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Density, Relative Permittivity, and Viscosity of Propylene Carbonate + Dimethoxyethane Mixtures from 25 °C to 125 °C

Josef Barthel,* Roland Neueder, and Hadumar Roch

Institut für Physikalische und Theoretische Chemie, Universität Regensburg, D-93040 Regensburg, Germany

Density, relative permittivity, and viscosity measurements on propylene carbonate + dimethoxyethane were made over the temperature range 25 °C to 125 °C at all mole fractions, completing the previous data for the temperature range −45 °C to 25 °C. New measuring equipment developed in our laboratory for measurements over the temperature range 20 °C to 180 °C is described.

Introduction

Mixtures of high-permittivity−high-viscosity solvents and low-viscosity−low-permittivity solvents are preferred solvents for various electrochemical applications requiring suited solvents for the production of nonaqueous aprotic electrolyte solutions of high conductivity. Primary and secondary batteries are well-known examples.

In this paper we report on density, viscosity, and relative permittivity measurements on mixtures of propylene carbonate (PC, $\epsilon = 64.96$, $\eta = 2.512 \times 10^{-3}$ Pa s at 25 °C) + dimethoxyethane (DME, $\epsilon = 7.03$, $\eta = 0.409 \times 10^{-3}$ Pa s at 25 °C) in the temperature range from 25 °C to 125 °C with the highly precise equipment of our laboratory[1] which was suitably modified for measuring the temperature dependence of solution properties at temperatures up to 180 °C. The low-temperature data from −45 °C to 25 °C are given in ref 2.

Experimental Section

Materials. Propylene carbonate (Fluka, purum, mp −49 °C, bp 241 °C) was dried over a molecular sieve (5 Å) for several days, boiled for 2 h in the presence of dried CaO, and then distilled at reduced pressure (≈2 mbar).[3] Nitrogen was bubbled through the solvent at 60 °C to remove the volatile impurities. The final distillation was carried out in a specially designed column[1] at reduced pressure (≈2 mbar) to yield a product with organic impurities less than 20 ppm (detected by gas chromatography) and a nondetectable water content (Karl Fischer titration). The specific conductivity was less than 2×10^{-8} S cm^{-1} (25 °C).

Dimethoxyethane (Fluka, purum, mp −69 °C, bp 85 °C) was initially passed through an Al_2O_3 column (acid Al_2O_3, Woelm Eschwege, W200), and then CaH_2 was added and the solvent was boiled for 24 h.[4] It was distilled and then refluxed for 2 days in the presence of $LiAlH_4$ (2.5 g per liter of DME). The final distillation under pure nitrogen over a packed column containing approximately 50 plates yields a product with no detectable organic impurities (gas chromatography) and a specific conductivity of less than 1 $\times 10^{-8}$ S cm^{-1} (25 °C). Water could not be detected by Karl Fischer titration.

The solvent mixtures were prepared by mass under purified nitrogen. Solvents and mixtures were stored in the dark in a glovebox.

Thermostat. The new high-temperature thermostat (20 °C to 180 °C) was constructed according to the principles of the previously described low-temperature thermostat (−60 °C to 35 °C)[1]. Temperature resistant materials and a suitable oil-bath mixture of almost constant viscosity in the high-temperature range were used to construct the thermostat assembly shown in Figure 1. The assembly consists of a measurement thermostat (MT) (content: 60 L of silicon oil) coupled to a prethermostat (PT) (cold bath) by means of a heat exchanger (HE). The amount of heat exchanged can be controlled with a valve (f). A highly efficient stirrer (b) allows vigorous stirring of the bath liquid. The prethermostat is cooled either with water or, at higher temperatures, with air.

A circular opening (a) in the cover plate of the MT permits the immersion of a measuring cell (permittivity) or the connection of external measuring cells (viscosity or density) via a circulating pump immersed in the thermostat. For viscosity and density measurements the temperature is determined in the external cells via a NTC-thermistor and the temperature of the thermostat is adjusted appropriately.

The high-temperature thermostat is capable of yielding a temperature reproducibility and short and long time deviations of $<10^{-3}$ K over the whole temperature range.

Permittivity Measurements. Temperature-dependent permittivity measurements were made by the use of a low-frequency (0.1−10 kHz) capacitance bridge (General Radio capacitance bridge, type 1616), equipped with a conductance balancing network, and a three-terminal dielectric cell[1] which is immersed in the thermostat.

The cell constant ($C_0 = 11.063$ pF, 25 °C) was determined from 25 °C to 140 °C in steps of 10 °C by measuring the capacitance of the cell filled with pure argon, for which the temperature and pressure-dependent data are available in the literature.[5−7] The temperature coefficient of the cell constant is $+2.0 \times 10^{-4}$ pF/K.

The relative permittivity of each sample was calculated from the ratio of the capacitances of the cell filled with the sample and the cell filled with dry argon. The measured data with an uncertainty of 0.2% are given in Table 1.

Viscosity Measurements. The temperature-dependent viscosities of Table 1 were determined with an Ubbelhode viscometer placed in a Dewar flask which was connected via a circulating pump with the thermostat. Calibration of the viscosimeter and evaluation of the sample viscosities, η, were obtained from the flow time of the liquids

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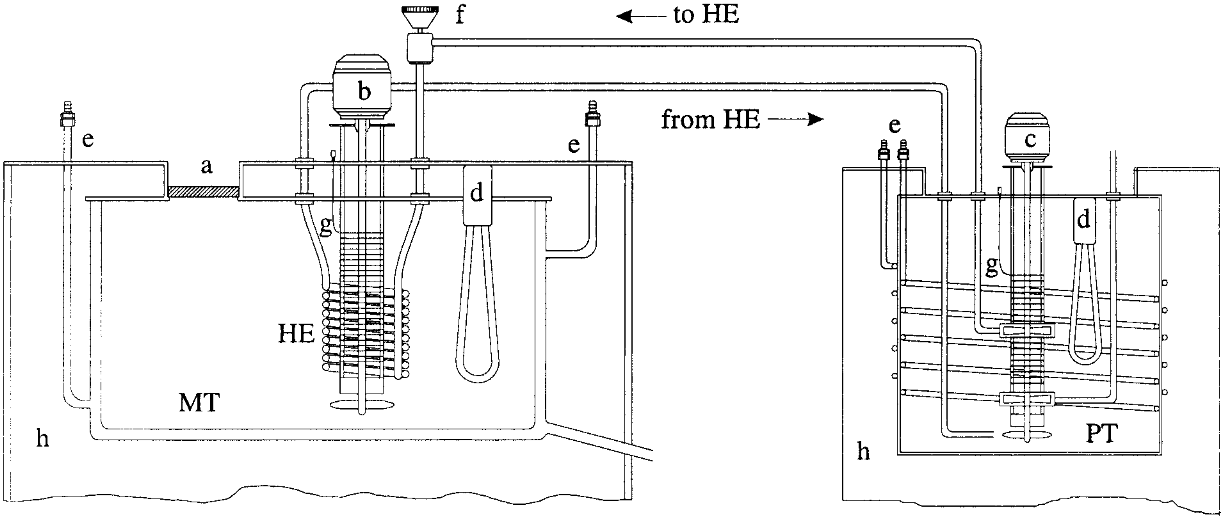

Figure 1. High-temperature thermostat (20 °C to 180 °C): MT, measurement thermostat; PT, prethermostat; HE, heat exchanger; a, circular opening in the cover plate for measuring cells or circulating pump; b, highly efficient bath stirrer; c, heat exchanger pump; d, auxiliary heater; e, auxiliary cooler; f, adjustable valve for heat exchanger; g, controlled heater; h, thermal insulation.

through the capillary. The flow time was detected by a control unit (Schott AVS/G) using photodiodes and optical fiber bundles for transmission of the signal to the viscometer. Each measurement was automatically repeated at least five times and yielded reproducibility of the flow time of less than 0.01%. The errors from calibration and temperature control yield an uncertainty of 0.2% of viscosity.

***Density Measurements*.** The densities were determined with the help of a vibrational tube densimeter (Paar, DMA 60, DMA 602 HT) by the method of Kratky et al.,[8] which relates the vibration time of the tube τ to the solution density d according to

$$\tau^2 = Ad + B \tag{1}$$

The characteristic constants of the vibrational tube A and B were determined by calibration based on density measurements of nitrogen and water in the temperature range from 25 °C to 95 °C.

The densimeter was connected via a circulating pump with the precision thermostat. The measuring cell with a temperature controlled to 10 °C less than the measuring temperature is situated in a thermostated box made from PTFE. With the help of this assembly the temperature fluctuations were less than 0.001 K and the temperature gradient in the cell was less than 0.001 K at 25 °C and less than 0.01 K at 135 °C.

Each measurement was repeated at least five times and yielded reproducibility of less than 1×10^{-5} g/cm^3. Due to the calibration uncertainty and temperature errors, accuracies of $\pm 2 \times 10^{-5}$ g/cm^3 near room temperature and of $\pm 3 \times 10^{-5}$ g/cm^3 at higher temperatures is estimated. The results are given in Table 1.

***Data Analysis*.** The dependence on composition of the physical properties P of a binary solvent mixture is well reproduced by a polynomial in the mole fractions x_i of the components 1 and 2

$$P = a_0 x_1 + a_1 x_2 + x_1 x_2 \sum_{j=0}^{m} a_{2+j} (x_1 - x_2)^j \tag{2}$$

Temperature dependence commonly is expressed with temperature-dependent coefficients a_k. The investigation

Table 1. Density, Relative Permittivity, and Viscosity of Propylene Carbonate (1) + Dimethoxyethane (2)

T/K	x_1	d/kg m^{-3}	x_1	ϵ	x_1	$10^3\eta$/Pa s
298.15	0.0	861.09	0.0	7.03	0.0	0.4089
	0.2769	949.68	0.1472	14.37	0.2217	0.5911
	0.3383	969.59	0.4064	28.40	0.3749	0.7628
	0.5492	1039.50	0.6012	39.56	0.5841	1.0990
	0.6813	1084.56	0.6944	45.31	0.7836	1.5940
	1.0	1199.93	1.0	64.96	1.0	2.5120
308.15	0.0	850.01	0.0	6.71	0.0	0.3659
	0.2769	939.21	0.1472	13.69	0.2217	0.5265
	0.3383	959.13	0.4064	27.16	0.3749	0.6716
	0.5492	1029.01	0.6012	38.05	0.5841	0.9453
	0.6813	1075.01	0.6944	43.49	0.7836	1.3560
	1.0	1189.16	1.0	62.63	1.0	2.0860
318.15	0.0	838.85	0.0	6.41	0.0	0.3302
	0.2769	928.52	0.1472	13.04	0.2217	0.4711
	0.3383	948.69	0.4064	25.99	0.3749	0.5956
	0.5492	1018.84	0.6012	36.51	0.5841	0.8342
	0.6813	1064.50	0.6944	41.76	0.7836	1.1680
	1.0	1178.71	1.0	60.24	1.0	1.7500
328.15	0.0	827.54	0.0	6.13	0.0	0.2994
	0.2769	917.63	0.1472	12.43	0.2217	0.4227
	0.3383	937.99	0.4064	24.84	0.3749	0.5323
	0.5492	1008.15	0.6012	35.02	0.5841	0.7372
	0.6813	1053.53	0.6944	40.13	0.7836	1.0180
	1.0	1168.16	1.0	58.04	1.0	1.4980
338.15	0.0	815.83	0.0	5.86	0.0	0.2724
	0.2769	906.64	0.1472	11.85	0.2217	0.3821
	0.3383	926.86	0.4064	23.77	0.3749	0.4795
	0.5492	997.28	0.6012	33.59	0.5841	0.6587
	0.6813	1042.69	0.6944	38.55	0.7836	0.8990
	1.0	1157.53	1.0	55.91	1.0	1.3010
348.15	0.0	804.15	0.0	5.62	0.0	0.2492
	0.2769	895.57	0.1472	11.30	0.2217	0.3475
	0.3383	915.86	0.4064	22.73	0.3749	0.4348
	0.5492	986.57	0.6012	32.24	0.5841	0.5926
	0.6813	1032.12	0.6944	37.04	0.7836	0.8017
	1.0	1146.96	1.0	53.94	1.0	1.1440
358.15	0.2769	884.26	0.1472	10.80	0.2217	0.3167
	0.3383	904.49	0.4064	21.76	0.3749	0.3964
	0.5492	975.50	0.6012	30.93	0.5841	0.5382
	0.6813	1022.24	0.6944	35.59	0.7836	0.7247
	1.0	1136.33	1.0	51.93	1.0	1.0140
368.15	0.5492	964.35	0.4064	20.82	0.3749	0.3615
	0.6813	1013.58	0.6012	29.68	0.5841	0.4917
	1.0	1125.71	0.6944	34.23	0.7836	0.6550
			1.0	50.08	1.0	0.9067
378.15			0.6012	28.48	0.5841	0.4551
			0.6944	32.85	0.7836	0.5977
			1.0	48.34	1.0	0.8168
388.15					0.7836	0.5491
					1.0	0.7403
398.15					0.7836	0.5074
					1.0	0.6748

Table 2. Coefficients of Eqs 3 and 4 for Propylene Carbonate (1) + Dimethoxyethane (2)[a]

	d/kg m^{-3}	ϵ	ln(η/Pa s)
$b_0^{(0)}$	0.157 532 61 × 10^4	−0.993 481 00 × 10^6	0.112 354 10 × 10^7
$b_1^{(0)}$	−1.606 930 02	0.232 738 00 × 10^5	−0.789 077 50 × 10^4
$b_2^{(0)}$	0.167 289 88 × 10^{-2}	13.931 000 0	15.049 910 0
$b_3^{(0)}$	−0.169 916 07 × 10^{-5}	−0.053 405 910	−0.024 188 22
$b_0^{(1)}$	0.118 901 46 × 10^4	0.125 338 73 × 10^6	0.216 637 00 × 10^6
$b_1^{(1)}$	−1.305 941 14	0.182 463 90 × 10^4	−0.120 847 00 × 10^4
$b_2^{(1)}$	0.138 621 40 × 10^{-2}	0.429 465 00	−3.629 450 00
$b_3^{(1)}$	−0.232 934 37 × 10^{-5}	−0.311 855 77 × 10^{-2}	−0.857 329 00 × 10^{-2}
$b_0^{(2)}$	−0.140 911 54 × 10^4	−0.149 381 61 × 10^8	−0.389 287 03 × 10^7
$b_1^{(2)}$	11.983 218 0	0.141 586 47 × 10^6	0.351 193 07 × 10^5
$b_2^{(2)}$	−0.034 841 12	−0.447 254 48 × 10^3	−0.106 565 01 × 10^3
$b_3^{(2)}$	0.340 772 82 × 10^{-4}	0.440 400 64	0.108 304 65
$b_0^{(3)}$	−0.200 384 96 × 10^5	0.211 199 07 × 10^8	0.220 329 32 × 10^7
$b_1^{(3)}$	0.185 147 42 × 10^3	−0.200 147 47 × 10^6	−0.192 289 65 × 10^5
$b_2^{(3)}$	−0.569 236 90	0.625 239 39 × 10^3	55.211 658 6
$b_3^{(3)}$	0.582 115 94 × 10^{-3}	−0.642 350 89	−0.052 460 969

[a] Mole fraction x: x_1. Coefficients $b_i^{(0)}$ obtained from pure propylene carbonate data: density, refs 10−13; relative permittivity, refs 13−16; viscosity, refs 10, 11, and 14. Coefficients $b_i^{(1)}$ obtained from pure dimethoxyethane data: density, refs 2 and 17−22; relative permittivity, ref 22; viscosity, refs 2 and 20.

of the temperature dependence of the densities of a large variety of liquids leads to the polynomial

$$a_k(T) = b_0^{(k)} + b_1^{(k)}T + b_2^{(k)}T^2 + b_3^{(k)}T^3 \tag{3}$$

For relative permittivity and the logarithm of the viscosity, a polynomial of the type

$$a_k(T) = b_0^{(k)}T^{-2} + b_1^{(k)}T^{-1} + b_2^{(k)} + b_3^{(k)}T \tag{4}$$

is more suited.[9]

These equations are useful to represent the properties of liquid mixtures over the whole concentration range (mole fraction x) and wide temperature ranges. It is appropriate to combine them in the form

$$P(x,T) = a_0(T)x + a_1(T)(1 - x) + a_2(T)x(1 - x) + a_3(T)x(1 - x)(2x - 1) \tag{5}$$

where $P(x,T)$ is the density $d(x,T)$, the relative permittivity $\epsilon(x,T)$, or the logarithm of viscosity ln $\eta(x,T)$.

At x equal to zero and unity, the temperature-dependent properties of the pure solvent components 1 and 2 of the mixture are represented by the coefficients a_0 and a_1. The respective coefficients $b_i^{(0)}$ and $b_i^{(1)}$ for the pure solvents can be found in the literature; see the footnote of Table 2. The coefficients $b_i^{(2)}$ and $b_i^{(3)}$ were obtained from the experimental data of Table 1 by use of a least-squares fit and are also given in Table 2. With these coefficients the solvent properties can be reproduced over the whole range of solvent composition within the temperature range from 298 K to 348 K at a precision of 0.05% (density), 0.1% (permittivity), and 0.2% (viscosity). For pure propylene carbonate and propylene carbonate rich mixtures with high boiling points, the high-temperature limits of these coefficients are 368 K (density), 378 K (permittivity), and 398 K (viscosity).

Discussion

In Figure 2 the relative deviations of the calculated densities, relative permittivities, and viscosities from various experimental sources at different temperatures are plotted as a function of the mole fraction of propylene carbonate.

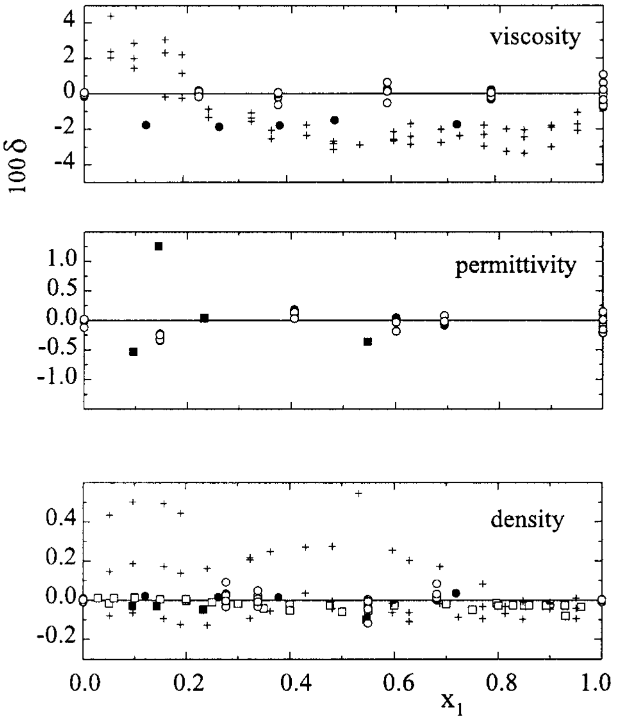

Figure 2. Relative deviations δ of experimental data (○, this paper; ●, ref 2; □, ref 23; ■, ref 24; +, ref 25) from values calculated with parameters from Table 2, where $\delta = y_{\text{exp}} - y_{\text{calc}}/y_{\text{exp}}$.

Reliable temperature- and composition-dependent density, permittivity, and viscosity data are needed as the input parameters of a wide variety of property equations of solutions. They also are the base of empirical studies of liquid structure properties and their influence on kinetic, spectroscopic, dielectric, and transport phenomena. A detailed study on the density and viscosity of propylene carbonate + dimethoxyethane mixtures at temperatures from −45 °C to 25 °C is given in refs 2 and 26. Dielectric properties require for a deeper understanding frequency-

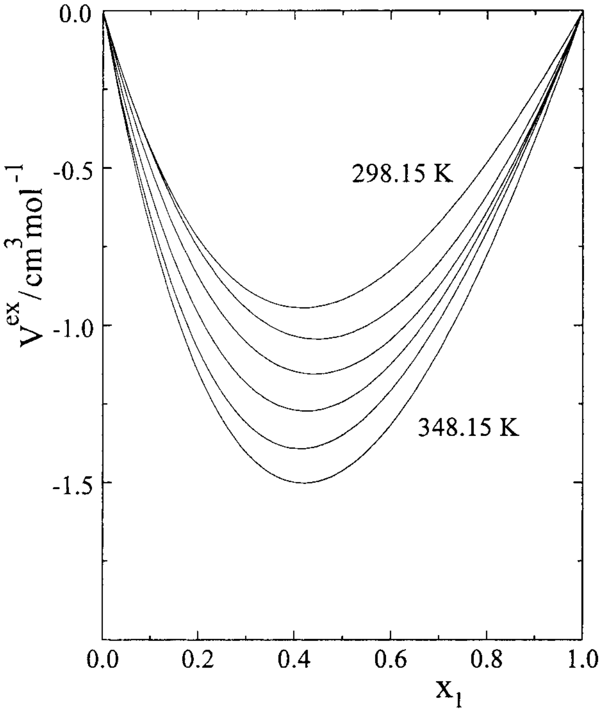

Figure 3. Molar excess volume of propylene carbonate + dimethoxyethane mixtures.

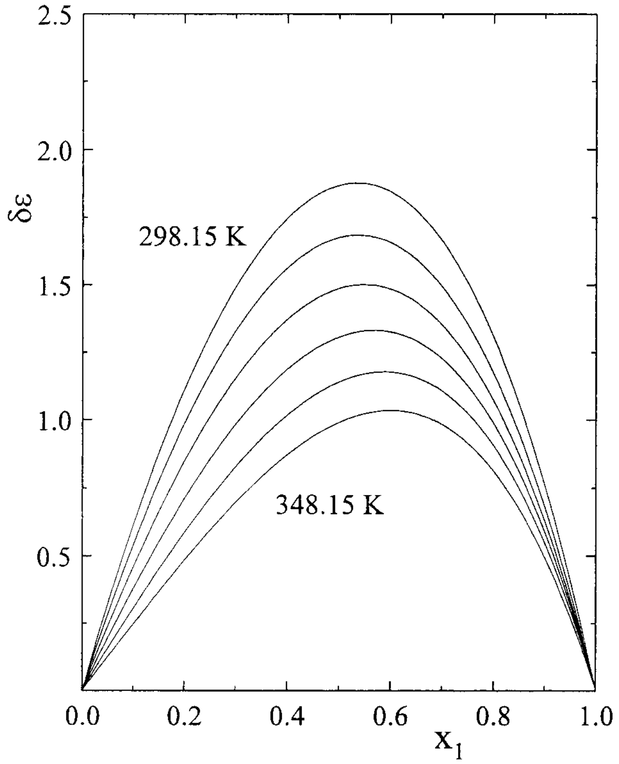

Figure 4. Deviation $\delta\epsilon$ of experimental relative permittivity of propylene carbonate + dimethoxyethane mixtures from eq 7.

dependent data,[27] which can be found for propylene carbonate, dimethoxyethane, and propylene carbonate + dimethoxyethane mixtures in ref 28.

For practical purposes isothermal excess properties, $P^{ex} = P - P^{id}$, are in use which can be calculated with the help of the parameters of Table 2 at different temperatures for all solvent compositions; see Figures 3−5. In the following equations the properties of pure components are indicated by a superscript *.

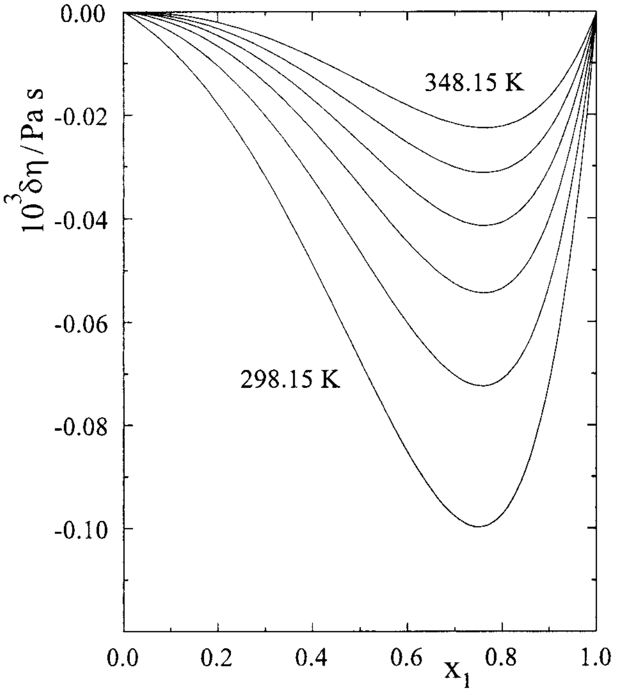

Figure 5. Deviation $\delta\eta$ of experimental viscosity of propylene carbonate + dimethoxyethane mixtures from eq 8.

The ideality of density is based on the thermodynamic expression of the ideal molar volume of a mixture

$$V^{id} = x_1 V_1^* + x_2 V_2^* \tag{6}$$

The excess molar volumes of the studied system are represented in Figure 3. For the calculation of the deviation from the ideal additivity of permittivities in Figure 4, we used the additivity of molar polarization according to a simplified equation

$$\left(\frac{\epsilon - 1}{d}\right)^{id} = x_1 \frac{\epsilon_1^* - 1}{d_1^*} + x_2 \frac{\epsilon_2^* - 1}{d_2^*} \tag{7}$$

The ideal viscosity underlying the deviations shown in Figure 5 is assumed to follow the additivity law

$$\ln \eta^{id} = x_1 \ln \eta_1^* + x_2 \ln \eta_2^* \tag{8}$$

The deviations presented in Figures 3−5 are useful for the study of arbitrary properties in mixed solvents by empirical correlations with solvent density, permittivity, and viscosity. It is interesting to note that for propylene carbonate + dimethoxyethane mixtures only rather small deviations occur from the values calculated with the help of eqs 6−8 of maximum 1% for density, 4% for permittivity, and 5% for viscosity.

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