Excess Molar Volumes, Viscosities, and Heat Capacities for the Mixtures of Ethylene Glycol + Water from 273.15 K to 353.15 K

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Experimental densities, viscosities, and heat capacities at different temperatures were measured over the entire mole fraction range for the binary mixture of ethylene glycol + water. Density values were used in the determination of excess molar volumes, V^E. The excess volumes, V^E, are fitted as a function of the mole fraction to a polynomial. $\Delta\eta$ was fitted to the Redlich−Kister equation. The excess volumes are negative over the entire composition at the lower temperatures. They show a U-shaped concentration dependence and decrease in absolute value with increasing temperature. At high temperatures, the excess molar volume shows an S-shaped dependence. $\Delta\eta$ values are negative over the entire range of mole fraction and have a very similar trend to that for V^E. The heat capacities of mixtures have the maximum values at x_1= 0.0312.

Introduction

Glycols find widespread application in the automotive, aviation, explosives, textile, surface coatings, food, cosmetic, pharmaceutical, tobacco, petroleum, and other industries. Ethylene glycol (1,2-ethanediol) is the simplest diol. It is one of the major products of the chemical industry. Its economic importance is founded on its two major commercial uses as an antifreeze and for fiber production. Ethylene glycol lowers the freezing point of water. Its ease of handling makes it a perfect antifreeze which is used, for example, in motor vehicles, solar energy units, heat pumps, water heating systems, solar energy systems, and industrial cooling systems. Ethylene glycol is also a commercially important raw material for the manufacture of polyester fibers. Other uses of ethylene glycol are as a humectant (moisture-retaining agent), plasticizer, softener, hydraulic fluid, and solvent. The manufacture of the ethylene glycol involves the oxidation of ethylene to produce ethylene oxide which is subsequently hydrated to ethylene glycol. From the point of the use and manufacture, the physical properties of ethylene glycol + water mixtures are, therefore, extremely important. The density, ρ, and viscosity, η, data of binary mixtures are important from the practical and theoretical viewpoint. The data for mixtures of ethylene glycol + water are required for the design of the heat-exchanger equipment used with antifreeze and its manufacture. As a result, the measurement and correlation of their thermophysical properties have attracted a great deal of attention and effort. Ashoka Ray et al.[1] determined the densities and partial molar volumes of water + ethylene glycol mixtures at 25 °C and 38.5 °C. Mauric Morenas et al.[2] measured the densities of the mixtures at 288.15 K, 298.15 K, and 308.15 K. Thomas et al.[3] and Rong-Jwyn Lee et al.[4] reported the viscosities of pure ethylene glycol at different temperatures. Manuchehr Dizechi et al.[5] measured densities and viscosities of ethylene glycol + water mixtures at 30.0 °C. Amalendu Pal et al.[6] measured the viscosity of mixtures at 303.15 K and 308.15 K. Reddy et al.[7] also measured the density and viscosity of mixtures at 308.15 K. No reports were available in the literature on the heat capacities of the mixtures. However, detailed studies of these physical properties for the ethylene glycol + water mixture over a wide range of temperatures and compositions have not been seen in the literature. Therefore, this situation prompted us to present the experimental data on ρ, η, and C_p over the temperature range from 293.15 K to 353.15 K and the entire mole fraction range of the mixture. From density, ρ, viscosity, η, and heat capacity, C_p, measurements, excess molar volume, V^E, and viscosity deviation, $\Delta\eta$, values have been calculated.

Experimental Section

The ethylene glycol used is of analytical grade and obtained from Tianjin Reagent Company. It was dried over molecular sieves and distilled under reduced nitrogen gas pressure. The purity was checked by gas chromatograph. The purity was greater than 99.8%. The purities of the liquids were further ascertained by measuring their densities and viscosities at 293.15 K and atmospheric pressure and comparing them with the corresponding literature values, as shown in Table 1. The data agree with the published results. Binary mixtures were prepared by mass on an analytical BP 210 S balance with ±0.01 mg accuracy. The possible error in the mole fraction is estimated to be around ±0.0001.

The measurement of densities of the pure components and the aqueous ethylene glycol solution was carried out using a bicapillary pycnometer having a bulb volume of 15 cm^3 and a capillary bore with an internal diameter of 1 mm. The pycnometer was placed in a transparent glass-walled water bath with the thermal stability 0.01 K. The pycnometer was calibrated by using density values of double-distilled water with a reproducibility within 1×10^{-4} $g\cdot cm^{-3}$. Densities were measured with an uncertainty of $\pm 5 \times 10^{-5}$ $g\cdot cm^{-3}$.The excess molar volumes were uncertain to ± 0.003 $cm^3\cdot mol^{-1}$.

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Table 1. Heat Capacities of Water

	C_p/kJ·kg^{-1}·K^{-1}					
T/K	run 1	run 2	run 3	run 4	ave value	ref 9
293.15	4.157	4.228	4.160	4.182	4.181	4.1799
298.15	4.156	4.233	4.166	4.186	4.184	4.1760
303.15	4.155	4.234	4.165	4.187	4.185	4.1785
308.15	4.150	4.236	4.159	4.173	4.179	4.1782
313.15	4.149	4.235	4.158	4.172	4.179	4.1786
318.15	4.149	4.233	4.157	4.172	4.178	4.1795
323.15	4.148	4.231	4.158	4.175	4.179	4.1807
328.15	4.148	4.256	4.159	4.176	4.179	4.1824
333.15	4.149	4.275	4.160	4.169	4.184	4.1844
338.15	4.149	4.286	4.161	4.171	4.189	4.1868
343.15	4.152	4.288	4.168	4.179	4.196	4.1896
348.15	4.156	4.301	4.169	4.180	4.198	4.1928
353.15	4.156	4.228	4.179	4.186	4.206	4.1964
ave % dev	−0.75%	1.61%	−0.49%	−0.21%	−0.09	

Viscosity was measured using an Ubbelohde suspended-level viscometer, calibrated with double-distilled water, whose viscosity was reported in the literature.[8] An electronic digital stopwatch with a readability of ±0.01 s was used for flow time measurement. Experiments were repeated a minimum of four times at each temperature for all compositions, and the results were averaged. The viscosity, η, of the liquid was then calculated from the following relationship

$$\nu = \frac{\eta}{\rho} = k(t - \theta) \tag{1}$$

where t is the flow time, ν is the kinetic viscosity, and k and θ are the viscometer constant and the Hagenbach correction factor, respectively.

The overall uncertainty of the viscosity measurements is dependent on the equilibrium stability of the viscometer, the time of flow, and the change of concentration, which are in the order of 1×10^{-2}, 1×10^{-2}, and 1×10^{-4}, respectively; viscosity values are uncertain to within the range ±0.003 mPa·s. All measurements were carried out in a well-stirred water bath with temperature controlled to within ±0.01 K.

The differential scanning calorimeter (NETZSCH Producer, mode DSC-204, Phoeuix, Germany) used for this study consists of a DSC and a thermal analysis controller from TA Instruments. The DSC operating range was from (−160 to 700) °C. Both the temperature and the heat flow associated with materials can be easily and rapidly measured with the system. The DSC features the temperature reproducibility within 0.1 K. The purge gas was nitrogen with a flow rate of 70 mL·min^{-1}. By using the sample-encapsulating press, the liquid sample was prepared within a hermetic sample pan. Hermetically sealed thermal contact between sample, pan, and constant disk reduced the thermal gradient in the sample and created an airtight seal that can resist higher internal pressures up to 0.3 MPa. These hermetic pans were used for volatile liquids, subliming materials, and those in a self-generated atmosphere. The internal volume of the hermetic pan was approximately 15 mm^3. The sample mass is in the range (8 to 15) mg.

To obtain accurate results, the calibration must be checked periodically. The DSC calibrations consist of that for the baseline slope, that for the cell constant, and temperature calibrations. The baseline calibration involves heating an empty cell through the entire range of temperature expected in subsequent experiments. Both the cell constant and the temperature calibration are obtained from the run in which a calibration material (e.g., indium) is heated to its melting point. The enthalpy of fusion and the melting point of indium are utilized to obtain the cell constant and the temperature calibrations.

Table 2. Comparison of Measured Densities, ρ, Viscosities, η, and Heat Capacities of the Ethylene Glycol with Literature Values

	ρ/g·cm^{-3}		η/mPa·s		C_p/J·mol^{-1}·K^{-1}	
T/K	exp	lit.	exp	lit.	exp	lit.
293.15	1.1137	1.1135[9]	20.833	20.16[4]	2.3728	2.344[11] 2.3458[12]
298.15	1.1085	1.1094[1] 1.1100[2] 1.1099[10]			2.3922	2.3984[13] 2.4103[14] 2.4135[15]
303.15	1.1032	1.1063[5] 1.1066[6]	13.646	13.61[3] 13.543[6] 13.76[5]		
313.15			9.443	9.409[4] 9.407[3]		
323.15			6.992			
333.15			5.060	5.075[4] 5.030[3]		
343.15			3.987			
353.15			3.021	3.078[4] 3.068[3]		

The heat capacities of a substance can be determined conveniently with a DSC by creating a baseline profile, a standard sample profile, and a sample profile. The heat capacities of the substances can be calculated as follows

$$C_p(\mathrm{s}) = \frac{M_{\mathrm{std}}(\mathrm{DSC_s} - \mathrm{DSC_{bl}})}{M_{\mathrm{s}}(\mathrm{DSC_{std}} - \mathrm{DSC_{bl}})} C_p(\mathrm{std}) \tag{2}$$

where C_p(s) and C_p(std) are the heat capacities of the sample and the standard at temperature T, respectively, M_{std} and M_{s} are the masses of the standard and sample, and $\mathrm{DSC_s}$ $\mathrm{DSC_{std}}$, and $\mathrm{DSC_{bl}}$ are the values of DSC signal at temperature T from the sample cure, the standard, and the baseline.

During a run, a temperature program was provided to cool the cell from ambient temperature to 10 °C with liquid nitrogen and hold it at that temperature for 10 min. And then the cell was heated to 90 °C at the heating rate 2 K·min^{-1}. The equipment was maintained at constant temperature for 10 min. For each sample, the average number of runs was four.

Four test runs for the measurement of the C_p of water and the averaged values were presented in Table 1. The average percentage deviations between the four test runs and the literature values[9] were −0.75%, 1.61%, −0.49%, and −0.21%. The average values given in the last row in Table 1 deviated by only 0.09% from the literature values. Thus, the measured C_p values of liquid water for temperatures from 293.15 to 353.15 K are in good agreement with the C_p values of Osborne et al.[9] On the basis of comparison with literature values for water, the average error of C_p measurements was estimated to be less than ±1%. The C_p values were reproducible within ±0.02 J·g^{-1}·K^{-1}.

The experimental results of density, viscosity, and heat capacity of the pure ethylene glycol and comparison with those from the literatures were summarized in Table 2. The absolute average deviations between the measured and literature values[1,2,5,6,10] for the densities at 293.15 K, 298.15 K, and 303.15 K were less than 0.17%. For the viscosities, the average absolute deviations between our data and literature values[4−6] were about 1.0% over the experimental temperature range from 293.15 K to 353.15 K. Excellent agreement was found between the three sets of data, with the average absolute deviation being 0.39%. However, there

Table 3. Experimental Densities, ρ, for Ethylene Glycol (1) + Water (2)

	ρ/g·cm^{-3} at the following values of x_1										
T/K	0.0000	0.0312	0.0676	0.1105	0.1620	0.2248	0.3031	0.4036	0.5370	0.7230	1.0000
293.15	0.9982	1.0117	1.0246	1.0372	1.0513	1.0648	1.0767	1.0878	1.0978	1.1057	1.1137
303.15	0.9957	1.0061	1.0184	1.0300	1.0434	1.0562	1.0675	1.078	1.087	1.0955	1.1032
313.15	0.9922	1.0013	1.0106	1.0219	1.0347	1.0468	1.0579	1.0679	1.0771	1.0858	1.0936
323.15	0.9881	0.9949	1.0051	1.0151	1.0274	1.0386	1.0492	1.059	1.068	1.0767	1.0847
333.15	0.9832	0.9898	0.9979	1.0072	1.0192	1.0303	1.0406	1.0503	1.0592	1.068	1.0764
343.15	0.9778	0.9837	0.9915	1.0004	1.0115	1.0223	1.0319	1.0413	1.0501	1.0588	1.0675
353.15	0.9718	0.9771	0.9847	0.9936	1.0038	1.0140	1.0237	1.033	1.042	1.0509	1.0600

Table 4. Experimental Viscosities for Ethylene Glycol (1) + Water (2)

	η/mPa·s at the following values of x_1										
T/K	0.0000	0.0312	0.0676	0.1105	0.1620	0.2248	0.3031	0.4036	0.5370	0.7230	1.0000
293.15	1.002	1.337	1.785	2.272	2.951	3.941	5.102	6.898	9.696	14.042	20.833
303.15	0.797	1.047	1.331	1.694	2.200	2.832	3.728	4.955	6.749	9.399	13.646
313.15	0.653	0.835	1.022	1.319	1.677	2.132	2.756	3.575	4.776	6.449	9.443
323.15	0.547	0.685	0.868	1.064	1.322	1.657	2.113	2.709	3.593	4.837	6.992
333.15	0.467	0.576	0.713	0.875	1.073	1.312	1.626	2.053	2.684	3.510	5.060
343.15	0.405	0.498	0.597	0.728	0.881	1.071	1.320	1.648	2.136	2.817	3.987
353.15	0.356	0.434	0.525	0.629	0.758	0.906	1.100	1.359	1.748	2.296	3.021

Table 5. Heat Capacities for Ethylene Glycol (1) + Water (2)

	C_p/J·mol^{-1}·K^{-1} at the following values of x_1										
T/K	0.0000	0.0312	0.0676	0.1105	0.1620	0.2248	0.3031	0.4036	0.5370	0.7230	1.0000
293.15	4.179	4.540	4.173	3.789	3.554	3.353	3.136	2.924	2.743	2.565	2.376
303.15	4.184	4.533	4.183	3.804	3.565	3.382	3.161	2.961	2.777	2.596	2.412
313.15	4.175	4.557	4.201	3.823	3.597	3.399	3.198	3.019	2.824	2.631	2.449
323.15	4.176	4.571	4.220	3.825	3.618	3.436	3.248	3.060	2.874	2.675	2.478
333.15	4.175	4.580	4.239	3.835	3.638	3.460	3.264	3.101	2.908	2.715	2.517
343.15	4.181	4.584	4.244	3.859	3.680	3.491	3.304	3.127	2.948	2.769	2.573
353.15	4.190	4.598	4.262	3.888	3.670	3.532	3.347	3.180	2.999	2.819	2.594
363.15		4.603	4.268	3.901	3.718	3.568	3.396	3.214	3.043	2.851	2.658
373.15				3.912	3.733	3.590	3.426	3.241	3.077	2.895	2.714

Table 6. Excess Molar Volumes, V^E, for Ethylene Glycol (1) + Water (2)

	V^E/cm^3·mol^{-1} at the following values of x_1										
T/K	0.0000	0.0312	0.0676	0.1105	0.1620	0.2248	0.3031	0.4036	0.5370	0.7230	1.0000
293.15	0.0000	−0.0587	−0.1056	−0.1508	−0.2299	−0.3008	−0.3378	−0.3533	−0.3298	−0.1995	0.0000
303.15	0.0000	−0.0118	−0.0594	−0.0940	−0.1710	−0.2410	−0.2759	−0.2919	−0.2520	−0.1697	0.0000
313.15	0.0000	0.0043	0.0071	−0.0285	−0.1016	−0.1622	−0.2046	−0.2157	−0.1936	−0.1392	0.0000
323.15	0.0000	0.0403	0.0193	−0.0036	−0.0662	−0.1158	−0.1516	−0.1675	−0.1495	−0.1099	0.0000
333.15	0.0000	0.0386	0.0554	−0.0489	−0.0183	−0.0682	−0.1013	−0.1197	−0.1057	−0.0739	0.0000
343.15	0.0000	0.0474	0.0635	0.0607	−0.0080	−0.0427	−0.0637	−0.0779	−0.0682	−0.0431	0.0000
353.15	0.0000	0.0579	0.0767	0.0713	0.0353	−0.0012	−0.0242	−0.0381	−0.0342	−0.0203	0.0000

was a large error at 293.15 K; the error was 3.2%. The C_p values obtained in this study were in good agreement with the literature.[11−15] The average absolute deviation was 0.86%

Results and Discussion

The experimental densities, viscosities, and heat capacities for ethylene glycol−water mixtures at different temperatures and compositions are listed in Tables 3−5.

The excess molar volume, V^E, was calculated from density measurements according to the following equation[16,17]

$$V^E = \frac{x_1M_1 + x_2M_2}{\rho_{12}} - \frac{x_1M_1}{\rho_1} - \frac{x_2M_2}{\rho_2} \tag{3}$$

where ρ_{12} is the density of the mixture and x_1, ρ_1, M_1, x_2, ρ_2, and M_2 are the mole fractions, densities, and molecular weights of pure components 1 and 2, respectively. The results of V^E are given in Table 6 and shown in Figure 1. Several methods have been proposed to correlate the viscosity data of liquid mixtures which generally require adjustable parameters.[18,19] However, the most common viscosity data are correlated using the viscosity deviation

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \tag{4}$$

where η, η_1, and η_2 are the viscosity of the mixture and those of pure components 1 and 2, respectively. The viscosity deviation of the system at different temperatures is graphically presented in Figure 2.

Theory gives little practical guidance to the selection of a suitable function for excess volume. Many authors have fitted the data to the Redlich−Kister polynomial equation.[20,21] However, this is not satisfactory for mixtures of complex polar compounds. Here the experimental excess volumes, V^E, are fitted as a function of the mole fraction to a polynomial of the form

$$V^E = x_1x_2\sum_{i=0}^{n} A_i x_1^n \tag{5}$$

where V^E is in cm^3·mol^{-1} and x_1 is the mole fraction of the ethylene glycol. The coefficients A_i and corresponding standard deviations are given in Table 7.

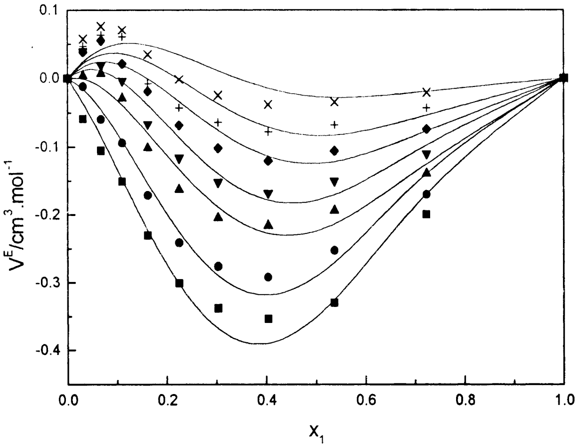

Figure 1. Excess volume variation with mole fraction for ethylene glycol (1) + water (2): ■, 293.15 K; ●, 303.15 K; ▲, 313.15 K; ▼, 323.15 K; ◆, 333.15 K; +, 343.15 K; ×, 353.15 K.

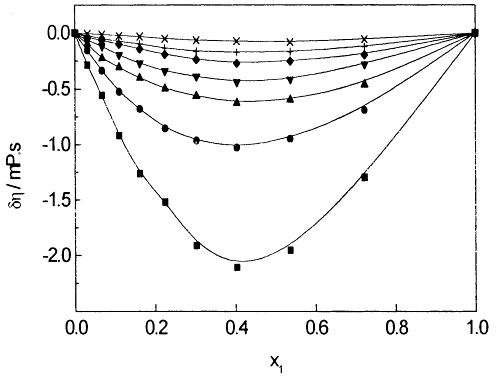

Figure 2. Viscosity deviation with mole fraction for ethylene glycol (1) + water (2): ■, 293.15 K; ●, 303.15 K; ▲, 313.15 K; ▼, 323.15 K; ◆, 333.15 K; +, 343.15 K; ×, 353.15 K.

Table 7. Coefficients of the Polynomial Equation and Standard Deviation for V^E

T/K	A_0	A_1	A_2	A_3	σ/cm^3·mol^{-1}
293.15	−0.948	−6.496	14.48	−7.105	0.079
303.15	−0.2113	−8.085	16.01	−7.759	0.006
313.15	0.2276	−6.606	10.68	−4.321	0.001
323.15	0.6834	−7.776	12.46	−5.378	0.054
333.15	0.7860	−6.078	8.443	−3.136	0.079
343.15	0.9362	−5.803	7.912	−3.023	0.144
353.15	1.002	−4.913	6.691	−2.752	0.245

The standard error values, SD, between the calculated and experimental data points are obtained by using

$$\mathrm{SD} = [\sum(Y^E_{\mathrm{cal}} - Y^E_{\mathrm{exp}})^2/(n-1)]^{1/2} \qquad (6)$$

where Y^E_{cal} is the calculated value and Y^E_{exp} is the experimental value.

From the plot it can be seen that the excess volumes are negative over the entire composition at the lower temperatures. They show a U-shaped concentration dependence and a decrease in absolute values with increasing temperature. At the high temperature, the excess molar volumes show an S-shaped dependence. Their qualities change from zero to positive and then to negative with increasing x_1.

For the ethylene glycol + water mixtures, the main factors which affects the excess volumes are the hydrogen bonding and the molecular sizes and shapes. Water molecules are strongly self-associated through hydrogen bonding. The ethylene glycol itself as well as glycol with water can also associate by hydrogen bonding. The strength of interaction decreases in the sequence water−water > water−ethylene glycol > ethylene glycol−ethylene glycol. The rupture among water molecules leads to a positive contribution to V^E. So the excess volumes at small x_1 are positive. Another factor is the molecular sizes. The volume of ethylene glycol is much larger than that of a water molecule. In the ethylene glycol + water mixture, the major factor is geometrical fitting. As these two kinds of solution are mixed, the small water molecules enter into the space between the ethylene glycol molecules and lead to negative excess volume. At large x_1 the effects of molecular sizes and shapes are stronger than those of interaction. The excess volumes are negative. The composition and temperature dependences of the excess volumes of the ethylene glycol + water mixtures can been explained by the two mentioned effects.

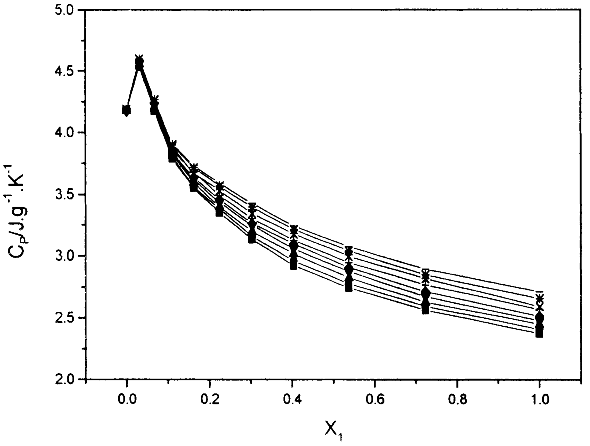

Figure 3. Heat capacities of the ethylene glycol (1) + water (2) system at various temperatures: ■, 293.15 K; ●, 303.15 K; ▲, 313.15 K; ▼, 323.15 K; ◆, 333.15 K; +, 343.15 K; ×, 353.15 K; ∗, 363.15 K; −, 373.15 K.

Table 8. Coefficients of the Redlich−Kister Equation and Standard Deviation for $\Delta\eta$

T/K	B_0	B_1	B_2	σ/mPa·s
293.15	−8.1139	2.807	1.803	0.033
303.15	−4.0491	1.4791	0.1661	0.022
313.15	−2.4472	−0.5202	−0.4193	0.022
323.15	−1.7009	0.5544	0.2697	0.011
333.15	−1.0438	0.1242	0.1206	0.007
343.15	−0.7078	0.1655	0.1892	0.007
353.15	−0.3174	0.0232	0.2047	0.002

The well-known Redlich−Kister polynomial equation, which has the following form,

$$\Delta\eta = x_1x_2\sum_{i=0}^{n}B_i(x_1 - x_2)^i \qquad (7)$$

is used to correlate the experimental data of the viscosity deviation. The coefficients B_i were determined by a multiple regression analysis based on the least-squares method and were summarized along with the standard deviations between the experimental and fitted values of the corresponding function in Table 8.

The heat capacities of the mixtures have been determined at different mole fractions and temperatures. The heat capacities are plotted against x_1 in Figure 3. The heat capacities of mixtures have the maximum values at x_1 = 0.0312. The heat capacities of pure water and the mixtures containing 0.0312 and 0.0676 mole fractions of ethylene glycol change very little with temperature.

Conclusions

New experimental values of densities and viscosities for the ethylene glycol + water mixtures at different temperatures and compositions are measured. In addition, the heat capacities are measured for the same temperature range with a differential scanning calorimeter. The experimental excess volumes, V^E, are fitted as a function of the mole fraction to a polynomial. $\Delta\eta$ was fitted to the Redlich−Kister equation. Good agreements were observed for these two properties. The excess volumes are negative over the entire composition at the lower temperatures. They show a U-shaped concentration dependence and decrease in absolute value with increasing temperature. At the high temperatures, the excess molar volumes show an S-shaped dependence. $\Delta\eta$ values are negative over the entire range of the mixtures and have a very similar changing trend to that for V^E. The heat capacities of mixtures have the maximum values at $x_1 = 0.0312$.

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