Glossary

a	attraction constant for equation of state
K	distribution coefficient
n	number of moles
P	pressure
r	moles of phenolic solution in sample/total moles of phenolic solution
R	gas constant
T	temperature
v	volume
x	mole fraction in liquid phase
y	mole fraction in supercritical phase
z	compressibility

Greek Letters

$\hat{\phi}$	fugacity coefficient

Subscripts

i	component i
j	component j
ij	cross term for components i and j

Superscripts

L	liquid phase
SC	supercritical phase

Registry No. PhOH, 108-95-2; CO_2, 124-38-9; benzene, 71-43-2.

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Excess Volumes and Viscosities of Binary Systems Containing 4-Methyl-2-pentanone

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Excess volumes and kinematic viscosities have been measured by means of a vibrating tube densimeter and an Ubbelohde viscometer for 12 binary systems containing 4-methyl-2-pentanone (MIBK). Excess volumes have been correlated by means of a polynomial expression, viscosities by means of the Mc Allister equation. The first component in all the binary systems studied is 4-methyl-2-pentanone (MIBK); the second component is 3-pentanone, 2-hexanone, cyclohexanone, *n*-heptane, toluene, *p*-xylene, ethylcyclohexane, 1,1,1-trichloroethane (chlorotene), *n*-butyl acetate, 4-hydroxy-4-methyl-2-pentanone (diacetone alcohol, DAA), 1-methoxy-2-propanol (PGM), or 1-acetoxy-2-ethoxyethane (ethylcellosolve). All the systems have been measured at atmospheric pressure and 298.15 K.

Introduction

Mixing volumes and viscosity effects are important from both the theoretical and practical points of view: indeed, strong deviations from linearity are often encountered in liquid mixtures, even if they are of similar nature.

Different approaches have been suggested for the prediction of mixtures viscosity. Among these, the group contribution methods seem to be particularly promising. The development of a group contribution method requires the availability of an adequate data base. The data base should consist of a sufficient number of systems, all containing the chemical functional groups taken into account in the operating area of the method. The present work adds a contribution to the development of the general data base and, in this respect, makes up a continuation of the project started in ref 1.

In addition to the scientific interest, the characterization of mixtures containing 4-methyl-2-pentanone (MIBK) is useful for a number of practical applications in the fields of paints, varnishes, and printing inks: in particular, the systems MIBK/PGM (PGM = 1-methoxy-2-propanol), MIBK/DDA (DDA = diacetone alcohol), MIBK/toluene, and MIBK/*p*-xylene for air-drying epoxy polyamine and polyamide coatings and the systems MIBK/ethylcellosolve and MIBK/cyclohexanone for air-drying polyurethane coatings.

Experimental Section

All the chemicals used in the present study were supplied by Fluka and used as received. The stated purity of all the chemicals exceeded 99.0 mol %. Nitrogen and bidistilled water were used for the densimeter calibration and cyclohexane for the viscometer calibration. The purity of the cyclohexane and water was higher than 99.9 mole % and that of nitrogen higher than 99.9999 mole %. Densities and viscosities of the pure components at 298.15 K and atmospheric pressure were measured before preparing the mixtures and compared with literature values (see Table I).

All the solutions were prepared with use of a Mettler balance (precision of 1×10^{-5} g) and airtight stoppered bottles following a procedure described elsewhere (*1*). The possible error in the mole fraction is estimated to be lower than 3×10^{-5}.

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Table I. Comparison between Measured and Literature Data for Pure Components at 298.15 K

component	d, g/cm^3 this work	d, g/cm^3 lit.	η, m Pa s this work	η, m Pa s lit.	ν, mm^2/s this work
3-methyl-2-butanone	0.79609	0.79610	0.541	0.542	0.680
3-pentanone	0.80932	0.80945	0.442	0.442	0.546
2-hexanone	0.80714		0.585		0.725
n-heptane	0.67949	0.67951	0.398	0.397	0.571
toluene	0.86217	0.86231	0.554	0.552	0.642
p-xylene	0.85668	0.86231	0.601	0.605	0.702
ethylcyclohexane	0.78359	0.78390	0.783	0.787	0.999
cyclohexanone	0.94249	0.94207	1.994		2.116
chlorotene	1.32990	1.32928	0.789	0.795	0.594
n-butyl acetate	0.87599	0.87636	0.677		0.772
DAA	0.93371	0.9342	2.884		3.088
PGM	0.91606		1.659		1.811
ethylcellosolve	0.96740		1.175		1.215

Table II. Experimental Results for the MIBK (1)/3-Pentanone (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.8789	0.662	0.79753	0.528	−0.00913
0.8066	0.652	0.79841	0.521	−0.01395
0.7509	0.644	0.79911	0.515	−0.01774
0.5932	0.623	0.80113	0.499	−0.02701
0.4466	0.604	0.80308	0.485	−0.03188
0.3004	0.585	0.80511	0.471	−0.03462
0.1609	0.566	0.80711	0.457	−0.03157
0.0749	0.557	0.80832	0.450	−0.02109

Table III. Experimental Results for the MIBK (1)/2-Hexanone (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.8841	0.687	0.79753	0.548	−0.02769
0.7765	0.693	0.79876	0.553	−0.03559
0.6632	0.697	0.80001	0.558	−0.03699
0.5503	0.701	0.80123	0.562	−0.03332
0.4413	0.706	0.80240	0.566	−0.02800
0.3357	0.711	0.80354	0.571	−0.02305
0.2196	0.715	0.80479	0.575	−0.01621
0.1164	0.721	0.80588	0.581	−0.00724

Table IV. Experimental Results for the MIBK (1)/*n*-Heptane (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.8854	0.656	0.77975	0.511	0.16062
0.7444	0.628	0.76060	0.478	0.35066
0.6443	0.612	0.74782	0.458	0.43917
0.5341	0.597	0.73450	0.438	0.49873
0.4417	0.586	0.72361	0.424	0.54621
0.3088	0.574	0.70905	0.407	0.51927
0.2052	0.568	0.69840	0.397	0.43757
0.1142	0.566	0.68961	0.390	0.30129

A digital densimeter (Anton Paar Model DMA 602H–DMA 60) was employed for the determination of the densities of the pure components and the binary mixtures. Calibration and measurement procedures are described in ref 1; the precision of the measured densities is estimated to be higher than 1×10^{-5} g/cm^3.

Kinematic viscosities of the pure liquids and their mixtures were measured with a Model AVS 300 Schott apparatus, consisting of a basic control unit, a measuring stand, a constant-temperature bath, and a calibrated Ubbelohde suspended level viscometer (*1*). The precision of the kinematic viscosity data was estimated to be higher than 5×10^{-4} mm^2/s.

Results and Correlation

Table I reports the comparison between the pure-component densities and viscosities measured in this work and the literature

Table V. Experimental Results for the MIBK (1)/Toluene (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.8774	0.673	0.80382	0.541	−0.11124
0.7653	0.664	0.81109	0.539	−0.20077
0.6441	0.655	0.81893	0.544	−0.25143
0.5357	0.650	0.82603	0.537	−0.27185
0.4196	0.645	0.83372	0.538	−0.26627
0.3183	0.641	0.84051	0.539	−0.23927
0.2070	0.638	0.84800	0.541	−0.17813
0.1094	0.638	0.85464	0.545	−0.10425

Table VI. Experimental Results for the MIBK (1)/*p*-Xylene (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.9061	0.680	0.80243	0.546	−0.11366
0.8014	0.681	0.80922	0.551	−0.19217
0.6366	0.682	0.81963	0.559	−0.26483
0.5673	0.683	0.82395	0.563	−0.28307
0.4463	0.685	0.83122	0.569	−0.27191
0.3369	0.688	0.83782	0.576	−0.26087
0.2260	0.691	0.84419	0.583	−0.20136
0.1409	0.695	0.84895	0.590	−0.13509

Table VII. Experimental Results for the MIBK (1)/Ethylcyclohexane (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.9079	0.689	0.79363	0.547	0.18674
0.8091	0.703	0.79141	0.556	0.33134
0.7041	0.719	0.78938	0.567	0.44181
0.5856	0.741	0.78742	0.583	0.52279
0.4730	0.764	0.78591	0.600	0.54785
0.3669	0.791	0.78475	0.621	0.53187
0.2549	0.823	0.78387	0.645	0.46161
0.1314	0.888	0.78332	0.695	0.31118

Table VIII. Experimental Results for the MIBK (1)/Cyclohexanone (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.8880	0.739	0.81038	0.599	−0.06944
0.7694	0.814	0.82622	0.672	−0.15009
0.6373	0.916	0.84434	0.773	−0.19200
0.5340	1.017	0.85894	0.872	−0.19586
0.3931	1.179	0.87949	1.037	−0.16277
0.3146	1.305	0.89139	1.163	−0.14003
0.2067	1.510	0.90838	1.371	−0.11228
0.1218	1.713	0.92220	1.580	−0.08333

Table IX. Experimental Results for the MIBK (1)/1,1,1-Trichloroethane (Chlorotene) (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.8536	0.669	0.83443	0.558	3.78549
0.7245	0.656	0.87404	0.573	6.28426
0.5907	0.644	0.92302	0.594	7.87801
0.4692	0.629	0.97618	0.614	8.40272
0.3765	0.619	1.02363	0.634	8.18374
0.2653	0.609	1.09243	0.665	6.96565
0.1769	0.600	1.15734	0.694	5.36353
0.0803	0.595	1.24297	0.739	2.82008

Table X. Experimental Results for the MIBK (1)/*n*-Butyl Acetate (2) System at 298.15 K

x_1	ν, mm^2/s	d, g/cm^3	η, m Pa s	V^E, cm^3/mol
0.8902	0.691	0.80526	0.556	0.00297
0.7901	0.701	0.81353	0.570	0.00546
0.6863	0.709	0.82201	0.583	0.00860
0.5881	0.717	0.82995	0.595	0.01136
0.4715	0.729	0.83927	0.612	0.01412
0.3594	0.740	0.84814	0.628	0.01416
0.2471	0.750	0.85694	0.643	0.00675
0.1280	0.762	0.86618	0.660	0.00675

Table XI. Experimental Results for the MIBK (1)/4-Hydroxy-4-methyl-2-pentanone (DAA) (2) System at 298.15 K

x_1	ν, mm²/s	d, g/cm³	η, m Pa s	V^E, cm³/mol
0.9001	0.751	0.809 18	0.608	0.080 44
0.8046	0.836	0.821 98	0.687	0.115 76
0.6974	0.952	0.836 35	0.796	0.157 99
0.5836	1.107	0.851 82	0.943	0.176 48
0.4821	1.280	0.865 73	1.108	0.179 95
0.3676	1.532	0.881 57	1.351	0.168 30
0.2473	1.900	0.898 38	1.707	0.140 02
0.1301	2.357	0.915 16	2.157	0.063 99

Table XII. Experimental Results for the MIBK (1)/1-Methoxy-2-propanol (PGM) (2) Systems at 298.15 K

x_1	ν, mm²/s	d, g/cm³	η, m Pa s	V^E, cm³/mol
0.8745	0.721	0.807 53	0.582	0.100 21
0.7612	0.770	0.818 75	0.630	0.140 90
0.6413	0.837	0.831 41	0.696	0.164 58
0.5295	0.917	0.844 08	0.774	0.160 21
0.4185	1.020	0.857 41	0.875	0.147 51
0.3075	1.154	0.871 61	1.006	0.121 77
0.2037	1.318	0.885 75	1.167	0.086 57
0.1057	1.526	0.899 92	1.373	0.043 38

Table XIII. Experimental Results for the MIBK (1)/1-Acetoxy-2-ethoxyethane (Ethylcellosolve) (2) Systems at 298.15 K

x_1	ν, mm²/s	d, g/cm³	η, m Pa s	V^E, cm³/mol
0.8892	0.720	0.816 39	0.588	0.017 96
0.8240	0.745	0.828 18	0.617	0.024 76
0.7252	0.785	0.845 77	0.664	0.039 34
0.6235	0.831	0.863 57	0.718	0.055 01
0.5110	0.886	0.882 98	0.782	0.060 12
0.3972	0.947	0.902 31	0.854	0.057 14
0.2727	1.022	0.923 14	0.943	0.042 24
0.1481	1.105	0.943 58	1.043	0.027 78

data taken from ref 2 at 298.15 K and atmospheric pressure: the agreement is satisfactory.

Tables II–XIII report the results of the measurements in terms of mole fractions; the tables include kinematic viscosity data (ν) and density data (d) (quantities directly measured), together with the dynamic viscosities (η) and the excess volumes (V^E). The excess volumes V^E were calculated by means of the following equation (*3*):

$$V^E = \frac{x_1M_1 + x_2M_2}{d} - \frac{x_1M_1}{d_1} - \frac{x_2M_2}{d_2} \tag{1}$$

where x_i are mole fractions and M_i and d_i are molecular weights and densities of the pure components.

The experimental data have been fitted and parameters of empirical models have been obtained for estimating densities and viscosities in the full concentration range. The parameter estimation has been performed by minimizing an objective function, consisting of the sum of the square of the differences between the experimental and calculated values. The Levenberg–Marquardt algorithm implemented by Fletcher (*4*) was used.

The following equation was used (*5*) to described the composition dependence of the excess volumes:

$$V^{E\,\mathrm{calc}} = x_1x_2\sum_{j=0}^{3}a_j(x_1 - x_2)^j \tag{2}$$

For the composition dependence of the kinematic viscosity, the Mc Allister equation (*6*) was employed:

$$\ln \nu = x_1^3 \ln \nu_1 + 3x_1^2x_2 \ln \nu_{12} + 3x_1x_2^2 \ln \nu_{21} + x_2^3 \ln \nu_2 + R^\circ$$

$$R^\circ = x_2^3 \ln \left(\frac{M_2}{M_1}\right) + 3x_1x_2^2 \ln \left(\frac{1 + 2M_2/M_1}{3}\right) + 3x_1^2x_2 \ln \left(\frac{2 + M_2/M_1}{3}\right) - \ln \left(\frac{x_1 + x_2M_2}{M_1}\right) \tag{3}$$

The experimental tests indicated that no appreciable differences can be detected within each data set with regard to the standard deviations for the single data points. Accordingly, the same weight has been assigned to all the data points in the minimization procedure.

The coefficients a_i (eq 2) and ν_{ij} (eq 3) obtained are listed in Table XIV along with the standard deviations σ_{V^E} and σ_ν for each system. The standard deviations were calculated by means of the following equations (*7*):

Table XIV. Values of the Coefficients of the Correlation Equation for Excess Volumes, Kinematic Viscosities, and Standard Deviations

		correlation equation coefficients				
system	function	a_0	a_1	a_2	a_3	σ
MIBK/3-pentanone	V^E	−0.1185	0.0653	−0.0960	0.0955	0.0006
	ν	0.6324	0.5921			0.0005
MIBK/2-hexanone	V^E	−0.1253	−0.0794	−0.0776	−0.0837	0.0007
	ν	0.6998	0.7096			0.0006
MIBK/*n*-heptane	V^E	2.0897	−0.6769	0.3395	−0.3716	0.0123
	ν	0.6114	0.5527			0.0009
MIBK/toluene	V^E	-1.1061	−0.0309	0.0779	0.0884	0.0039
	ν	0.6578	0.6318			0.0006
MIBK/*p*-xylene	V^E	-1.1344	0.0376	−0.1213	−0.2782	0.0059
	ν	0.6816	0.6826			0.0002
MIBK/ethylcyclohexane	V^E	2.1709	-0.2939	0.5241	−0.1083	0.0039
	ν	0.7400	0.7228			0.0054
MIBK/cyclohexanone	V^E	−0.7551	−0.3697	0.0097	0.7188	0.0017
	ν	0.8773	1.1479			0.0032
MIBK/chlorotene	V^E	33.4797	-4.6270	0.5937	−0.6138	0.0271
	ν	0.6672	0.6020			0.0010
MIBK/*n*-butyl acetate	V^E	0.0537	−0.0354	−0.0152	0.0278	0.0001
	ν	0.7128	0.7428			0.0009
MIBK/DAA	V^E	0.7348	−0.1149	−0.0463	0.5328	0.0071
	ν	0.9367	1.5075			0.0041
MIBK/PGM	V^E	0.6377	0.1754	0.1000	0.2033	0.0023
	ν	0.7844	1.0148			0.0019
MIBK/ethylcellosolve	V^E	0.2336	−0.0250	−0.0752	0.0023	0.0028
	ν	0.8077	0.9802			0.0002

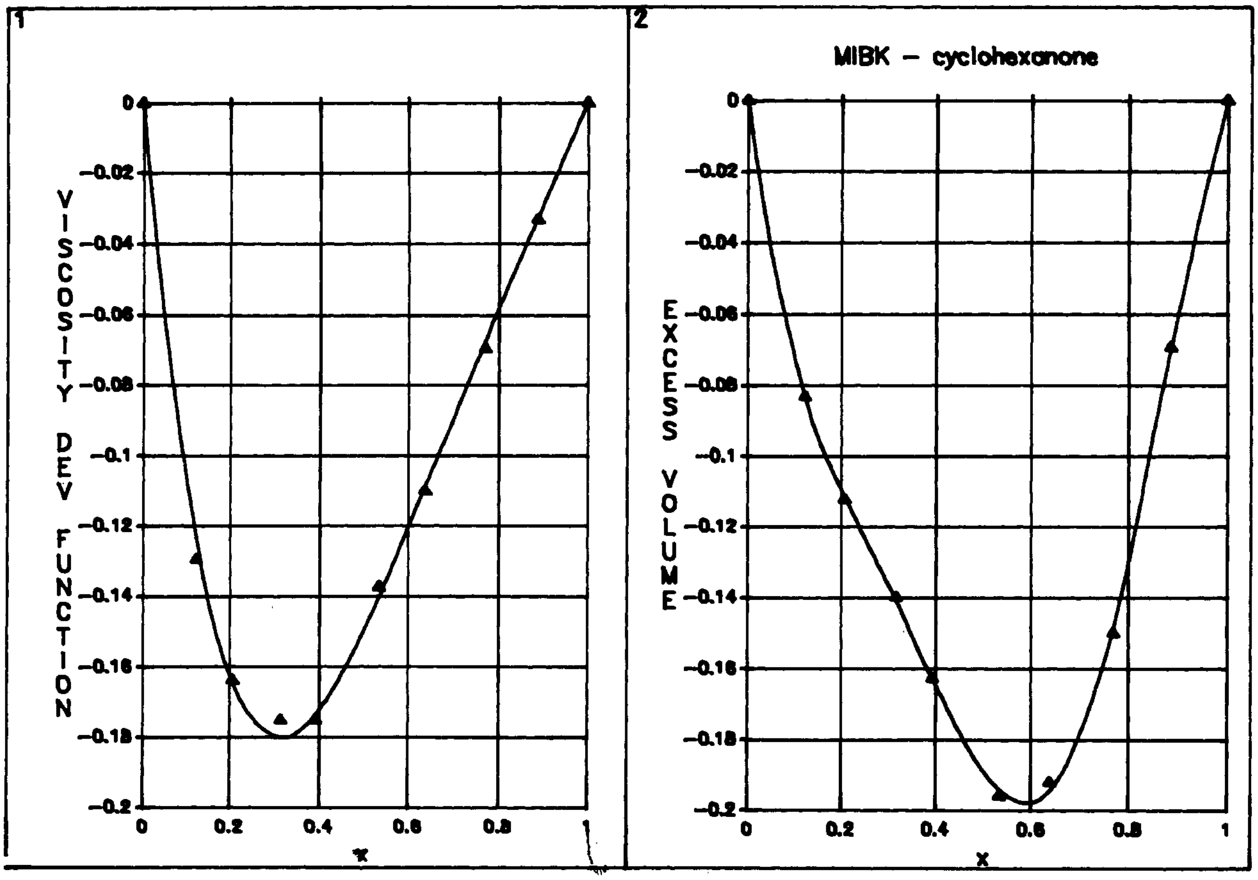

Figure 1. Viscosity deviation function (1) and excess volume (2) for the MIBK/cyclohexanone system at 298.15 K: comparison between calculated (line) and experimental data.

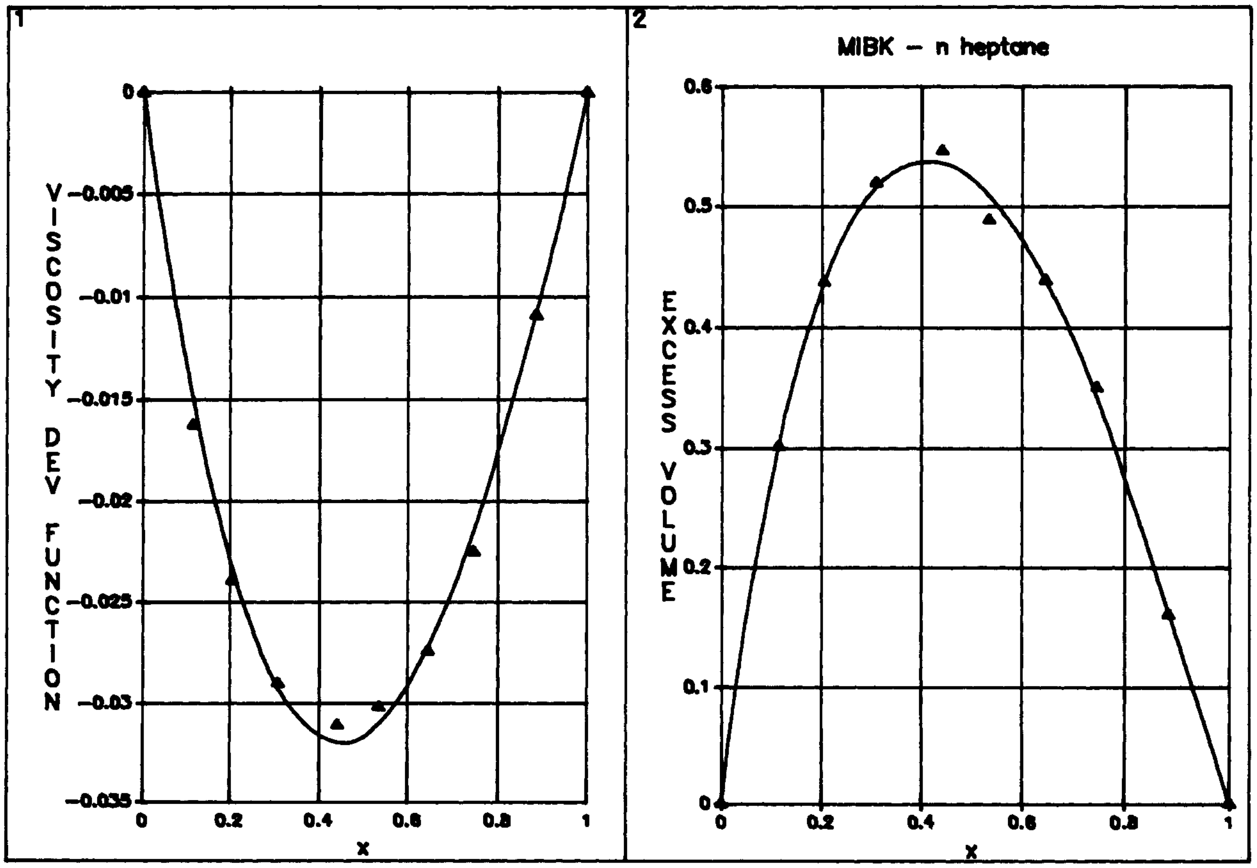

Figure 2. Viscosity deviation function (1) and excess volume (2) for the MIBK/*n*-heptane system at 298.15 K: comparison between calculated (line) and experimental data.

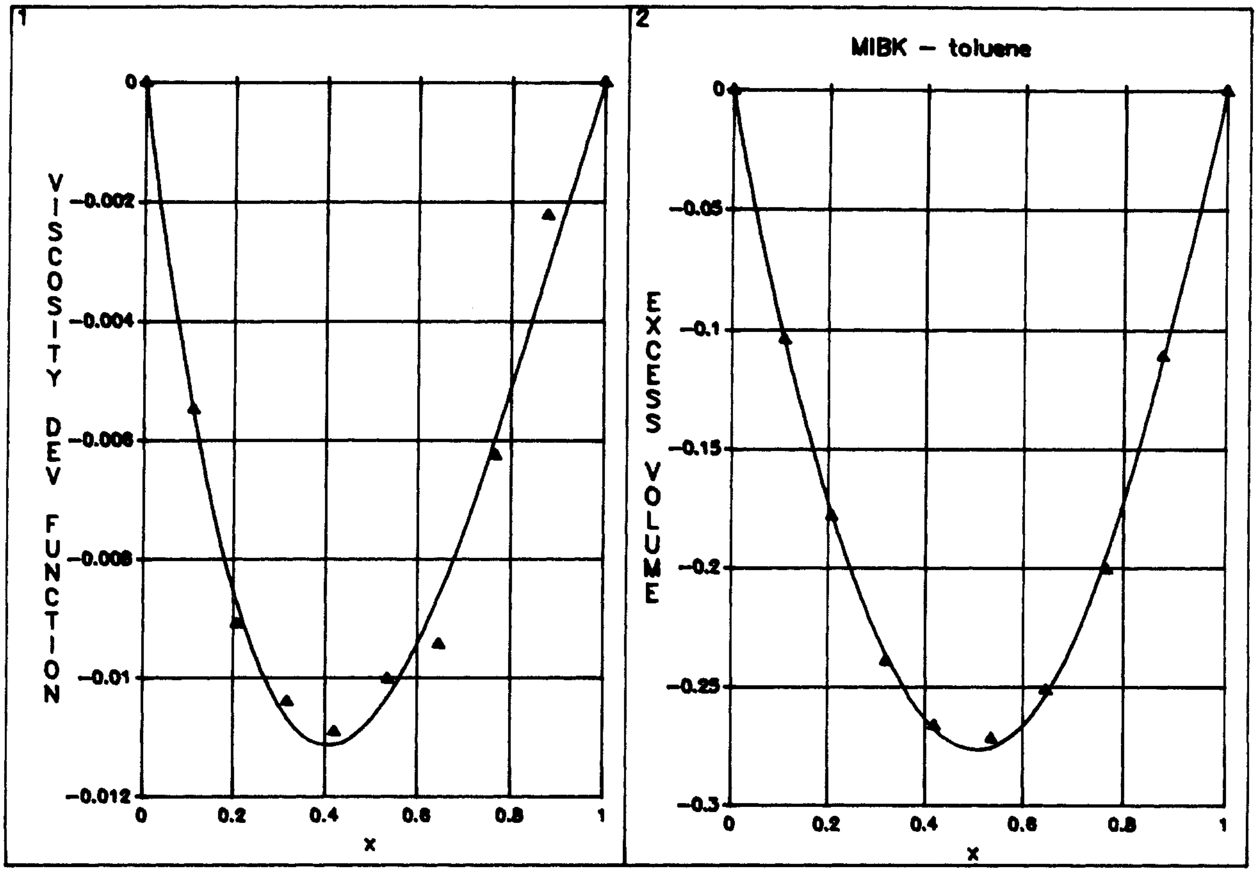

Figure 3. Viscosity deviation function (1) and excess volume (2) for the MIBK/toluene system at 298.15 K: comparison between calculated (line) and experimental data.

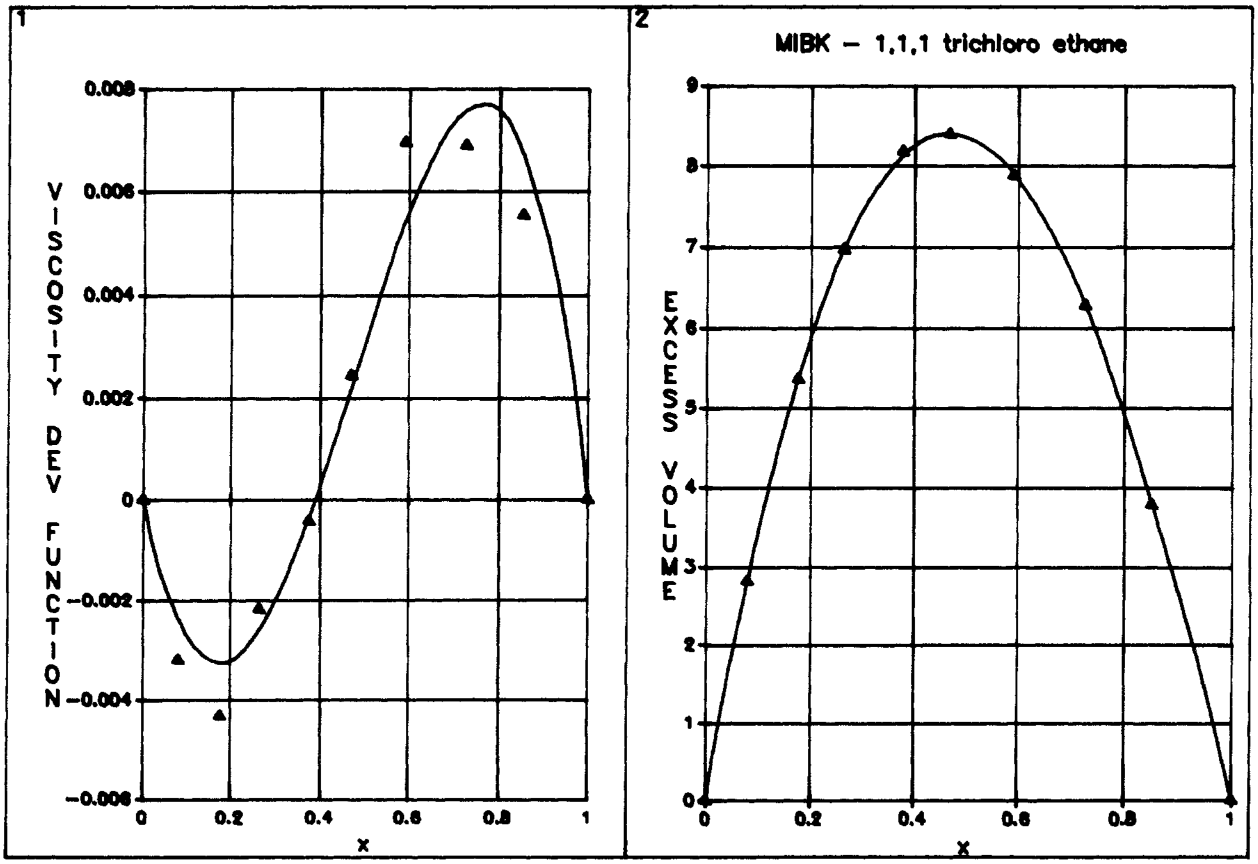

Figure 4. Viscosity deviation function (1) and excess volume (2) for the MIBK/1,1,1-trichloroethane (chlorotene) system at 298.15 K: comparison between calculated (line) and experimental data.

$$\sigma_{V^E} = \left[\sum \frac{(V^{E\,\mathrm{calc}} - V^{E\,\mathrm{expt}})^2}{N - n}\right]^{1/2} \qquad (4)$$

$$\sigma_\nu = \left[\sum \frac{(\nu^{\mathrm{calc}} - \nu^{\mathrm{expt}})^2}{N - n}\right]^{1/2} \qquad (5)$$

where N is the number of data points for each system and n is the number of parameters.

The Mc Allister correlation equation gave excellent results for all the systems investigated ($\sigma_\nu < 0.005$). The results obtained from the correlation equation (eq 2) were quite satisfactory for all the systems investigated ($\sigma_{V^E} < 0.007$) with the exception of the MIBK/n-heptane and MIBK/1,1,1-trichloroethane systems for which a small data scattering was found.

The viscosity deviation function is defined according to ref 8 as

$$\Delta f(\eta V) = \ln (\eta V) - \sum x_i \ln (\eta_i \nu_i) \qquad (6)$$

Discussion

Several different behaviors can be detected among the binary systems investigated as far as $\Delta f(\eta V)$ and V^E are concerned. Accordingly, the systems can be grouped as follows:

(a) The systems MIBK/3-pentanone, 2-hexanone, butyl acetate, and 1-acetoxy-2-ethoxyethane are one group. The deviations of the mixture viscosity and the volume from linearity are not pronounced ($\Delta f(\eta V) \leq |0.01|$ and $V^E \leq |0.06|$).

(b) The MIBK/cyclohexanone system shows a more pronounced deviation from the ideality than the other ketone-containing systems tested with regard to both viscosity and volume (Figure 1). The same considerations can be extended to the hydroxy-containing compounds (4-hydroxy-4-methyl-2-pentanone and 1-methoxy-2-propanol), but in the latter instance, the V^E is positive.

(c) Another group is the aromatics. The excess volume is of the same order of magnitude as for the compounds of b but negative. The viscosity deviation function is almost as negligible as it is in a (see Figure 3).

(d) The alkanes and cycloalkanes are grouped together. For n-heptane and cyclohexane, V^E is large and positive, $\Delta f(\eta V)$ is intermediate between the correponding values of the components of groups b and c.

(e) The MIBK/1,1,1-trichloro-ethane system exhibits a very peculiar behavior (Figure 4). It displays the maximum volume deviation from linearity and, in addition, reveals a sign inversion in the viscosity deviation function.

Among the various group contribution methods published so far, the one proposed by Wu (*8*) is particularly interesting. Since this work aims at giving a contribution to the data base of the above mentioned Wu's method, our experimental data were treated according to the Wu approach in all the cases for which model parameters were available. Details of the applied procedure and results are given elsewhere (*9*). Suffice it to mention here that satisfactory results were obtained for the systems MIBK + n-heptane, 2-hexanone, and 3-pentanone (mean percent deviations around 0.4), but the method failed with the systems containing the cyclic CH_2 group (mean percent deviations from 3 to 10).

Accordingly, work is in progress to improve the group contribution method as far as systems containing cyclic CH_2 groups are concerned.

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Glossary

a	coefficients, eq 2
M	molecular weight
N	number of experimental data points
n	numer of parameters
V^E	excess volume, cm^3/mol
x	mole fraction
ν	kinematic viscosity, mm^2/s = cSt
ν_{ij}	coefficients in the Mc Allister equation
$\Delta f(\eta V)$	viscosity deviation function
η	dynamic viscosity, mPa s = cP
d	density, g/cm^3
σ_ν	kinematic viscosity standard deviation
σ_{V^E}	excess volumes standard deviation

Registry No. DAA, 123-42-2; PGM, 107-98-2; 4-methyl-2-pentanone, 108-10-1; 3-methyl-2-butanone, 563-80-4; 3-pentanone, 96-22-0; 2-hexanone, 591-78-6; n-heptane, 142-82-5; toluene, 108-88-3; p-xylene, 106-42-3; ethylcyclohexane, 1678-91-7; cyclohexanone, 108-94-1; chlorotene, 71-55-6; n-butyl acetate, 123-86-4; ethylcellosolve, 110-80-5.

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