

Densities and Viscosities of 1,1,1-Trichloroethane with 13 Different Solvents at 298.15 K

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The aim of this investigation is to study the behavior of 1,1,1-trichloroethane as a component of binary mixtures. Density and kinematic viscosity were measured at 298.15 K and atmospheric pressure with a vibrating tube densimeter and an Ubbelohde capillary viscometer, respectively, for binary mixtures of 1,1,1-trichloroethane with 13 solvents of different chemical nature: methyl and ethyl acetate, acetone, butanone, cyclohexanone, 2-methyltetrahydrofuran, dipropyl ether, 1,4-dioxane, nitromethane, 1-hexanol, 1-methoxy-2-propanol, α,α,α -trichlorotoluene and α,α,α -trifluorotoluene. The dynamic viscosity, excess volume, and viscosity deviation function were obtained from the experimental results. A polynomial expression was employed to correlate excess volume data, and the Cao et al. model (UNIMOD) was used to correlate mixture viscosity. Parameters for these models are reported.

Introduction

Group contribution methods are powerful tools for the prediction of thermodynamic and transport properties of multicomponent mixtures, in particular the excess volume and viscosity deviation function. The knowledge of the group contribution parameters of the solvents is useful in formulating solvent mixtures for specific applications. In this context, a thorough characterization of the groups CCl_3- and CCl_3CH_3 is important, since among the chlorinated solvents of industrial interest 1,1,1-trichloroethane plays an important role, owing to its high threshold limit value (TLV) in air: 350 ppm, 1900 $\text{mg}\cdot\text{m}^{-3}$ (Riddick et al., 1986). Density, kinematic and dynamic viscosity, excess volume, and viscosity deviation function data for mixtures of 1,1,1-trichloroethane with paraffins and cycloparaffins are already available (De Lorenzi et al., 1994). To extend the characterization of the behavior of 1,1,1-trichloroethane as a component of binary mixtures, the same data were evaluated for mixtures of 1,1,1-trichloroethane and 13 solvents. Solvents were selected with the aim to bring out interaction effects between different chemical groups and the 1,1,1-trichloroethane molecules, which add to the effects given by the dimension and shape of the mixed solvents. They are aliphatic esters and ketones (methyl and ethyl acetate, acetone, butanone), cyclic ketones (cyclohexanone), aliphatic and cyclic ethers (dipropyl ether, 2-methyltetrahydrofuran, 1,4-dioxane), nitromethane, aliphatic alcohols and alcohol-ethers (1-hexanol, 1-methoxy-2-propanol), α,α,α -trichlorotoluene, and α,α,α -trifluorotoluene.

In general, models suitable to predict thermodynamic properties of mixtures borrow the concept that mixture thermodynamic properties can be written as a sum of an ideal term and a perturbation (excess) term. In equilibrium thermodynamics the model for the ideal term is exactly defined. Indeed, as regards volume and density, ideal mixing volumes and excess volumes are thermodynamically well defined quantities. As regards viscosity the situation is more complicated. Indeed, besides some useful, empirical approaches (Grunberg and Nissan, 1949; Mc Allister, 1960), the ideal mixing viscosity is not a defined quantity.

Several different models have been put forward to describe it (Kendall and Monroe, 1917; Reed and Taylor, 1959; Wei and Rowley, 1989). Once a model is adopted, however, the perturbation term can be derived on the basis of the corresponding state approach (Teja and Rice, 1981), the group contribution method (Wu, 1986), or mixture thermodynamic properties (Bloomfield and Dewan, 1971). Mention is made to the approach of Wei and Rowley, who tried to draw out a connection between the excess shear viscosity and thermodynamic properties, on the basis of the local composition concept (Wei and Rowley, 1989). Among the group contribution methods, a promising approach has been more recently developed by Cao et al., the UNIMOD model (Cao et al., 1992, 1993a,b). It combines the Eyring theory of viscosity and the local composition concept. It is suitable for application to both pure components and mixtures. A detailed description of the model is given in the original papers. This model gives the advantage that no ternary, or higher, parameters are needed for describing the multicomponent mixture behavior.

The experiments reported in this paper aim at extending the UNIMOD parameter table, including the functional group parameter for the 1,1,1-trichloroethane molecule.

Experimental Section

Materials. All the solvents used were from Aldrich, purity >99 mol %, used as received, without further treatment. The purities of nitrogen and water employed for the calibration of the instruments were 99.9999 and 99.9 mol %, respectively.

Apparatus. The same equipment used and described by De Lorenzi et al. (1994) was employed here. Densities were measured by a vibrating tube digital densimeter, Anton Paar, model DMA 602H-DMA 60, connected with a Heterofrig (Heto Birkerød) constant-temperature bath circulator, with a precision of ± 0.01 K. Calibration of the apparatus and working procedures are described elsewhere in more detail (Fermeglia and Lapasin, 1988; Fermeglia et al., 1990). The density estimated precision is higher than 3×10^{-5} $\text{g}\cdot\text{cm}^{-3}$.

Viscosities were measured by means of an Ubbelohde suspended-level capillary viscometer, coupled with a Schott electronic timer, AVS 300, with a precision of ± 0.01 s, and a Schott TA 20-T 100 automatic diluting device. The

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Table 1. Comparison between Measured and Literature Data for Density ρ and Viscosity η of Pure Components at 298.15 K

solvent	$\rho/(\text{gcm}^{-3})$			$\eta/(\text{mPa}\cdot\text{s})$		
	this work	lit. ^a	other lit.	this work	lit. ^a	other lit.
1,1,1-trichloroethane	1.329 29 ^m	1.329 9	1.330 3 ^c	0.7917	0.795	0.793 ⁱ
1,4-dioxane	1.027 60	1.027 97	1.027 7 ^e	1.1875		1.149 ^e 1.177 ⁱ
1-methoxy-2-propanol	0.916 50			1.6714		
2-methyltetrahydrofuran	0.848 82			0.4753		
α,α,α -trichlorotoluene	1.366 97			2.1801		
α,α,α -trifluorotoluene	1.180 98	1.181 29		0.5524		
acetone	0.784 59	0.784 4	0.784 7 ^b 0.784 4 ^c 0.784 3 ^l 0.785 ^j 0.785 6 ⁱ	0.3049	0.3029	0.3025 ^c 0.301 ^b 0.302 ^l 0.306 ⁱ
butanone	0.801 50	0.7997	0.799 5 ^e 0.799 7 ^j 0.799 4 ⁱ	0.3840	0.378	0.408 ^e 0.475 ^d 0.405 ⁱ
cyclohexanone	0.942 51		0.942 01 ^e 0.942 07 ^j 0.942 5 ⁱ 0.949 5 ^k	2.0027		1.819 ^e 2.017 ⁱ
dipropyl ether	0.742 28	0.741 9		0.4024		0.396 ⁱ
ethyl acetate	0.894 14	0.894 55	0.894 2 ⁱ	0.4277	0.426	0.439 ^d 0.424 ^j 0.423 ⁱ
methyl acetate	0.926 83	0.927 9	0.926 8 ^c 0.927 3 ⁱ	0.3670	0.364	0.3545 ^c 0.364 ⁱ
1-hexanol	0.815 24	0.815 34	0.815 3 ^f 0.815 05 ^g	4.4997	4.592	4.578 ⁱ
nitromethane	1.130 11	1.131 28	1.128 6 ⁱ 1.131 0 ^h	0.6205	0.614	0.627 ^d 0.630 ⁱ 0.61 ^k

^a Riddick et al. (1986). ^b Noda et al. (1982). ^c Wei and Rowley (1984). ^d Aminabhavi et al. (1987). ^e Rao and Reddy (1988). ^f Tejraj et al. (1993). ^g Amigo et al. (1993). ^h Yadava and Singh (1994). ⁱ CRC Handbook (1992–1993). ^j Timmermanns (1995). ^k Kirk-Otmer (1978). ^l Fort and Moore (1966). ^m Average value from the 13 1,1,1-trichloroethane measurements in the different systems.

thermostat was a Haake F3 instrument, with a precision, experimentally verified, of ± 0.02 K. Calibration of the apparatus and working procedures are described elsewhere (Fermeglia and Lapasin, 1988; Fermeglia et al., 1990). The estimated precision in the kinematic viscosity measurement is approximately $1 \times 10^{-10} \text{ m}^2\cdot\text{s}^{-1}$.

Nitrogen and bidistilled water were employed to calibrate the densimeter. For the calibration of the viscometer the Poulten Selfe & Lee standard oil K5 was employed.

Procedure. The same procedure used and described by De Lorenzi et al. (1994) was adopted here. For further details see Fermeglia and Lapasin (1988) and Fermeglia et al. (1990). A brief description is given here. The mixtures for the density measurements were prepared by mass. A Mettler balance with a precision of 3×10^{-5} g and air-tight bottles were used. The possible error in the mole fraction is estimated to be lower than 5×10^{-5} . Repeated experiments of V^E indicated the reproducibility to be around $2 \times 10^{-3} \text{ g}\cdot\text{cm}^{-3}$.

The mixtures for the kinematic viscosity measurements were obtained by volume. The calibration of the Schott TA 20-T 100 automatic diluting device indicated that the possible error in the mole fraction is approximately 2×10^{-3} (De Lorenzi et al., 1994).

Results and Correlations

All the measurements were carried out at 298.15 K and at atmospheric pressure. Table 1 reports a comparison between literature values and the pure component values measured in this work. The data of Table 1 generally agree with the literature data.

Experimental data, density ρ and kinematic viscosity ν , for the 13 binary mixtures are reported in Table 2 together with the dynamic viscosity η , excess volumes V^E , and

viscosity deviation function $\Delta\nu$, which are derived quantities and were calculated with the same procedure followed by De Lorenzi et al. (1994), i.e., by means of the following equations, respectively:

$$\eta = \nu\rho \quad (1)$$

$$V^E = \frac{x_1M_1 + x_2M_2}{\rho} - \frac{x_1M_1}{\rho_1} - \frac{x_2M_2}{\rho_2} \quad (2)$$

$$\Delta\nu = \nu - \nu^m$$

$$\ln(\nu^m M) = \sum x_j \ln(\nu_j M_j) \quad (3)$$

where x_j is the mole fraction of component j , M_j the molecular weight of component j , M the molecular weight of the mixture, ρ_j the density of component j , ν_j the kinematic viscosity of component j , and ν^m the viscosity as defined in eq 3.

The composition dependence of the excess volume was fit to the following equation:

$$V_{\text{calc}}^E / (\text{cm}^3 \cdot \text{mol}^{-1}) = x_1 x_2 \sum_{j=0}^{n-1} a_j (x_1 - x_2)^j \quad (4)$$

where a_j are parameters and n is the number of polynomial coefficients. The coefficients have been calculated by minimizing an objective function written in terms of the sum of the squares of the differences between experimental and calculated values, divided by the experimental values.

The parameters a_j for the mixtures are reported in Table 3, along with the standard deviations of the fit. The most appropriate order of the polynomial eq 4 to be used was

Table 2 (Continued)

x	$\rho/(\text{g}\cdot\text{cm}^{-3})$	$V^E/(\text{cm}^3\cdot\text{mol}^{-1})$	x	$\nu/(10^{-6}\text{m}^2\cdot\text{s}^{-1})$	$\Delta\nu/(10^{-6}\text{m}^2\cdot\text{s}^{-1})$	$\eta/(\text{mPa}\cdot\text{s})$	x	$\rho/(\text{g}\cdot\text{cm}^{-3})$	$V^E/(\text{cm}^3\cdot\text{mol}^{-1})$	x	$\nu/(10^{-6}\text{m}^2\cdot\text{s}^{-1})$	$\Delta\nu/(10^{-6}\text{m}^2\cdot\text{s}^{-1})$	$\eta/(\text{mPa}\cdot\text{s})$
x 1,1,1-Trichloroethane + (1-x) Methyl Acetate													
0.0000	0.926 83	0.000	0.000	0.3959	0.0000	0.3670	0.9217	1.302 84	0.072	0.499	0.4471	-0.0185	0.5130
0.0997	0.974 94	0.087	0.100	0.4005	-0.0046	0.3907	1.0000	1.329 27	0.000	0.500	0.4471	-0.0188	0.5132
0.1955	1.019 07	0.156	0.101	0.4003	-0.0050	0.3907				0.500	0.4455	-0.0184	0.5114
0.2944	1.062 76	0.200	0.198	0.4075	-0.0090	0.4156				0.599	0.4656	-0.0193	0.5525
0.3931	1.104 58	0.225	0.199	0.4074	-0.0092	0.4157				0.599	0.4672	-0.0197	0.5545
0.5145	1.153 57	0.241	0.297	0.4174	-0.0129	0.4438				0.699	0.4920	-0.0167	0.6024
0.5936	1.184 36	0.230	0.300	0.4179	-0.0129	0.4450				0.799	0.5216	-0.0135	0.6574
0.6982	1.223 59	0.205	0.398	0.4306	-0.0162	0.4763				0.901	0.5541	-0.0103	0.7178
0.8039	1.261 83	0.153	0.401	0.4311	-0.0162	0.4774				1.000	0.5972	0.0000	0.5943
x 1,1,1-Trichloroethane + (1-x) 1-Hexanol													
0.0000	0.815 24	0.000	0.000	5.5196	0.0000	4.4997	0.5210	1.052 78	0.186	0.701	1.0342	-0.1173	1.1883
0.0923	0.853 85	0.015	0.101	4.4570	0.0605	3.8239	0.6111	1.099 69	0.194	0.800	0.8313	-0.0926	1.0021
0.1926	0.897 30	0.048	0.200	3.5420	0.0250	3.1885	0.7131	1.155 23	0.196	1.000	0.5956	0.0000	0.7917
0.2149	0.907 22	0.057	0.301	2.7823	-0.0208	2.6311	0.8042	1.207 59	0.157				
0.2919	0.941 89	0.113	0.402	2.1565	-0.0810	2.1423	0.8986	1.264 20	0.122				
0.3296	0.959 33	0.137	0.502	1.6718	-0.1185	1.7438	1.0000	1.329 21	0.000				
0.4164	1.000 84	0.146	0.601	1.3066	-0.1295	1.4304							
x 1,1,1-Trichloroethane + (1-x) Nitromethane													
0.0000	1.130 11	0.0000	0.000	0.5490	0.0000	0.6205	0.9109	1.318 89	0.0321	0.551	0.5213	-0.0131	0.6609
0.0908	1.160 99	0.0143	0.101	0.5250	-0.0099	0.6114	1.0000	1.329 29	0.0000	0.600	0.5253	-0.0132	0.6702
0.1783	1.186 61	0.0393	0.201	0.5140	-0.0134	0.6128				0.600	0.5253	-0.0132	0.6702
0.1893	1.189 61	0.0399	0.251	0.5114	-0.0140	0.6165				0.650	0.5301	-0.0134	0.6803
0.2878	1.214 64	0.0495	0.301	0.5109	-0.0138	0.6220				0.651	0.5301	-0.0134	0.6805
0.3863	1.236 39	0.0640	0.351	0.5113	-0.0136	0.6284				0.700	0.5355	-0.0135	0.6912
0.4897	1.256 58	0.0695	0.400	0.5127	-0.0133	0.6356				0.749	0.5418	-0.0133	0.7030
0.5775	1.271 90	0.0712	0.451	0.5149	-0.0131	0.6435				0.812	0.5515	-0.0124	0.7202
0.7134	1.292 87	0.0679	0.500	0.5178	-0.0130	0.6518				0.867	0.5615	-0.0107	0.7375
0.8176	1.307 17	0.0545	0.550	0.5212	-0.0131	0.6607				1.000	0.5956	0.0000	0.7917

Table 3. Values of the Coefficients and Relevant Standard Deviations for Equations 4 and 6

coefficients of eqs 4 and 6	1,1,1-trichloroethane +						
	1,4-dioxane	1-methoxy-2-propanol	2-methyl tetrahydrofuran	α,α,α -trifluorotoluene	α,α,α -trichlorotoluene	acetone	
Equation 4							
a_0	-0.861 47	1.411 82	-1.229 44	1.752 13	-1.650 98	0.328 76	
a_1	-0.105 60	0.744 76	0.113 78	0.148 66	-0.595 16	0.050 53	
a_2	-0.014 45	0.302 31	-0.008 65	0.027 01	-0.139 83	0.135 09	
a_3		0.861 82		0.013 12		0.144 70	
a_4		0.656 16					
$\sigma/(\text{cm}^3\cdot\text{mol}^{-1})$	0.001 52	0.008 03	0.002 75	0.001 53	0.010 9	0.003 22	
Equation 6							
$\Delta U_{12}/\text{K}$	-15.66					165.51	
$\Delta U_{21}/\text{K}$	26.60					-132.75	
$\sigma/(10^{-6}\text{m}^2\cdot\text{s}^{-1})$	0.0004 31					0.001 23	
coefficients of eqs 4 and 6	1,1,1-trichloroethane +						
	butanone	cyclohexanone	dipropyl ether	ethyl acetate	methyl acetate	1-hexanol	nitromethane
Equation 4							
a_0	-0.192 08	-1.785 18	-1.045 93	0.314 54	0.968 66	0.732 73	0.294 85
a_1	0.042 37	-0.261 16	0.115 24	0.023 54	0.006 79	0.548 82	0.109 24
a_2	0.008 61	-0.155 23	0.025 24	-0.003 07		-0.169 37	-0.011 68
a_3							
a_4							
$\sigma/(\text{cm}^3\cdot\text{mol}^{-1})$	0.002 93	0.011 5	0.004 79	0.001 54	0.003 33	0.013 5	0.002 80
Equation 6							
$\Delta U_{12}/\text{K}$	120.31	-63.57	80.71	112.86	160.58	272.54	279.49
$\Delta U_{21}/\text{K}$	-96.73	96.53	-55.58	-91.71	-127.80	-188.51	-323.14
$\sigma/(10^{-6}\text{m}^2\cdot\text{s}^{-1})$	0.001 08	0.002 47	0.0009 19	0.001 14	0.001 32	0.0169	0.004 42

determined by means of the F test at a 95% confidence level. The standard deviation for the excess volumes ($\text{cm}^3\cdot\text{mol}^{-1}$) reported in Table 3 was evaluated with the following equation:

$$\sigma(V^E) = \left[\sum \frac{(V_{\text{calc}}^E - V_{\text{exp}}^E)^2}{N - n} \right]^{1/2} \quad (5)$$

where N is the number of points and n the number of parameters.

The UNIMOD model was used for the regression of the experimental kinematic viscosity values when pure solvent parameters, necessary for the calculation, were available. The UNIMOD model combines the Eyring theory of viscosity and the local composition concept and is applicable to both pure components and mixtures. A detailed description of the model together with the discussion of the relevant drawbacks is presented in the original papers.

The pure component parameters required for the description of the mixture viscosity were taken from the

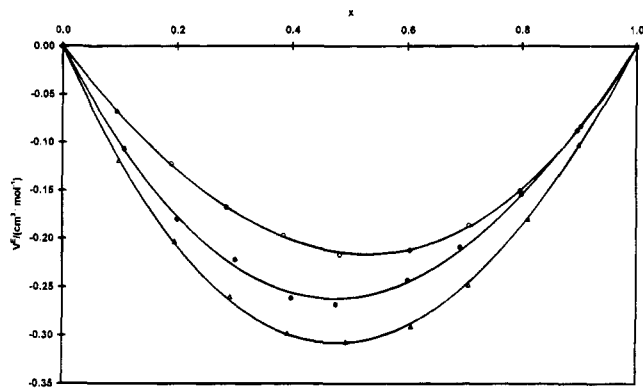


Figure 1. Excess volume for x 1,1,1-trichloroethane + $(1-x)$ (○) 1,4-dioxane, (●) dipropyl ether, and (Δ) 2-methyltetrahydrofuran.

compilation of UNIMOD. The two parameters $\Delta U_{12} = (U_{12} - U_{22})/R$ and $\Delta U_{21} = (U_{21} - U_{11})/R$ (K) in the following equations have been estimated by fitting the experimental results:

$$\ln(\eta V) = \sum_{i=1}^n x_i \ln(\eta_i V_i) - \sum_{i=1}^n q_i n_i x_i \sum_{j=1}^n \vartheta_{ji} \ln \tau_{ji} \quad (6)$$

$$\tau_{ij} = \exp \left\{ -\frac{z}{2} \left[\frac{U_{ji} - U_{ii}}{RT} \right] \right\} \quad (7)$$

An explanation of all the terms in eqs 6 and 7 is beyond the scope of this work; consequently, reference is made to Cao et al. (1992, 1993a,b). V is the molar volume of the mixture and V_j the molar volume of component j . The binary parameters ΔU_{12} and ΔU_{21} are the interaction potential energy parameters.

The standard deviations obtained in the fitting are generally satisfactory (see Table 3). The viscosity standard deviation ($10^{-6} \text{ m}^2 \text{ s}^{-1}$) reported in Table 3 was calculated with the equation below:

$$\sigma_v = \left[\frac{\sum (v^{\text{cal}} - v^{\text{exp}})^2}{N - 2} \right]^{1/2} \quad (8)$$

The UNIMOD model was not applied to systems based on 2-methyltetrahydrofuran, 1-methoxy-2-propanol, α,α,α -trichlorotoluene, and α,α,α -trifluorotoluene, since UNIMOD pure component parameters for these solvents were not available. Since UNIMOD pure component parameters are temperature dependent, only an application at 298.15 K would be possible; no reasonable extrapolation to other temperatures could be expected. A determination of the UNIMOD parameters for the above-mentioned pure components is expedient to allow the application of the model to be extended also to the systems containing these pure components.

Excess volumes are plotted in Figures 1–4. Here, the continuous lines were obtained by applying eq 4 with the parameters reported in Table 3. Excess volumes of the ethers examined are all negative (Figure 1). For ketones there is a trend from slight positive to increasing negative excess volumes with increasing molecular volume (Figure 2). The rigid, planar structure of the benzene ring, coupled with the steric encumbrance of the $-\text{CF}_3$ group, may explain the high positive excess volumes of the α,α,α -trifluorotoluene system. This effect is overturned for the α,α,α -trichlorotoluene system, where strong interactions between the $-\text{CCl}_3$ groups, coupled with a closer arrange-

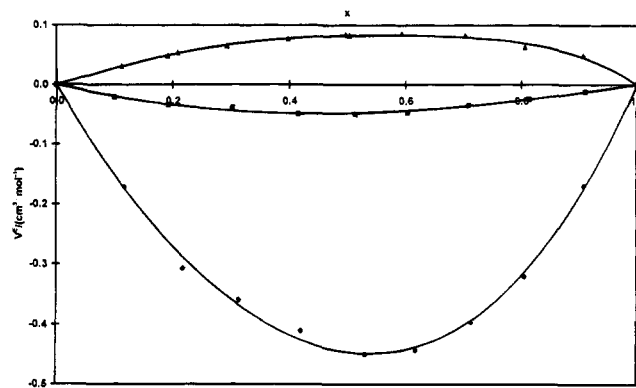


Figure 2. Excess volume for x 1,1,1-trichloroethane + $(1-x)$ (■) butanone, (●) cyclohexanone, and (Δ) acetone.

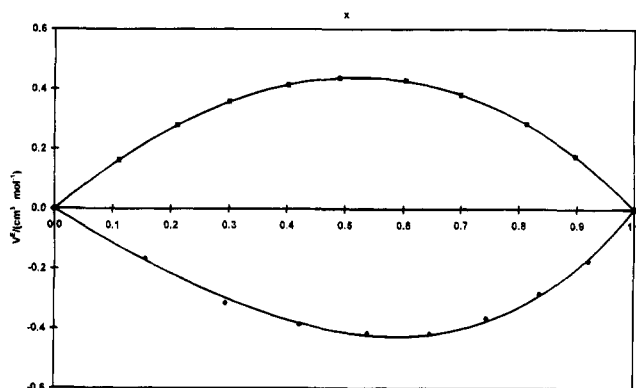


Figure 3. Excess volume for x 1,1,1-trichloroethane + $(1-x)$ (■) α,α,α -trifluorotoluene and (●) α,α,α -trichlorotoluene.

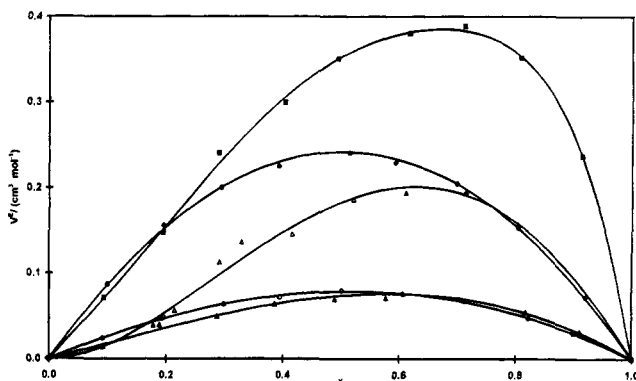


Figure 4. Excess volume for x 1,1,1-trichloroethane + $(1-x)$ (■) 1-methoxy-2-propanol, (○) ethyl acetate, (●) methyl acetate, (▲) nitromethane, and (Δ) 1-hexanol.

ment of the molecules, may explain the negative excess (Figure 3). Positive excess volume values are given by all the other systems examined. Maxima for the acetates fall around a 1:1 ratio between 1,1,1-trichloroethane and acetates, whereas for alcohols maxima are shifted toward systems rich in 1,1,1-trichloroethane (Figure 4).

A comparison between experimental and evaluated viscosity data is shown in Figures 5 and 6. Butanone and dipropyl ether curves show a slight minimum; the nitromethane curve shows a larger minimum. All the other curves are monotonous. The UNIMOD model proved to be a suitable correlating instrument for the systems to which it was applied.

As regards the viscosity deviation function, attention is drawn to the behavior of 1-hexanol and acetone systems, characterized by a sign inversion and to the behavior of nitromethane, which shows two minima.

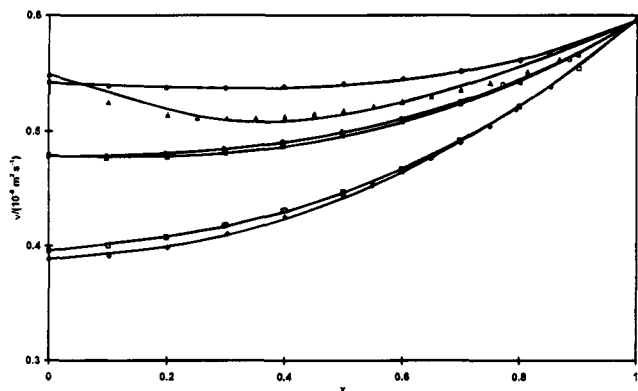


Figure 5. Kinematic viscosity for x 1,1,1-trichloroethane + $(1-x)$ (■) butanone, (○) ethyl acetate, (◆) acetone, (□) methyl acetate, (Δ) nitromethane, and (●) dipropyl ether.

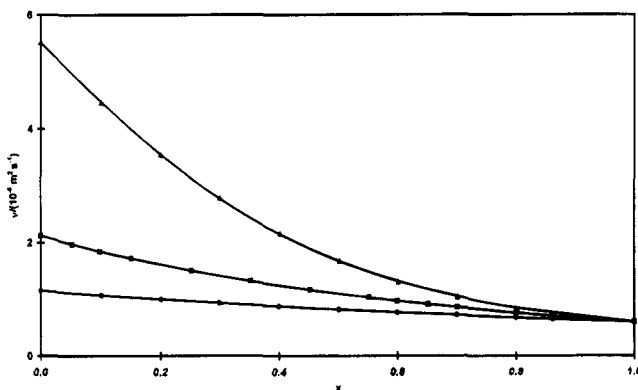


Figure 6. Kinematic viscosity for x 1,1,1-trichloroethane + $(1-x)$ (■) cyclohexanone, (Δ) 1-hexanol, and (●) 1,4-dioxane.

Conclusions

Correlation models were found to be generally valid; when their validity is ascertained, they can be employed with confidence as interpolation instruments.

A contribution was given to the availability of data, suitable for the determination of group contribution parameters of viscosity models for $-CCl_3$ and CCl_3CH_3 groups in binary mixtures of 1,1,1-trichloroethane with solvents of different chemical nature.

The general validity of the UNIMOD approach suggests its application also be extended to the four systems for which UNIMOD pure component parameters are not yet available. Evaluation of pure component parameters involves the determination of a series of viscosity and density data in an extended range of temperature. An investigation with this purpose is being started.

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