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Densities, Viscosities, and Derived Functions of Binary Mixtures: (Triethylene Glycol Dimethyl Ether + Water) and (*N*-Acetylmorpholine + Water) from 298.15 K to 343.15 K

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This paper reports the density and viscosity of aqueous triethylene glycol dimethyl ether solutions at five temperatures in the range of 303.15 K to 343.15 K and aqueous *N*-acetylmorpholine solutions at six temperatures in the range of 298.15 K to 343.15 K over the whole concentration range. The results are compared with data published in the literature when available. The excess molar volumes, the partial molar volumes at infinite dilution, the Grunberg−Nissan parameters, and the viscosity deviations were derived.

Introduction

Triethylene glycol dimethyl ether (TEGDME, triglyme) and *N*-acetylmorpholine (NAM) are excellent solvents for carbon dioxide capture from gas streams.[1] TEGDME is one of the main components forming Selexol[2], a widely used solvent in natural gas sweetening, and is made of a mixture of poly(ethylene glycol) dimethy ethers. Triglyme is used as an inert special solvent for reduction, alkylation reactions and those involving alkali metals and is also used as a component in brake fluids and hydraulic fluids. It is used as a solvent for adhesives, paints, graffiti remover formulations, and delacquers. TEGDME has been of interest to researchers in their attempts to understand interactions of water with amphiphilic molecules.

A mixture of *N*-formyl morpholine (NFM) and *N*-acetylmorpholine (NAM) is commercialized under the name Morphysorb. Pure NFM was initially proposed (alone) as a promising physical solvent for the removal of acid gas from gas streams by Zawacki et al.[3] NAM has a lower melting point (14 °C) compared to NFM (23 °C) and thus allows for the operation of the plant at lower temperatures where solubility is optimized. NAM allows also for preferential absorption of H_2S over CO_2.

Our laboratory has been active in measuring the physical and transport properties of chemical and physical solvents of interest to the gas processing industry. In this study, we have measured the density and viscosity of aqueous TEGDME and NAM solutions at various temperatures in order to cover a wide range of temperature found in industrial applications. Wallace et al.,[4] Pal et al.[5,6] and Dethlfesen et al.[7] have measured the densities and viscosities of aqueous TEGDME solutions at 25 °C. To the best of our knowledge, no data is available for aqueous NAM solutions.

Experimental Section

TEGDME (>98% pure) and NAM (>98% pure) were purchased from Fluka and were used without further purification. The solutions were prepared by mass on an analytical balance (model Ap 250 D, Ohaus, Florham Park, NJ) with ±0.01 mg accuracy (up to 55 g) and ±0.1 mg accuracy (up to 220 g). The possible uncertainty in the mole fraction is estimated to be around ±0.0001. TEGDME and NAM were kept over molecular sieves in a glass container. The densities of the binary mixtures were measured with an Anton Paar DMA-4500 density meter. A built-in Peltier thermostat automatically ensures the correct measuring temperature in the 0 °C to 90 °C range. Two integrated Pt 100 platinum thermometers provide temperature control (±0.01 °C) and are traceable to national standards. The calibration was done using air and water at ambient temperature. The densities of water were measured in the temperature range of 279.15 K to 353.15 K and were compared with the values measured by Bettin et al.[8] provided by Anton Paar in the instruction manual. The calibration was accepted if the measurements were within $\pm 3 \times 10^{-5}$ g·cm^{-3} of the published values. Uncertainties in our densities are about $\pm 5 \times 10^{-5}$ g·cm^{-3} when compared with the densities of pure MDEA published in the literature[9] in the temperature range of 298.15 K to 353.15 K. The densities of water were measured using deionized water.[10] Density measurements were reproducible to $\pm 2 \times 10^{-5}$ g·cm^{-3}.

For the viscosity measurements, the temperature was controlled by means of a digital controller (±0.004 °C) in a well-stirred water bath to better than ±0.01 °C as measured by a Cole-Parmer resistance thermometer (model H-01158-65, Anjou, Québec, Canada). Viscosities were determined with three different viscometers to cover, with precision, the range of temperature from 298.15 K to 343.15 K. A series of Cannon-Ubbelohde viscometers (Cole-Parmer) were used. The flow time was measured with a hand-held digital stopwatch capable of measuring time to within ±0.01 s. Experiments were repeated a minimum of four times at each temperature for all compositions. The equation for kinematic viscosity, according to Poiseuille's law, is

$$\nu = k_1 t - \frac{k_2}{t} \qquad (1)$$

where t is the efflux time and k_1 and k_2 are the viscometer

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Table 1. Review of the Literature Data for the Density and Viscosity of TEGDME

	ρ/g·cm^{-3}		η/mPa·s	
T/K	this work	literature	this work	literature
298.15	0.98063	0.9795[4] 0.98067[19] 0.98001[20] 0.98013[21] 0.9807[22]	1.95	1.960[4] 2.009[5] 1.95[22]

Table 2. Densities of Water (1) + TEGDME (2) from 303.15 K to 343.15 K

x_2	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
			ρ/g·cm^{-3}		
0.0000	0.99564	0.99221	0.98804	0.98312	0.97777
0.0198	1.00497	1.00031	0.99502	0.98918	0.98278
0.0500	1.01535	1.00877	1.00179	0.99439	0.98660
0.0700	1.01910	1.01154	1.00367	0.99546	0.98696
0.1000	1.02068	1.01219	1.00347	0.99452	0.98533
0.2000	1.01262	1.00321	0.99371	0.98405	0.97427
0.3000	1.00298	0.99351	0.98393	0.97423	0.96441
0.4000	0.99548	0.98598	0.97637	0.96669	0.95686
0.5000	0.99009	0.98057	0.97093	0.96120	0.95136
0.6000	0.98594	0.97637	0.96677	0.95706	0.94725
0.7000	0.98266	0.97309	0.96350	0.95378	0.94399
0.8000	0.97980	0.97025	0.96068	0.95098	0.94133
0.9000	0.97761	0.96806	0.95850	0.94890	0.93930
1.0000	0.97586	0.96636	0.95685	0.94732	0.93772

Table 3. Densities of Water (1) + NAM (2) from 298.15 K to 343.15 K

x_2	298.15 K	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
			ρ/g·cm^{-3}			
0.0000	0.99704	0.99565	0.99221	0.98804	0.98312	0.97777
0.0500	1.03497	1.03247	1.02712	1.02129	1.01501	1.00829
0.0700	1.04613	1.04332	1.03736	1.03103	1.02427	1.01717
0.1000	1.06006	1.05685	1.05017	1.04319	1.03589	1.02829
0.2000	1.08771	1.08385	1.07573	1.06757	1.05920	1.05063
0.3000	1.10016	1.09595	1.08743	1.07879	1.06999	1.06111
0.4000	1.10600	1.10168	1.09298	1.08420	1.07532	1.06632
0.5000	1.10855	1.10419	1.09541	1.08660	1.07766	1.06866
0.6000	1.10977	1.10539	1.09665	1.08784	1.07893	1.07000
0.7000	1.11029	1.10593	1.09723	1.08843	1.07963	1.07082
0.8000	1.11046	1.10612	1.09740	1.08871	1.07997	1.07121
0.9000	1.11040	1.10608	1.09741	1.08878	1.08010	1.07143
1.0000	1.11033	1.10600	1.09735	1.08874	1.08014	1.07155

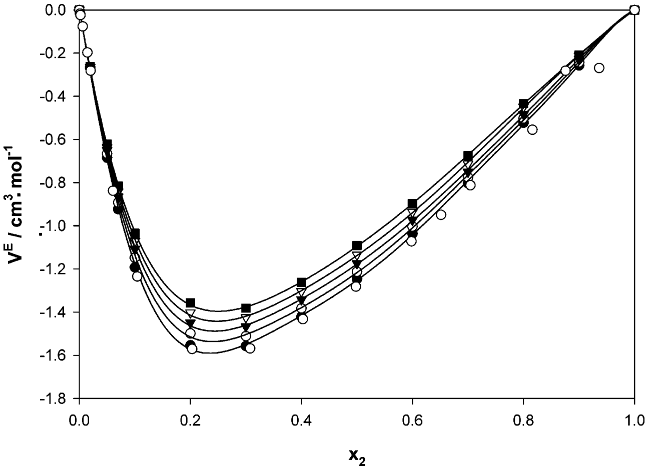

Figure 1. Excess molar volumes of the water (1) + TEGDME (2) system at various temperatures: o, 298.15 K;[6] ●, 303.15 K; ○, 313.15 K; ▼, 323.15 K; ▽, 333.15 K; ■, 343.15 K.

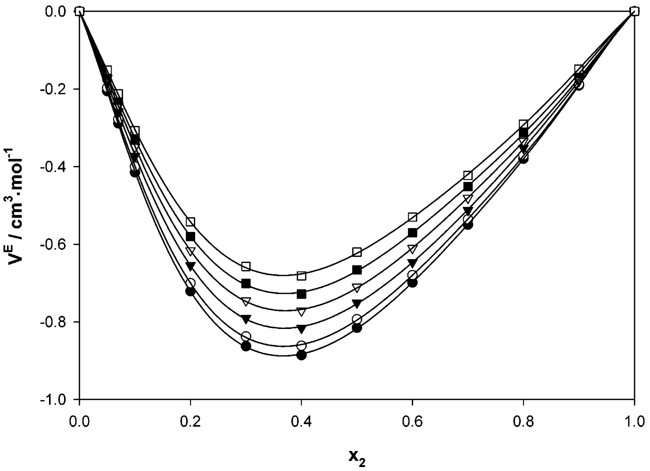

Figure 2. Excess molar volumes of the water (1) + NAM (2) system at various temperatures: ●, 298.15 K; ○, 303.15 K ▼, 313.15 K; ▽, 323.15 K; ■, 333.15 K; □, 343.15 K.

constants. The second term representing the correction due to the kinetic energy was found to be negligible.[11] The value of the absolute viscosity (η) was obtained by multiplying the measured kinematic viscosity (ν) by the measured density. Calibration constants for the two viscometers were checked using high-purity ethylene glycol (EG), diethylene glycol (DEG), and water. A review of the values of the kinematic and dynamic viscosities of EG and DEG, with new experimental data, was published by Lee and Teja.[12] Values of the viscosity of water were taken from Stokes and Mills.[13] From the overall average percent deviation of the means of the average flow time and the accuracy of the density measurement, we estimate the uncertainty of the absolute viscosity to be 1.0%.

Results and Discussion

Values of the density and viscosity for pure TEGDME are compared with published literature values and are shown in Table 1.

Experimentally measured densities of aqueous TEGDME solutions at 303.15, 313.15, 323.15, 333.15, and 343.15 K and NAM solutions at 298.15, 303.15, 313.15, 323.15, 333.15, and 343.15 K throughout the whole concentration range are listed in Tables 2 and 3. The densities of the TEGDME solutions increased with increasing mole fractions reaching maxima at 5 mol % followed by a steady decrease until the densities reached a lower value than that of pure water. The densities of NAM solutions increased steadily to reach maxima at 80 mol % and stayed virtually at the same values as those of pure NAM. The density values of the binary mixtures were used to calculate the excess molar volume, V_m^E, as

$$V_m^E = V_m - V^\circ{}_1 x_1 - V^\circ{}_2 x_2 \tag{2}$$

where V_m is the molar volume of the mixture and V_1° and V_2° are those of pure water and pure TEGDME or NAM, respectively; x_1 and x_2 are the mole fractions of pure water and pure TEGDME or NAM, respectively. Figures 1 and 2 display the dependence of V_m^E on the composition at various temperatures. In all cases, V_m^E is negative as is common for other completely miscible (water + polar organic) solvents with a minimum at around 25 mol % for TEGDME and 40 mol % for NAM. These V_m^E values become less negative with increasing temperature, as is also common.

A Redlich−Kister[13] relation was used to correlate the excess volume data:

$$V_m^E/\text{cm}^3\cdot\text{mol}^{-1} = x_1 x_2 \sum_{i=0}^{n} a_i (x_1 - x_2)^i \tag{3}$$

Table 4. Redlich−Kister Equation Fitting Coefficients of the Excess Volumes for (Water + TEGDME) and (Water + NAM) from 298.15 K to 343.15 K

T/K		a_0	a_1	a_2	a_3	a_4	σ/cm^3·mol^{-1}
298.15	NAM	−3.2589	−2.0053	−0.9672	0.7337	1.3468	0.004
303.15	TEGDME	−4.9413	−3.9486	−4.4582	−3.7744		0.021
	NAM	−3.1704	−1.9509	−0.9567	0.7758	1.3471	0.004
313.15	TEGDME	−4.8034	−3.8335	−4.2113	−3.6824		0.018
	NAM	−3.0089	−1.8075	−0.8356	0.74921	1.2470	0.003
323.15	TEGDME	−4.6593	−3.7104	−4.0257	−3.6384		0.014
	NAM	−2.8436	−1.7264	−0.7255	0.80271	1.0706	0.003
333.15	TEGDME	−4.5015	−3.6816	−3.8202	−3.5590		0.017
	NAM	−2.6707	−1.6409	−0.6993	0.76211	1.0302	0.003
343.15	TEGDME	−4.3700	−3.7070	−3.0581	−3.07945	−1.2299	0.008
	NAM	−2.4909	−1.5523	−0.6975	0.73100	1.0450	0.003

Table 5. Partial Molar Volumes, V_1^∞ (Water) and V_2^∞ (TEGDME or NAM), at Infinite Dilution from 298.15 K to 343.15 K

	V_1^∞/cm^3·mol^{-1}		V_2^∞/cm^3·mol^{-1}	
T/K	TEGDME	NAM	TEGDME	NAM
298.15		18.0		116.3
303.15	15.1	18.0	169.1	116.8
313.15	15.5	18.1	170.7	117.7
323.15	15.9	18.2	172.5	118.6
333.15	16.0	18.3	174.1	119.6
343.15	16.2	18.4	176.1	120.5

The coefficients and the standard deviation (σ) for TEGDME and NAM solutions are presented in Table 4. It is well known that the Redlich−Kister relation does not correlate unsymmetrical data very well. The F test was used to determine the optimum number of coefficients.

Hepler[15] and Neal and Goring[16] recognized the usefulness of thermal expansion data for revealing correlations between the molecular structure of solutes and their effects on water structure. They suggested using the sign of the second derivative of the partial molar volume at infinite dilution of the solute with respect to temperature $(\partial^2 V_2^\circ/\partial T^2)_P$ to classify solutes as "structure makers" or "structure breakers". A positive sign corresponds to a structure-making solute, whereas a negative sign corresponds to a structure-breaking solute.

The partial molar volumes of TEGDME and NAM at infinite dilution (V_2^∞) in water were obtained using the method proposed by Maham et al.[17] The apparent molar volume was first calculated as

$$V_{\phi,1} = V^\circ{}_1 + \frac{V_\mathrm{m}^\mathrm{E}}{1 - x_2} \tag{4}$$

$$V_{\phi,2} = V^\circ{}_2 + \frac{V_\mathrm{m}^\mathrm{E}}{x_2} \tag{5}$$

where $V_1^\circ$and V_2° are the molar volumes of pure water and TEGDME or NAM, respectively. An extrapolation of the regressed values of $V_{\phi,1}$ (as a function of x_2) to $x_2 = 1$ leads to V_1^∞, and a similar extrapolation to $x_2 = 0$ leads to V_2^∞. Partial molar volumes at infinite dilution are listed for both systems in Table 5. V_2^∞ values for TEGDME and NAM varied linearly with temperature. According to the criterion described above, the solute (TEGDME or NAM) would be considered to have no net effect on the structure of water. All values of the molar volume at infinite dilution (V_2^∞) for TEGDME and NAM were smaller than the corresponding molar values of pure TEGDME or pure NAM (V_2°). The change in the excess volumes can be explained by TEGDME or NAM molecules fitting (partially) into the open or empty space in liquid water. This picture does not invoke either structure-making or structure-breaking properties and is consistent with the observation that $(\partial^2 V_2^\infty/\partial T^2)_P$ values are approximately equal to zero.

Table 6. Viscosities for Water (1) + TEGDME (2) Mixtures from 303.15 K to 343.15 K

x_2	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
			η/mPa·s		
0.0000	0.8900	0.6531	0.5470	0.4660	0.4049
0.0198	1.38	1.09	0.8W76	0.728	0.617
0.0500	2.38	1.79	1.39	1.29	0.922
0.0700	2.99	2.19	1.67	1.33	1.08
0.1000	3.60	2.60	1.96	1.53	1.24
0.2000	3.76	2.80	2.10	1.660	1.34
0.3000	3.13	2.39	1.88	1.52	1.26
0.4000	2.67	2.09	1.69	1.40	1.17
0.5000	2.38	1.94	1.60	1.30	1.10
0.6000	2.14	1.73	1.44	1.21	1.04
0.7000	1.99	1.65	1.37	1.16	0.998
0.8000	1.90	1.58	1.32	1.13	0.976
0.9000	1.82	1.51	1.27	1.10	0.952
1.0000	1.77	1.48	1.26	1.08	0.945

Table 7. Viscosities for Water (1) + NAM (2) Mixtures from 298.15 K to 343.15 K

x_2	298.15 K	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
			η/mPa·s			
0.0000	0.8903	0.7976	0.6531	0.5470	0.4660	0.4049
0.0500	1.79	1.56	1.23	0.998	0.857	0.743
0.0700	2.22	1.96	1.52	1.21	1.04	0.861
0.1000	3.22	2.75	2.10	1.65	1.24	1.03
0.2000	5.96	4.94	3.55	2.68	2.05	1.66
0.3000	8.70	7.01	4.80	3.51	2.66	2.09
0.4000	10.1	8.02	5.47	3.96	3.00	2.34
0.5000	10.4	8.29	5.68	4.13	3.13	2.46
0.6000	10.5	8.48	5.77	4.40	3.41	2.59
0.7000	9.61	7.89	5.56	4.13	3.18	2.54
0.8000	9.18	7.56	5.42	4.06	3.17	2.53
0.9000	8.79	7.30	5.28	4.00	3.14	2.54
1.0000	8.48	7.10	5.18	3.96	3.12	2.55

Experimentally measured viscosities of the binary solutions of aqueous TEGDME solutions at 303.15, 313.15, 323.15, 333.15, and 343.15 K and NAM solutions at 298.15, 303.15, 313.15, 323.15, 333.15, and 343.15 K are listed in Tables 6 and 7. The largest viscosity values were those of 15 mol % for TEGDME and 60 mol % for NAM at all temperatures.

Experimental viscosity values of the binary mixtures were used to calculate the viscosity deviation, defined by

$$\Delta\eta = \eta - \eta_1 x_1 - \eta_2 x_2 \tag{6}$$

where η is the viscosity of the mixture and η_1 and η_2 are those of pure water and pure TEGDME or pure NAM, respectively. The viscosity deviation represents deviations from a rectilinear dependence of viscosity on mole fraction. Figures 2 and 3 display the dependence of $\Delta\eta$ on the composition and temperature. Values of $\Delta\eta$ were positive

Table 8. Redlich−Kister Equation Fitting Coefficients of the Viscosity Deviations for (Water + TEGDME) and (Water + NAM) from 298.15 K to 343.15 K

T/K		a_0	a_1	a_2	a_3	a_4	σ/mPa·s
298.15	NAM	23.58	14.07	−14.75	−17.84		0.12
303.15	TEGDME	3.82	6.95	15.71	14.55		0.13
	NAM	18.03	10.92	−9.860	−12.68		0.09
313.15	TEGDME	3.114	4.930	11.00	10.77		0.07
	NAM	11.38	7.252	−4.590	−7.617		0.05
323.15	TEGDME	2.470	3.609	7.555	7.743		0.05
	NAM	7.976	3.259	−3.318			0.07
333.15	TEGDME	1.917	2.487	6.039	6.691		0.06
	NAM	5.726	2.326	−2.047			0.06
343.15	TEGDME	1.669	2.204	2.911	4.085	2.483	0.01
	NAM	4.119	1.998	−0.973			0.03

with a maximum around 15 mol % for all temperatures for TEGDME and 40 mol % for NAM solutions The curves for both systems were asymmetrical with a more pronounced skewing effect for the TEGDME solutions.

The calculated values of $\Delta\eta$ were correlated with a Redlich−Kister[13] relation:

$$\Delta\eta/\text{mPa·s} = x_1x_2\sum_{i=0}^{n} a_i(x_1 - x_2)^i \qquad (7)$$

The coefficients and the standard deviation (σ) for both systems are presented in Table 8. The F test was used to determine the optimum number of coefficients. The Grunberg−Nissan[18] parameters (d_{12}) were calculated for all compositions and temperatures. They were all positive and varied from a maximum of 25.55 in the water-rich region at 313.15 K (2 mol %) to 1.02 in the TEGDME-rich region (343.15 K, 90 mol %). The values of the parameters drop very quickly in the water-rich region to reach an almost constant value at concentrations greater than 60 mol %. The parameters were also all positive for aqueous NAM solutions and varied from a maximum of 8.2 in the water-rich region at 298.15 K (7 mol %) to 2.0 in the NAM-rich region (343.15 K, 90 mol %). After an initial increase at 7 mol %, the values of the parameters dropped steadily in the water-rich region to reach minimum values in the NAM-rich region. The positive values of the coefficients suggest the predominance of interaction forces through hydrogen bonding as supported by the negative values of the excess volumes.

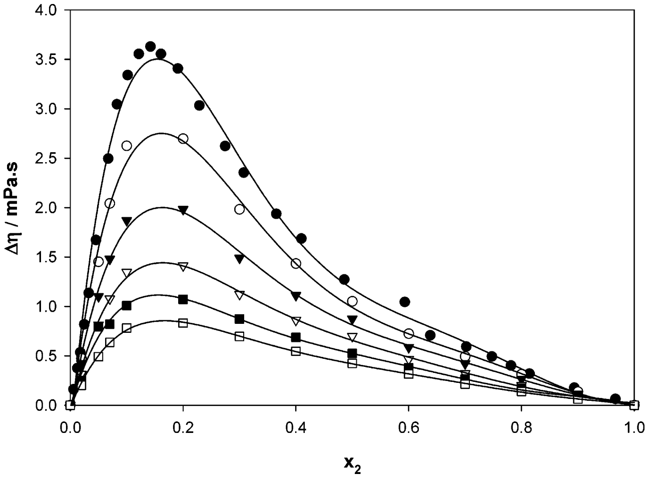

Figure 3. Viscosity deviations of the water (1) + TEGDME (2) system at various temperatures: ●, 298.15 K;[4] ○, 303.15 K; ▼, 313.15 K; ▽, 323.15 K; ■, 333.15 K; □, 343.15 K.

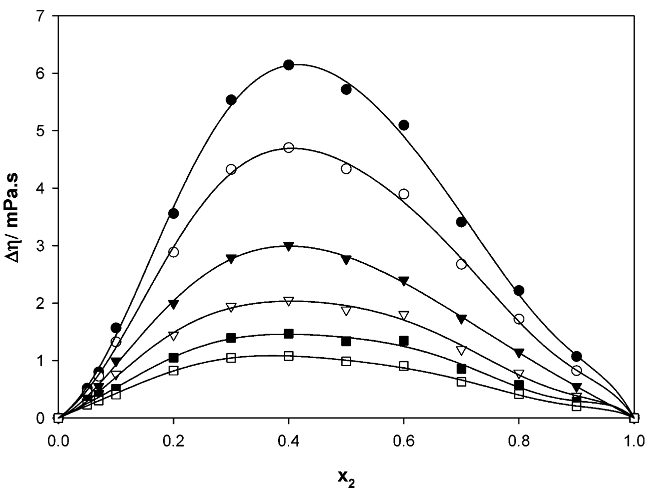

Figure 4. Viscosity deviations for water (1) + NAM (2) system at various temperatures: ●, 298.15 K; O, 303.15 K; ▼, 313.15 K; ▽, 323.15 K; ■, 333.145 K; □, 343.15 K.

Conclusions

This paper reports experimental data for the densities and viscosities of aqueous TEGDME and NAM solutions over ranges of temperature from 303.15 K to 343.15 K and 298.15 K to 343.15 K, respectively. The calculated V^E values for the aqueous TEGDME and NAM solutions were all negative at all temperatures and compositions. The criterion proposed by Hepler suggests that the addition of TEGDME or NMA to water had no effect on the structure. The viscosity deviations $\Delta\eta$ for TEGDME + water and NAM + water were positive at all concentrations and temperatures. In contradiction to Hepler's criteria, the S shape of the viscosity of the aqueous solutions for both TEGDME and NAM and the presence of a maximum in the viscosity deviation curves (15 mol % for TEGDME and 40 mol % for NAM) suggest the presence of complexes according to Fort and Moore.[23] It is important to note that Hepler's criteria were found to inadequately suggest that the addition of NMP has no effect on the structure of water, whereas other studies strongly suggested the formation of stable complexes.[24] The positive values of the Grunberg−Nissan parameters and the excess volumes at all temperatures and compositions suggest the predominance of interaction forces for both aqueous systems.

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