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Thermophysical Properties for Diethylene Glycol + Nitrobenzene and Triethylene Glycol + (Chloro-, Bromo-, Nitro-) Benzene Systems at Different Temperatures

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Abstract The densities, viscosities, sound speeds, and relative permittivities for four binary mixtures of glycols + organic solvents that are miscible over the complete composition range, namely, diethylene glycol (DEG) + nitrobenzene and triethylene glycol (TEG) + chlorobenzene, + bromobenzene, and + nitrobenzene have been measured at atmospheric pressure and at temperatures from 298.15 to 313.15 K. The excess molar volumes are calculated and fitted with a Redlich–Kister type equation. The qualitative analysis of excess molar volumes revealed that the structure-making effects probably in the form of weak Cl···H–O hydrogen bonding, Cl···O electron acceptor–donor interactions, and interstitial accommodation of chlorobenzene in associate structures of triethylene glycol, etc. are predominant in these mixtures.

Keywords Binary mixtures · Density · Glycols · Halogenated benzene · Nitrobenzene · Relative permittivity · Sound speeds

1 Introduction

As part of our continued interest in the thermophyscial properties of esters + nonpolar and polar organic solvents, we reported various thermophyscial properties and derived functions for binary systems of aliphatic esters (methyl, ethyl, butyl, and isoamyl acetates) + aromatic hydrocarbons (benzene, toluene, *o*-, *m*-, and *p*-xylenes), + chloro-, bromo-, and nitro benzenes [1] and the same aliphatic esters + glycols (ethylene, diethylene, triethylene, and propylene) [2]. In order to explain the thermophysical behavior of complex esters + glycols + organic solvents ternary systems, data for the constituent binary pairs are essential. Hence, the present work reports the densities, ρ, dynamic

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viscosities, η, speeds of sound, υ, and relative permittivities, ε_r, for four binary mixtures of diethylene glycol (DEG) + nitrobenzene and triethylene glycol (TEG) + chlorobenzene, + bromobenzene, and + nitrobenzene at atmospheric pressure and at temperatures from 298.15 K to 313.15 K. We could study only these four binary mixtures because of miscibility problems with other glycols + organic solvent liquid mixtures.

2 Experimental

2.1 Materials

Diethylene and triethylene glycols were purchased locally and were fractionally distilled in vacuum; the middle fractions were collected and dried over sodium sulfate. After decantation, the liquids were fractionally distilled. The gas chromatographic analysis of the distilled glycols revealed that various impurities such as free acid (as acetic acid) and water, in general, and peroxides (as H_2O_2) in diethylene and triethylene glycols and ethylene glycol in diethylene glycol, in particular, were reduced, and the final purity of the glycols was found to be greater than 99.5% on a mole basis. Chloro-, bromo-, and nitrobenzenes were the same as described elsewhere [1].

2.2 Methods

The binary solutions were prepared by mass in hermetically sealed glass vials. The solutions of each composition were prepared fresh, and all the properties were measured the same day. The mass measurements, accurate to ±0.01 mg, were made on a single-pan analytical balance (Dhona 100 DS, India). The estimated uncertainty in the mole fraction was ±0.0001. Densities of the pure liquids and their mixtures were measured with a high-precision vibrating-tube digital densimeter (Anton Paar, DMA 5000). The repeatability of the temperature has been found to be ±0.003°C. The uncertainty in the temperature during the measurements, however, is ±0.01°C since Pt 100 measuring sensors were used. The instrument was calibrated with air and with four-times-distilled and freshly degassed water at each temperature using an in-built software-driven special adjustment procedure. The repeatability in the densities for the freshly distilled pure liquids and prepared binary mixtures has been found to be better than $3 \times 10^{-6}\ \mathrm{g \cdot cm^{-3}}$. The uncertainty in densities of the pure liquids was determined by comparing our measured values at different temperatures with literature data (Table 1). This comparison gave a mean absolute deviation of $3.45 \times 10^{-5}\ \mathrm{g \cdot cm^{-3}}$. Hence, the precision and uncertainty of the densities reported in the present work are 3×10^{-6} and $3.5 \times 10^{-5}\ \mathrm{g \cdot cm^{-3}}$, respectively. The viscosity, η, of the pure liquids and liquid mixtures were determined using an Ubbelohde suspended-level viscometer. The viscometer was suspended in a thermostatted water bath maintained at ±0.01°C. Four sets of readings for the flow times were taken using a Racer stop watch that can register time to ±0.1 s, and the arithmetic mean was taken for the calculation of the viscosity. The viscometer was calibrated with four-times-distilled water and dry cyclohexane. The estimated precision and uncertainty in the viscosity measurements were found to be $\pm 0.001\ \mathrm{mPa \cdot s}$ and $\pm 0.012\ \mathrm{mPa \cdot s}$, respectively. The sound speeds,

υ, were measured using an ultrasonic interferometer (Mittal Enterprises, New Delhi, India) operating at a fixed frequency of 2 MHz. The measured speeds of sound have a precision of $\pm 0.8\,\mathrm{m \cdot s^{-1}}$ and an uncertainty better than $\pm 1.5\,\mathrm{m \cdot s^{-1}}$. The relative permittivities of the individual pure components and binary mixtures were calculated from the capacitance measurements made with a universal dielectrometer, Type OH-301 of Radelkis, Hungary. The measured relative permittivities have an estimated precision and uncertainty of ±0.001 and ±0.009, respectively. The temperatures (accurate to ±0.01°C) during the υ and ε_r measurements were maintained using an INSREF (India) circulator (Model 020A).

3 Results

3.1 Densities (ρ) and Excess Molar Volumes (V_m^E)

The experimental densities at different mole fractions of glycols for the four binary mixtures of diethylene glycol + nitrobenzene and triethylene glycol + chlorobenzene, + bromobenzene, and + nitrobenzene are listed in Table 2. The compositional variation of V_m^E was mathematically represented through the following equation:

$$A^\mathrm{E} = x_1\,(1 - x_1) \sum_{i=\mathrm{o}}^{i=\mathrm{n}} a_i\,(2x_1 - 1)^i \tag{1}$$

where $A^\mathrm{E} =$ an excess quantity or a deviation function, a_i's are the fitting coefficients, and x_1 is the glycol mole fraction. The values of a_i (as estimated through multiple regression analysis based on least-squares analysis) and σ, the standard deviation between experimental and fitted V_m^E values are given in Table 3. The graphical representation of the compositional dependence of V_m^E for the four binary mixtures at $T = 298.15\,\mathrm{K}$ is shown in Fig. 1. The V_m^E values for triethylene glycol + chlorobenzene mixtures are small and negative over the complete composition range at the four temperatures. The replacement of the chloro-group with the bromo-group in bromobenzene resulted in remarkably high and positive V_m^E. The trend in V_m^E versus x_1 profiles for triethylene glycol + nitrobenzene mixtures was similar to that for triethylene glycol + bromobenzene systems, but with smaller positive values. The diethylene glycol + nitrobenzene system exhibited the same trend in V_m^E as that for triethylene glycol + nitrobenzene except that the values are less positive, especially in the mole fraction range $x_1 = 0.1 - 0.8$.

3.2 Speed of Sound (ν)

The speed-of-sound data for the four binary mixtures at $T = (298.15$ and $308.15)$ K are listed in Table 4.

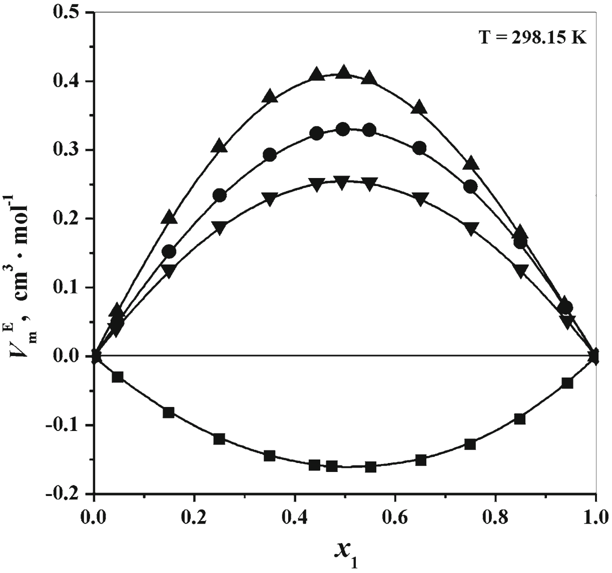

Fig. 1 Excess molar volumes of glycols (1) + organic solvents (2) at $T = 298.15$ K: (▼) diethylene glycol + nitrobenzene; triethylene glycol + (●) nitrobenzene, + (▲) bromobenzene, and + (■) chlorobenzene, _____ are fitted values as per Eq. 1 and coefficients from Table 3

3.3 Dynamic Viscosities (η)

The dynamic viscosities for the four binary mixtures at $T = (298.15$ and $308.15)$ K are listed in Table 4. The mixture viscosities were also correlated using two models.

The Grunberg–Nissan equation is given as

$$\ln \eta_{12} = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1 x_2 G_{12} \tag{2}$$

The McAllister equation based on the Eyring theory of absolute reaction rates and the three-body interaction model is expressed as

$$\begin{aligned}\ln \nu_{12} = {} & x_1^3 \ln \nu_1 + 3x_1^2 x_2 \ln M_{12} + 3x_1 x_2^2 \ln M_{21} + x_2^3 \ln \nu_2 - \ln(x_1 + (x_2 M_2/M_1)) \\ & + 3x_1^2 x_2 \ln((2/3) + (M_2/3M_1)) + 3x_1 x_2^2 \ln((1/3) + (2M_2/3M_1)) \\ & + x_2^3 \ln(M_2/M_1)\end{aligned} \tag{3}$$

The terms G_{12} and M_{12}, M_{21} that appear in Eqs. 2 and 3 have been treated as adjustable parameters. η_i and ν_i are the dynamic and kinematic viscosities of the respective pure or mixture components. The values of the adjustable parameters, G_{12}, M_{12}, and M_{21} along with σ values between experimental and correlated values are given in Table 5. Both the equations correlated dynamic and kinematic viscosities of mixtures adequately as the observed standard deviation between experimental and correlated values did not exceed 0.001 mPa · s.

Table 1 Densities, ρ, viscosities, η, speeds of sound, υ, and relative permittivities, ε_r, for pure component liquids at T = (298.15 to 313.15) K, along with literature values

T (K)	298.15		303.15		308.15		313.15	
Diethylene glycol								
ρ (g · cm^{-3})	1.113510	1.1135 [3]	1.109560	1.1095 [4]	1.106221	1.1062 [4]	1.103021	1.1030 [5]
η (mPa · s)	30.012	30.0 [6]			16.972			
υ (m · s^{-1})	1580	1577 [7]			1550			
ε_r	30.921	30.925 [7]			29.163	29.160 [7]		
Triethylene glycol								
ρ (g · cm^{-3})	1.119590	1.1195 [6]	1.115882		1.111960		1.109532	1.1095 [5]
η (mPa · s)	34.398				21.308			
υ (m · s^{-1})	1612				1586			
ε_r	23.049	23.047 [7]			21.856	21.850 [7]		
Chlorobenzene								
ρ (g · cm^{-3})	1.100858	1.1009 [6]	1.095459	1.09547 [9]	1.089705	1.0897 [10]	1.084683	1.08468 [11]
η (mPa · s)	0.755	0.756 [8]			0.679	0.673 [8]		
υ (m · s^{-1})	1269	1268 [12]			1224	1224 [12]		
ε_r	5.698				5.489	5.489 [13]		
Bromobenzene								
ρ (g · cm^{-3})	1.488272	1.48820 [6]	1.481562	1.48150 [6]	1.475386	1.47539 [7]	1.467088	
η (mPa · s)	1.041	1.045 [14]			0.964			
υ (m · s^{-1})	1163				1122	1120.7 [15]		
ε_r	5.124				4.980			
Nitrobenzene								
ρ (g · cm^{-3})	1.198332	1.19833 [6]	1.193481	1.19347 [17]	1.188222	1.1883 [10]	1.183263	1.1832 [10]
η (mPa · s)	1.791	1.8172 [10]			1.543	1.5543 [16]		
υ (m · s^{-1})	1461	1463 [7]			1423			
ε_r	34.809	34.78 [6]			33.174			

Table 2 Experimental densities (ρ) for glycols (1) + organic solvents (2) at T = (298.15, 303.15, 308.15, and 313.15) K

x_1	ρ (g · cm^{-3}) T in K				x_1	ρ (g · cm^{-3}) T in K			
	298.15	303.15	308.15	313.15		298.15	303.15	308.15	313.15
TEG[a] + *Chlorobenzene*					*TEG*[a] + *Bromobenzene*				
0.0473	1.10231(9)	1.09701(3)	1.09135(3)	1.08648(8)	0.0462	1.46599(1)	1.45946(7)	1.45343(2)	1.44551(2)
0.1490	1.10520(5)	1.10008(8)	1.09463(2)	1.09007(9)	0.1502	1.41804(7)	1.41187(7)	1.40605(6)	1.39892(9)
0.2493	1.10774(9)	1.10280(9)	1.09755(3)	1.09328(3)	0.2493	1.37512(1)	1.36925(2)	1.36358(9)	1.35715(5)
0.3495	1.11002(7)	1.10525(9)	1.10019(7)	1.09619(5)	0.3505	1.33391(7)	1.32835(4)	1.32287(4)	1.31709(9)
0.4390	1.11186(2)	1.10724(4)	1.10235(2)	1.09858(0)	0.4433	1.29831(9)	1.29304(8)	1.28778(2)	1.28257(5)
0.4734	1.11252(0)	1.10796(9)	1.10313(1)	1.09944(7)	0.4974	1.27847(0)	1.27337(4)	1.26825(5)	1.26336(5)
0.5509	1.11391(2)	1.10948(0)	1.10479(3)	1.10130(3)	0.5484	1.26033(2)	1.25540(5)	1.25043(7)	1.24583(6)
0.6512	1.11553(3)	1.11126(6)	1.10675(6)	1.10351(5)	0.6475	1.22657(6)	1.22197(8)	1.21732(3)	1.21326(1)
0.7493	1.11692(4)	1.11281(8)	1.10847(7)	1.10547(4)	0.7514	1.19310(6)	1.18883(9)	1.18450(8)	1.18097(5)
0.8483	1.11813(5)	1.11418(8)	1.11001(3)	1.10724(5)	0.8484	1.16342(7)	1.15943(1)	1.15534(9)	1.15227(7)
0.9428	1.11910(4)	1.11530(6)	1.11128(7)	1.10873(5)	0.9370	1.13747(0)	1.13366(7)	1.12972(3)	1.12704(1)
TEG[a] + *Nitrobenzene*					*DEG*[a] + *Nitrobenzene*				
0.0465	1.19304(5)	1.18821(0)	1.18299(1)	1.17815(8)	0.0441	1.19437(5)	1.18960(6)	1.18438(5)	1.17949(8)
0.1495	1.18196(3)	1.17718(6)	1.17205(8)	1.16749(5)	0.1493	1.18499(0)	1.18030(7)	1.17522(8)	1.17050(6)
0.2501	1.17196(9)	1.16727(4)	1.16224(3)	1.15793(7)	0.2505	1.17605(7)	1.17138(3)	1.16649(0)	1.16192(3)
0.3505	1.16279(5)	1.15819(7)	1.15327(6)	1.14922(2)	0.3502	1.16736(6)	1.16270(1)	1.15801(1)	1.15360(0)
0.4432	1.15500(4)	1.15050(8)	1.14570(0)	1.14187(8)	0.4438	1.15931(8)	1.15469(5)	1.15020(1)	1.14594(4)
0.4957	1.15086(8)	1.14643(4)	1.14169(6)	1.13800(4)	0.4934	1.15509(7)	1.15051(8)	1.14612(7)	1.14195(5)
0.5486	1.14689(4)	1.14252(6)	1.13786(0)	1.13430(1)	0.5487	1.15042(8)	1.14591(5)	1.14163(6)	1.13756(2)
0.6486	1.13988(3)	1.13564(5)	1.13112(7)	1.12781(9)	0.6506	1.14192(1)	1.13757(5)	1.13349(7)	1.12961(1)
0.7501	1.13338(6)	1.12928(8)	1.12493(2)	1.12187(9)	0.7510	1.13365(4)	1.12950(0)	1.12561(6)	1.12192(4)
0.8489	1.12759(6)	1.12364(3)	1.11945(3)	1.11664(9)	0.8498	1.12561(3)	1.12162(5)	1.11793(8)	1.11444(1)
0.9400	1.12266(3)	1.11885(4)	1.11482(2)	1.11224(7)	0.9429	1.11810(0)	1.11418(3)	1.11070(0)	1.10738(7)

[a] TEG—triethylene glycol; DEG—diethylene glycol

Table 3 Fitting coefficients and standard deviation for least-squares representation of V_m^E (cm^3 . mol^{-1}) for glycols (1) + organic solvents (2) at T = (298.15, 303.15, 308.15 and 313.15) K

T (K)	a_0	a_1	a_2	σ
TEG[a] *(1) + Chlorobenzene (2)*				
298.15	−0.646	−0.038	−0.063	0.001
303.15	−0.622	−0.037	−0.067	0.001
308.15	−0.604	−0.038	−0.062	0.001
313.15	−0.587	−0.027	−0.074	0.001
TEG[a] *(1) + Bromobenzene (2)*				
298.15	1.644	−0.127	−0.343	0.001
303.15	1.718	−0.186	−0.445	0.001
308.15	1.829	−0.288	−0.688	0.001
313.15	1.907	−0.300	−0.713	0.001
TEG[a] *(1) + Nitrobenzene (2)*				
298.15	1.323	0.070	−0.160	0.001
303.15	1.431	0.071	−0.147	0.001
308.15	1.545	0.073	−0.154	0.001
313.15	1.635	0.078	−0.167	0.001
DEG[a] *(1) + Nitrobenzene (2)*				
298.15	1.022	−0.004	−0.065	0.001
303.15	1.088	−0.031	−0.283	0.001
308.15	1.119	−0.061	−0.217	0.001
313.15	1.152	−0.054	−0.246	0.001

[a] TEG—triethylene glycol; DEG—diethylene glycol

3.4 Relative Permittivities (ε_r)

The data of ε_r for the four binary mixtures at T = (298.15 and 308.15) K are also listed in Table 4.

3.5 Discussion

An understanding of the volumetric behavior of the glycol + chloro-, bromo-, and nitrobenzene mixtures must consider several factors. The observed small but negative V_m^E values in triethylene glycol + chlorobenzene mixtures indicate the predominance of structure-making effects in the form of weak Cl ··· H–O hydrogen bonding, Cl ··· O electron acceptor–donor interactions, and interstitial accommodation of chlorobenzene in associate structures of triethylene glycol, etc. However, such weak interactions are facilitated only at the expense of weakening of intermolecular hydrogen bonds within triethylene glycol molecules (and the later phenomenon would have contributed positively to V_m^E data). The replacement of the chloro-group by the bromo-group resulted in large expansion in the volumes. The decreased electronegativity and the increased molecular size (the molecular radius of cholorobenzene is 2.16 Å while it is 2.19 Å for bromobenzene at T = 298.15 K) of bromine are expected to play a major role in the observed positive V_m^E values. The same reason can be attributed for the positive V_m^E values for triethylene glycol and diethylene glycol + nitrobenzene mixtures. Nitrobenzene has a smaller radius of 2.17 Å as compared to bromoben-

Table 4 Experimental sound speeds (υ_{12}), dynamic viscosities (η), and relative permittivities (ε_r) for glycols (1) + organic solvents (2) at $T = (298.15 \text{ and } 308.15)$ K

υ_{12} (m · s^{-1})			η_{12} (mPa · s)		ε_r		υ_{12} (m · s^{-1})			η_{12} (mPa · s)		ε_r	
x_1	298.15 (K)	308.15 (K)	298.15 (K)	308.15 (K)	298.15 (K)	308.15 (K)	x_1	298.15 (K)	308.15 (K)	298.15 (K)	308.15 (K)	298.15 (K)	308.15 (K)
TEG[a] + *Chlorobenzene*							*TEG*[a] + *Bromobenzene*						
0.0473	1278	1239	0.941	0.808	6.950	6.658	0.0462	1179	1139	1.316	1.168	5.551	5.507
0.1490	1305	1272	1.491	1.172	9.590	9.082	0.1502	1217	1182	2.173	1.769	6.556	6.770
0.2493	1336	1305	2.307	1.682	12.058	11.318	0.2493	1256	1223	3.390	2.568	7.637	8.058
0.3495	1366	1338	3.504	2.400	14.331	13.363	0.3505	1299	1268	5.164	3.674	8.931	9.448
0.4390	1395	1369	5.014	3.283	16.168	15.013	0.4433	1340	1311	7.374	5.002	10.328	10.794
0.4734	1407	1381	5.733	3.699	16.817	15.601	0.4974	1365	1337	8.957	5.935	11.243	11.617
0.5509	1435	1410	7.695	4.829	18.169	16.831	0.5484	1388	1362	10.665	6.933	12.175	12.423
0.6512	1473	1451	11.088	6.787	19.679	18.233	0.6475	1435	1410	14.609	9.221	14.184	14.088
0.7493	1513	1489	15.579	9.422	20.899	19.417	0.7514	1487	1462	19.622	12.146	16.553	16.022
0.8483	1553	1530	21.583	13.054	21.894	20.459	0.8484	1537	1509	25.027	15.373	18.976	18.059
0.9428	1591	1566	28.992	17.737	22.657	21.347	0.9370	1580	1551	30.418	18.720	21.326	20.173
TEG[a] + *Nitrobenzene*							*DEG*[a] + *Nitrobenzene*						
0.0465	1470	1432	2.086	1.759	33.633	32.109	0.0441	1463	1426	1.987	1.691	34.505	32.889
0.1495	1486	1450	2.910	2.342	31.433	30.083	0.1493	1473	1437	2.565	2.115	33.817	32.250
0.2501	1502	1468	3.999	3.087	29.698	28.452	0.2505	1482	1446	3.312	2.642	33.219	31.693
0.3505	1518	1485	5.455	4.049	28.254	27.078	0.3502	1492	1458	4.303	3.310	32.706	31.197
0.4432	1532	1502	7.222	5.184	27.101	25.974	0.4438	1503	1469	5.550	4.117	32.296	30.780
0.4957	1541	1510	8.443	5.954	26.508	25.406	0.4934	1507	1475	6.373	4.632	32.108	30.577
0.5486	1550	1521	9.864	6.838	25.947	24.868	0.5487	1514	1484	7.457	5.293	31.920	30.366
0.6486	1565	1535	13.166	8.857	24.983	23.942	0.6506	1528	1498	10.036	6.804	31.630	30.018
0.7501	1581	1550	17.527	11.469	24.145	23.127	0.7510	1542	1511	13.583	8.774	31.401	29.721
0.8489	1594	1567	22.999	14.694	23.509	22.475	0.8498	1556	1524	18.470	11.343	31.210	29.472
0.9400	1604	1576	29.371	18.403	23.140	22.036	0.9429	1570	1541	24.890	14.535	31.037	29.271

[a] TEG—triethylene glycol; DEG—diethylene glycol

Table 5 Adjustable parameters for the correlation of mixture viscosities of glycols (1) + organic solvents (2) at $T = (298.15 \text{ and } 308.15)$ K

T in K	G_{12}	σ	M_{12}	M_{21}	σ
TEG[a] *(1) + Chlorobenzene (2)*					
298.15	0.880	0.001	2.452	1.186	0.001
308.15	0.256	0.001	1.897	0.756	0.001
TEG[a] *(1) + Bromobenzene (2)*					
298.15	1.650	0.001	2.726	1.469	0.001
308.15	1.110	0.001	2.209	1.807	0.001
TEG[a] *(1) + Nitrobenzene (2)*					
298.15	0.343	0.001	2.541	2.131	0.001
308.15	0.196	0.001	1.536	1.237	0.001
DEG[a] *(1) + Nitrobenzene (2)*					
298.15	−0.486	0.001	2.170	1.798	0.001
308.15	−0.335	0.001	1.206	0.975	0.001

[a] TEG—triethylene glycol; DEG—diethylene glycol

zene's 2.18 Å. In addition, the NO_2 group has a dipole structure and can interact relatively strongly compared to bromobenzene with triethylene glycol. Hence, V_m^E values for both the glycols + nitrobenzene mixtures are less positive compared to those for bromobenzene-containing mixtures. Interestingly, the triethylene glycol + bromobenzene, + nitrobenzene, and diethylene glycol + nitrobenzene systems are characterized by small but positive V_m^E values. This indicates that probably the structure-breaking effects are dominant in these systems.

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