviation (MAD) was found to be 6.9%. Figure 1 shows a graphical representation of the measured data and the fit using the above equation. At a given temperature, the viscosity attains a maximum value with respect to composition for concentrations of acetic acid between 75 and 85 wt%. The maximum is most pronounced at lower temperatures. This same type of composition dependence was noted during measurements of the density acetic acid–water mixtures in our laboratory [3]. The viscosities calculated from the correlation are in good agreement with the data reported by Melzer et al. [13] except at high concentrations.

The measured thermal conductivities of 25, 50, 75, and 100 wt% acetic acid mixtures are shown in Table V. Five measurements were performed at each temperature and the average of the five are listed in the table. Boiling was supressed by maintaining pressures up to 1 MPa. Error of the measured data is estimated to be ±2%. Our data agreed within experimental uncertainty with the data of Usmanov [2]. Temperature trends agreed

Table V. Experimental Thermal Conductivity of Acetic Acid–Water Mixtures

Wt% acid	T (K)	λ ($W \cdot m^{-1} \cdot K^{-1}$)
25	298.8	0.4709
	307.9	0.4785
	311.9	0.4834
	322.8	0.4913
	333.0	0.4993
	343.3	0.5061
	350.6	0.5094
	352.7	0.5115
	372.5	0.5103
	371.6	0.5138
50	297.5	0.3507
	313.5	0.3567
	332.5	0.3659
	352.1	0.3785
	372.6	0.3792
	392.7	0.3807
	411.9	0.3770
75	296.3	0.2432
	313.2	0.2426
	333.4	0.2424
	352.9	0.2422
	371.8	0.2410
	391.5	0.2434
	410.4	0.2423
100	298.4	0.1551
	314.0	0.1524
	333.2	0.1491
	353.4	0.1459
	373.0	0.1445
	392.0	0.1424
	392.0	0.1413
	411.4	0.1390

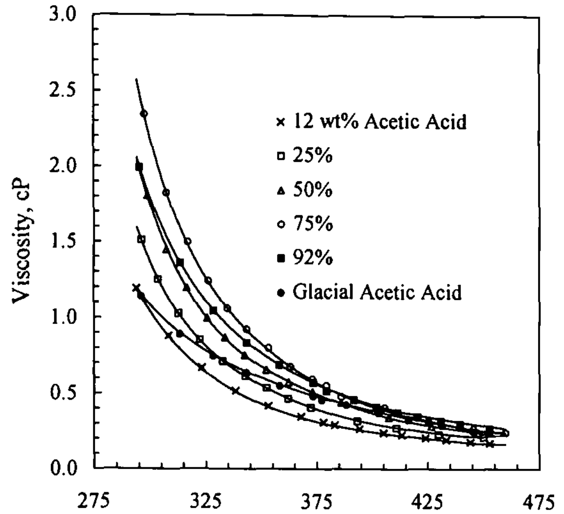

Fig. 1. Measured viscosity of acetic acid–water mixtures as a function of temperature and concentration. Solid lines are from Eq. (4).

with those reported by Usmanov (reported to 313 K). The thermal conductivity showed no maxima with respect to acid concentration as was observed for both density and viscosity.

4. GENERALIZED CORRESPONDING-STATES METHOD

Available estimation methods for the thermophysical properties of liquids are seldom adequate when applied to aqueous solutions because of strong hydrogen bonds that are typically present in these mixtures. In contrast, considerable success has been achieved by the application of the three-parameter corresponding-states method to nonpolar mixtures. The generalized corresponding-states principle (GCSP) proposed by Teja et al. [14] uses two real nonspherical reference fluids to predict the thermophysical properties of liquids and liquid mixtures. Reference fluids are not limited to spherical fluids as in the original three parameter corresponding states principle of Pitzer but can be chosen such that they are similar to the pure component of interest or, in the case of mixtures, to the key components of interest. This approach has been shown to work well in the predictions of transport properties [15–17] including those of aqueous mixtures [18].

The GCSP model was used as outlined in Refs. 15–18. Two binary interaction parameters (θ_{ij} and Ψ_{ij}) were used as follows:

$$T_{C_{ij}} V_{C_{ij}} = \psi_{ij} (T_{C_i} T_{C_j} V_{C_i} V_{C_j})^{1/2} \tag{5}$$

$$V_{C_{ij}} = \frac{(V_{C_i}^{1/3} + V_{C_i}^{1/3})^3}{8} \theta_{ij} \tag{6}$$

In general, it suffices to use one adjustable parameter to characterize each binary system. However, for highly nonideal mixtures such as those containing water, two binary interaction parameters may be required to correlate the data adequately.

The reference fluids may be chosen arbitrarily, although it is generally advantageous to select them from the principal components of the mixture. In the case of the acetic acid–water system, acetic acid and water were selected as the reference fluids. In order to apply the GCSP model to the acetic acid–water mixtures, the critical properties of pure acetic acid and water are required. These values were obtained from Ref. 19 and are $T_{c_1} = 592.7$ K, $V_{c_1} = 171.3\ \mathrm{cm^3 \cdot mol^{-1}}$, $T_{c_2} = 647.1$ K, and $V_{c_2} = 56.0\ \mathrm{cm^3 \cdot mol^{-1}}$. In this work, the viscosities of the reference fluids were calculated from Eq. (4) for acetic acid by letting $x = 1$ and for water with $x = 0$. In applying the model to thermal conductivity, a linear least-squares fit of the pure acetic acid data measured here was used and the expression for the thermal conductivity of saturated liquid water given by Kestin and Whitelaw [20] was used for the reference fluids.

Binary interaction parameters determined using this model are listed in Table VI. Previous work using the GCSP for the thermal conductivity of aqueous mixtures [18] used weight fraction based mixing rules rather then the mole fraction based van der Waals rules. Table VI shows the results obtained using both weight fraction- and mole fraction-based mixing rules. Viscosity was better modeled using mole fraction-based mixing rules. Moreover, it was found sufficient to use one adjustable binary interaction parameter to correlate this property. Hence, θ_{12} was set equal to unity in the viscosity calculations and Ψ_{12} was varied to obtain the best fit. It is interesting to note that the value of the binary interaction parameter (1.336) obtained in the viscosity correlation of acetic acid–water mixtures is very close to the value obtained for mixtures of water+methanol and water+2 propanol (1.33, 1.36). The fact that all the parameters for aqueous solutions are found to be between 1.33 and 1.36 is noteworthy.

In the application of the model to the thermal conductivity of acetic–water mixtures, marked improvement was obtained when two binary interaction parameters were used. Teja and Rice [18] showed that

Table VI. Binary Interaction Parameters Used in the GCSP Method for Acetic Acid–Water Mixtures

Property	Mixing rule basis	ψ_{12}	θ_{12}	AAD (%)	MAD (%)
η	Wt%	1.286	1	14.5	43.1
	Wt%	13.58	10.04	12.3	56.7
	Mol%	1.336	1	4.95	19.8
	Mol%	3.106	2.330	4.94	15.3
λ	Wt%	1.795	1	16.4	20.6
	Wt%	0.9602	0.4357	0.922	3.66
	Mol%	0.6450	1	4.73	8.48
	Mol%	1.487	1.422	0.935	3.56

binary mixtures of water with either ethanol, 1-propanol, 2-propanol, or acetone could be modeled using GCSP with only one binary interaction parameter, which had the same value (1.4) for all aqueous mixtures. They used a weight fraction-based mixing rule and the resulting average deviation between measured and calculated values was 3.9%. In the present work, a one-parameter model with mole fraction-based mixing rules

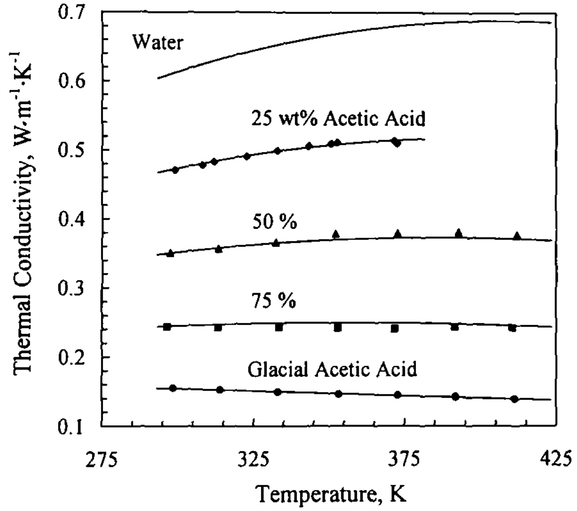

Fig. 2. Measured thermal conductivity of acetic acid–water mixtures as a function of temperature and concentration. Solid lines are from GCSP model using two binary interaction parameters (ψ_{12} = 1.487 and θ_{12} = 1.422).

resulted in an average deviation of 4.7%, whereas a 16.4% deviation was obtained with weight fraction-based mixing rules. In order to fit the data within experimental uncertainty, two binary interaction parameters were required with either weight or mole fraction-based mixing rules (Fig. 2).

5. CONCLUSIONS

The viscosity and thermal conductivity of acetic acid–water mixtures were measured at temperatures ranging from 290 to 460 K and concentrations from 12 to 100 wt%. Our data generally agree with the available literature data at low temperatures within experimental error. The generalized corresponding-states principle is capable of correlating both viscosity and thermal conductivity of the aqueous solutions and can be used to reliably extrapolate data to different temperatures. The model was shown to correlate adequately viscosity using only one adjustable binary interaction parameter. Thermal conductivity, however, required two binary interaction parameters to fit the data within experimental uncertainty.

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