Densities and Viscosities of Binary Mixtures of Some Cyclic Ethers + Chlorocyclohexane at 298.15 and 313.15 K

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Density and viscosity measurements are reported for the binary mixtures of some cyclic ethers (tetrahydrofuran, tetrahydropyran, 2-methyl-tetrahydrofuran and 2,5-dimethyl-tetrahydrofuran) with chlorocyclohexane at 298.15 K and 313.15 K. The viscosity data were correlated by the equations of Grunberg−Nissan, McAllister, and Heric. The experimental data were used to calculate excess volumes and viscosity deviations, and the results were fitted to a Redlich−Kister type equation.

Introduction

In this paper we present density and viscosity measurements for the binary mixtures of some cyclic ethers (tetrahydrofuran, tetrahydropyran, 2-methyl-tetrahydrofuran, and 2,5-dimethyltetrahydrofuran) with chlorocyclohexane at 298.15 K and 313.15 K. From these data, excess volumes and viscosity deviations were calculated. As far as we know, there are neither density nor viscosity measurements on the systems studied here.

This work follows our study on thermophysic properties of mixtures containing a cyclic ether and a halohydrocarbon. In previous papers from our laboratory, excess volumes and viscosities for the same cyclic ethers with bromocyclohexane (Rodríguez et al., 1996) and bromobenzene (Rodríguez et al., 1997) were reported.

Experimental Section

Materials. The liquids used were tetrahydrofuran (better than 99.5% purity), tetrahydropyran, 2-methyltetrahydrofuran, and 2,5-dimethyltetrahydrofuran (better than 99.0% purity) obtained from Aldrich and chlorocyclohexane (better than 99.5% purity) provided by Fluka. The purities of these compounds were checked by a chromatographic method, confirming the absence of other significant organic components, so no further purification was attempted.

Methods. Densities were measured with an Anton-Paar DMA-58 vibrating tube densimeter automatically thermostated at ±0.01 K. The accuracy of density measurements, after proper calibration, was $\pm 1 \times 10^{-5}$ g·cm^{-3}. The calibration was carried out with deionized doubly distilled water and dry air.

The kinematic viscosities were determined using an Ubbelhode viscosimeter and a Schott-Geräte automatic measuring unit model AVS-440. The temperature was kept constant within ±0.01 K by means of an Schott-Geräte thermostat. The accuracy of the time flow measurements was ±0.01 s. At least four time flow measurements were performed for each composition and temperature, and the results were averaged. Kinetic energy corrections were applied to the experimental data. The estimed error in the kinematic viscosity was $\pm 1 \times 10^{-4}$ mm^2·s^{-1}.

From density, ρ, and kinematic viscosity, ν, the absolute viscosity, η, can be obtained by using the equation

$$\eta = \rho\nu \tag{1}$$

The estimed uncertainty in the absolute viscosity was $\pm 1 \times 10^{-4}$ mPa·s.

The mixtures were prepared by mass using a Mettler H20T balance. The possible error in the mole fractions is estimated to be less than ±0.0001.

The comparison of measured densities and viscosities of the chemicals with literature values at 298.15 K is shown in Table 1.

Table 1. Densities, ρ, and Viscosities, η, of the Pure Compounds at 298.15 K

	ρ/g·cm^{-3}		η/mPa·s	
compound	exptl	lit.	exptl	lit.
tetrahydrofuran	0.882 09	0.882 5[a]	0.4637	0.461[d]
tetrahydropyran	0.879 15	0.879 16[b]	0.8008	0.764[e]
2-methyltetrahydrofuran	0.849 90	0.848 82[c]	0.4835	0.4753[c]
2,5-dimethyltetrahydrofuran	0.825 27		0.4850	
chlorocyclohexane	0.993 26		1.5675	

[a] TRC (1985). [b] Inglese et al. (1983). [c] De Lorenzi et al. (1995). [d] Carvajal et al. (1965). [e] Nicholls et al. (1968).

Results and Discussion

The experimental values of density and viscosity for the binary mixtures are given in Tables 2 and 3, respectively.

The viscosity data were correlated by using the following equations

Grunberg-Nissan (1949):

$$\ln \eta = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1 x_2 \delta_{12} \tag{2}$$

where η is the viscosity of the mixture, x_1 and x_2 are the mole fractions of components 1 and 2, η_1 and η_2 are the

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Table 2. Densities and Excess Volumes of Binary Mixtures of a Cyclic Ether (1) + Chlorocyclohexane (2)

x_1	ρ/g·cm^{-3}	V^E/ cm^3·mol^{-1}	x_1	ρ/g·cm^{-3}	V^E/ cm^3·mol^{-1}
Tetrahydrofuran (1) + Chlorocyclohexane (2) at 298.15 K					
0.0463	0.989 66	0.0029	0.6063	0.936 17	0.0039
0.1018	0.985 22	0.0042	0.6938	0.925 65	0.0018
0.1883	0.977 97	0.0063	0.8073	0.910 84	−0.0008
0.3044	0.967 57	0.0071	0.9019	0.897 35	−0.0017
0.3902	0.959 33	0.0073	0.9518	0.889 76	−0.0012
0.5005	0.947 97	0.0064			
Tetrahydropyran (1) + Chlorocyclohexane (2) at 298.15 K					
0.0472	0.988 91	−0.0128	0.6100	0.929 26	−0.0152
0.1028	0.983 62	−0.0197	0.6903	0.919 55	−0.0068
0.2044	0.973 61	−0.0254	0.8026	0.905 42	0.0054
0.2958	0.964 24	−0.0266	0.9026	0.892 29	0.0139
0.4000	0.953 13	−0.0251	0.9522	0.885 63	0.0111
0.4827	0.943 98	−0.0230			
2-Methyltetrahydrofuran (1) + Chlorocyclohexane (2) at 298.15 K					
0.0525	0.987 04	−0.0262	0.6039	0.913 14	−0.0884
0.1029	0.980 89	−0.0416	0.6965	0.899 19	−0.0777
0.2026	0.968 35	−0.0621	0.8012	0.882 82	−0.0580
0.2924	0.956 66	−0.0742	0.9000	0.866 75	−0.0302
0.3971	0.942 55	−0.0825	0.9532	0.857 85	−0.0131
0.5010	0.928 05	−0.0886			
2,5-Dimethyltetrahydrofuran (1) + Chlorocyclohexane (2) at 298.15 K					
0.0513	0.985 07	−0.0681	0.6001	0.894 24	−0.3295
0.0997	0.977 29	−0.1254	0.7089	0.875 74	−0.2935
0.2035	0.960 35	−0.2145	0.8024	0.859 71	−0.2357
0.3020	0.944 14	−0.2782	0.9025	0.842 40	−0.1423
0.3973	0.928 26	−0.3102	0.9556	0.833 15	−0.0780
0.4880	0.913 17	−0.3387			
Tetrahydrofuran (1) + Chlorocyclohexane (2) at 313.15 K					
0.0462	0.975 04	0.0019	0.6086	0.920 56	−0.0463
0.1013	0.970 57	−0.0019	0.6955	0.909 86	−0.0440
0.2010	0.962 10	−0.0129	0.8070	0.894 87	−0.0285
0.3064	0.952 50	−0.0236	0.8937	0.882 24	−0.0187
0.3941	0.943 97	−0.0335	0.9523	0.873 16	−0.0128
0.5016	0.932 74	−0.0439			
Tetrahydropyran (1) + Chlorocyclohexane (2) at 313.15 K					
0.0468	0.974 28	−0.0038	0.6041	0.914 95	−0.0226
0.0983	0.969 33	−0.0094	0.7067	0.902 47	−0.0150
0.2039	0.958 88	−0.0203	0.8009	0.890 54	−0.0072
0.3015	0.948 82	−0.0265	0.9005	0.877 17	0.0029
0.3926	0.939 07	−0.0297	0.9551	0.870 01	0.0023
0.4892	0.928 31	−0.0278			
2-Methyltetrahydrofuran (1) + Chlorocyclohexane (2) at 313.15 K					
0.0530	0.972 31	−0.0220	0.5982	0.898 60	−0.0870
0.1027	0.966 16	−0.0360	0.7038	0.882 63	−0.0815
0.2058	0.953 06	−0.0602	0.8055	0.866 60	−0.0621
0.2935	0.941 51	−0.0718	0.9040	0.850 48	−0.0348
0.3965	0.927 50	−0.0810	0.9500	0.842 74	−0.0187
0.5031	0.912 50	−0.0892			
2,5-Dimethyltetrahydrofuran (1) + Chlorocyclohexane (2) at 313.15 K					
0.0498	0.970 71	−0.0699	0.5016	0.896 15	−0.3919
0.1010	0.962 37	−0.1263	0.5958	0.880 38	−0.4112
0.2017	0.945 91	−0.2264	0.6961	0.863 30	−0.3822
0.2979	0.930 12	−0.3086	0.8012	0.845 21	−0.3120
0.3934	0.914 30	−0.3657	0.8994	0.828 29	−0.2308

corresponding viscosities, and δ_{12} is a parameter that is function of the nature of the mixing species.

McAllister (1960):

$$\ln \nu = x_1^3 \ln \nu_1 + 3x_1^2 x_2 \ln \nu_{12} + 3x_1 x_2^2 \ln \nu_{21} + x_2^3 \ln \nu_2 - \ln[x_1 + x_2 M_2/M_1] + 3x_1^2 x_2 \ln[(2 + M_2/M_1)/3] + 3x_1 x_2^2 \ln[(1 + 2M_2/M_1)/3] + x_2^3 \ln(M_2/M_1) \quad (3)$$

Table 3. Viscosities and Viscosity Deviations of Binary Mixtures of a Cyclic Ether (1) + Chlorocyclohexane (2)

x_1	η/mPa·s	$\Delta\eta$/mPa·s	x_1	η/mPa·s	$\Delta\eta$/mPa·s
Tetrahydrofuran (1) + Chlorocyclohexane (2) at 298.15 K					
0.1036	1.3801	−0.0730	0.6003	0.7631	−0.1418
0.2057	1.2204	−0.1200	0.7026	0.6748	−0.1171
0.3029	1.0874	−0.1457	0.8045	0.5979	−0.0816
0.4018	0.9655	−0.1585	0.9032	0.5278	−0.0428
0.5079	0.8521	−0.1548			
Tetrahydropyran (1) + Chlorocyclohexane (2) at 298.15 K					
0.1031	1.4586	−0.0298	0.6036	1.0405	−0.0642
0.2000	1.3638	−0.0504	0.6992	0.9774	−0.0540
0.3037	1.2703	−0.0644	0.8038	0.9135	−0.0377
0.4038	1.1877	−0.0702	0.9034	0.8561	−0.0187
0.4996	1.1139	−0.0705			
2-Methyltetrahydrofuran (1) + Chlorocyclohexane (2) at 298.15 K					
0.0984	1.3668	−0.0940	0.5995	0.7420	−0.1756
0.1994	1.1953	−0.1560	0.7010	0.6638	−0.1439
0.2986	1.0552	−0.1886	0.8020	0.5963	−0.1018
0.4009	0.9325	−0.2004	0.8995	0.5378	−0.0546
0.4991	0.8311	−0.1953			
2,5-Dimethyltetrahydrofuran (1) + Chlorocyclohexane (2) at 298.15 K					
0.0973	1.3598	−0.1024	0.6031	0.7279	−0.1867
0.2014	1.1789	−0.1705	0.7002	0.6563	−0.1532
0.3026	1.0334	−0.2066	0.7977	0.5937	−0.1103
0.3986	0.9191	−0.2169	0.8986	0.5366	−0.0582
0.4986	0.8180	−0.2098			
Tetrahydrofuran (1) + Chlorocyclohexane (2) at 313.15 K					
0.1036	1.0853	−0.0440	0.6003	0.6344	−0.0889
0.2057	0.9721	−0.0738	0.7026	0.5652	−0.0744
0.3029	0.8761	−0.0902	0.8045	0.5038	−0.0525
0.4018	0.7866	−0.0989	0.9032	0.4475	−0.0281
0.5079	0.7016	−0.0971			
Tetrahydropyran (1) + Chlorocyclohexane (2) at 313.15 K					
0.1031	1.1415	−0.0145	0.6036	0.8332	−0.0412
0.2000	1.0729	−0.0285	0.6992	0.7853	−0.0352
0.3037	1.0024	−0.0407	0.8038	0.7364	−0.0253
0.4038	0.9418	−0.0450	0.9034	0.6928	−0.0129
0.4996	0.8879	−0.0450			
2-Methyltetrahydrofuran (1) + Chlorocyclohexane (2) at 313.15 K					
0.0984	1.0753	−0.0598	0.5995	0.6167	−0.1162
0.1994	0.9535	−0.1005	0.7010	0.5561	−0.0954
0.2986	0.8519	−0.1224	0.8020	0.5018	−0.0686
0.4009	0.7611	−0.1312	0.8995	0.4546	−0.0375
0.4991	0.6848	−0.1287			
2,5-Dimethyltetrahydrofuran (1) + Chlorocyclohexane (2) at 313.15 K					
0.0973	1.0727	−0.0632	0.6031	0.6054	−0.1243
0.2014	0.9412	−0.1111	0.7002	0.5510	−0.1006
0.3026	0.8337	−0.1372	0.7977	0.4990	−0.0743
0.3986	0.7486	−0.1452	0.8986	0.4520	−0.0403
0.4986	0.6724	−0.1411			

where ν refers to the kinematic viscosity of the mixture, ν_1 and ν_2 are the kinematic viscosities of components 1 and 2, M_1 and M_2 are molecular mass of the components, and ν_{12} and ν_{21} are adjustable parameters characteristic of the system.

Heric (1966):

$$\ln \nu = x_1 \ln(\nu_1 M_1) + x_2 \ln(\nu_2 M_2) - \ln(x_1 M_1 + x_2 M_2) + x_1 x_2[\alpha_{12} + \alpha'_{12}(x_1 - x_2)] \quad (4)$$

in which α_{12} and α'_{12} are also adjustable parameters.

The estimated parameters of the viscosity equations and the corresponding standard deviations between experimental and calculated values are shown in Table 4. As one

Table 4. Coefficients and Standard Deviations, σ, for Viscosity Equations

	T = 298.15 K			T = 313.15 K		
equation	a	b	σ	a	b	σ
			Tetrahydrofuran + Chlorocyclohexane			
Grunberg–Nissan[a]	0.0389		0.0052	0.0824		0.0036
McAllister[b]	0.7863	1.0755	0.0016	0.6682	0.8905	0.0009
Heric[c]	0.1107	0.0843	0.0016	0.1507	0.0715	0.0009
			Tetrahydropyran + Chlorocyclohexane			
Grunberg–Nissan	−0.0206		0.0021	−0.0032		0.0017
McAllister	1.0904	1.2873	0.0012	0.8855	1.0428	0.0019
Heric	−0.0017	0.0270	0.0012	0.0149	0.0049	0.0019
			2-Methyltetrahydrofuran + Chlorocyclohexane			
Grunberg–Nissan	−0.1886		0.0046	−0.1294		0.0029
McAllister	0.7606	1.0245	0.0005	0.6476	0.8528	0.0003
Heric	−0.1785	0.0645	0.0005	−0.1198	0.0493	0.0003
			2,5-Dimethyltetrahydrofuran + Chlorocyclohexane			
Grunberg–Nissan	−0.2619		0.0051	−0.1937		0.0039
McAllister	0.7612	1.0091	0.0003	0.6507	0.8381	0.0021
Heric	−0.2733	0.0712	0.0003	−0.2049	0.0676	0.0021

[a] $a = \delta_{12}$. [b] $a = \nu_{12}$, $b = \nu_{21}$. [c] $a = \alpha_{12}$, $b = \alpha'_{12}$.

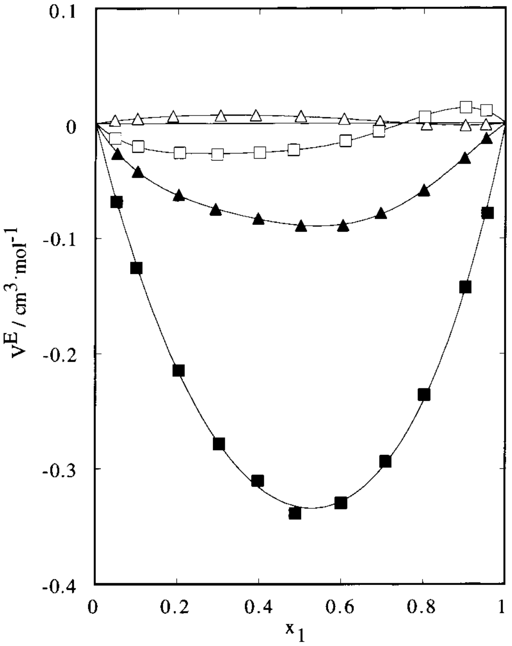

Figure 1. Excess volumes for cyclic ether (1) + chlorocyclohexane (2) at T = 298.15 K as a function of mole fraction x_1: tetrahydrofuran (△); tetrahydropyran (□); 2-methyltetrahydrofuran (▲); 2,5-dimethyltetrahydrofuran (■).

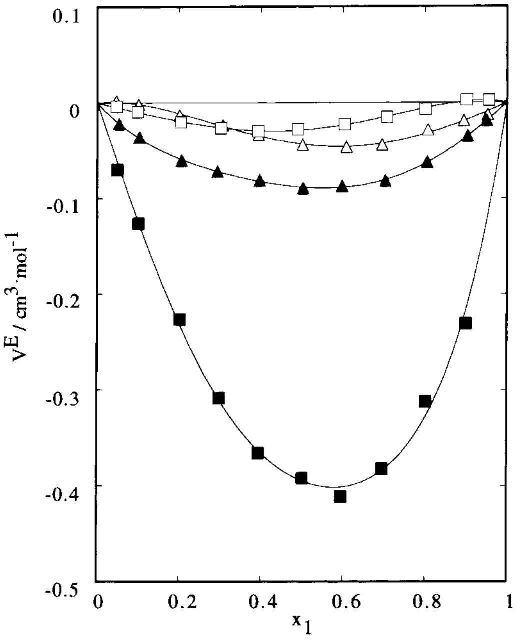

Figure 2. Excess volumes for cyclic ether (1) + chlorocyclohexane (2) at T = 313.15 K as a function of mole fraction x_1: tetrahydrofuran (△); tetrahydropyran (□); 2-methyltetrahydrofuran (▲); 2,5-dimethyltetrahydrofuran (■).

can see in this Table, the equations of McAllister and Heric correlated very well the viscosity data, and they give about the same results.

Excess volumes and viscosity deviations were calculated from our measurements according to the following equations

$$V^E = x_1M_1(1/\rho - 1/\rho_1) + x_2M_2(1/\rho - 1/\rho_2) \qquad (5)$$

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \qquad (6)$$

where ρ is the density of the mixture and ρ_1 and ρ_2 are the densities of components 1 and 2. The rest of the symbols have been considered above. These functions, i.e., excess volumes and viscosity deviations, are gathered in Tables 2 and 3 and graphically represented in Figures 1–4.

The values of these properties at each temperature were correlated with a Redlich–Kister polynomial equation

$$V^E \text{ (or } \Delta\eta) = x_1(1 - x_1)\sum_{i=0}^{i=N} A_i(2x_1 - 1)^i \qquad (7)$$

where A_i are adjustable parameters and x_1 is the mole fraction of cyclic ether. Table 5 lists the values of the parameters together with the standard deviations.

The results at 298.15 K show that excess volumes for mixtures containing tetrahydrofuran and tetrahydropyran are small and display sigmoidal curves. On the other hand, excess volumes for mixtures containing 2-methyltetrahydrofuran and 2,5-dimethyltetrahydrofuran are negative over the whole composition range. Equimolar V^E values follow the sequence: tetrahydrofuran > tetrahydropyran > 2-methyltetrahydrofuran < 2,5-dimethyltetrahydrofuran. When the temperature increases, the more noticeable is that excess volumes for mixtures containing tetrahydrofuran and tetrahydropyran decrease, showing negative

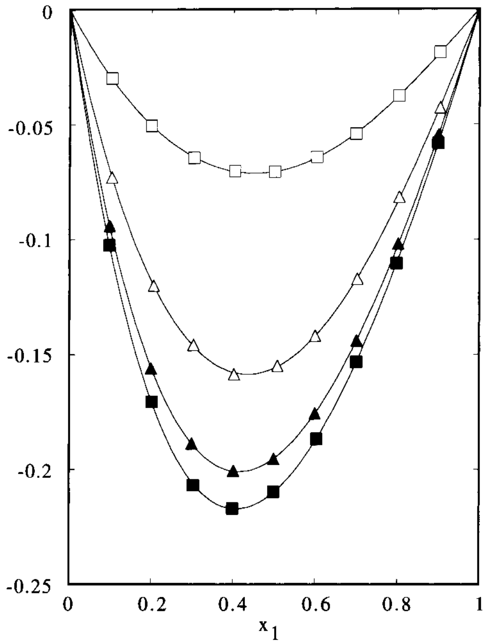

Figure 3. Viscosity deviations for cyclic ether (1) + chlorocyclohexane (2) at T = 298.15 K as a function of mole fraction x_1: tetrahydrofuran (△); tetrahydropyran (□); 2-methyltetrahydrofuran (▲); 2,5-dimethyltetrahydrofuran (■).

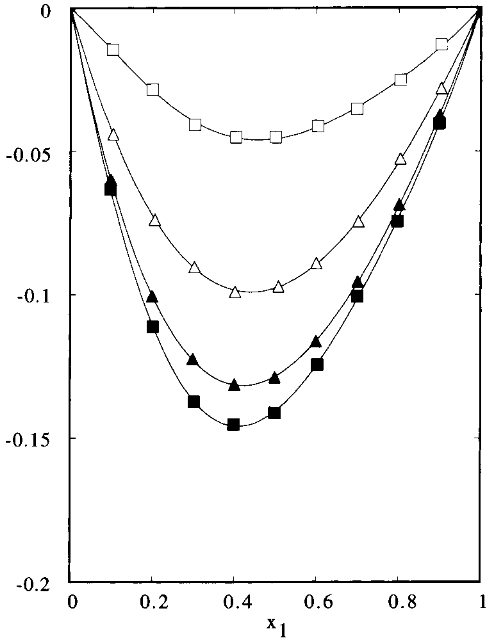

Figure 4. Viscosity deviations for cyclic ether (1) + chlorocyclohexane (2) at T = 313.15 K as a function of mole fraction x_1: tetrahydrofuran (△); tetrahydropyran (□); 2-methyltetrahydrofuran (▲); 2,5-dimethyltetrahydrofuran (■).

values over practically the whole composition range. For the other mixtures, the variation of V^E with temperature is small. The main contributions to excess volumes are a positive one due to the weakening of the dipole–dipole interactions between the pure components on the mixing process and a negative contribution due to a specific Cl–O interaction, in which the oxygen atom is the donor and the chlorine atom is the acceptor. The existence of this interaction is supported by the decrease of V^E values of these mixtures with respect the mixtures of tetrahydrofuran (Arm and Bánkay, 1969) and tetrahydropyran (Inglese et al., 1984) with cyclohexane and by the negative H^E values for the mixture of tetrahydropyran with chlorocyclohexane (Ruíz et al., 1988).

Table 5. Coefficients, A_i, and Standard Deviations, σ, for Equation 7

function	A_0	A_1	A_2	A_3	σ
Tetrahydrofuran + Chlorocyclohexane at 298.15 K					
V^E/cm^3·mol^{-1}	0.0242	−0.0301	−0.0156	−0.0193	0.0003
$\Delta\eta$/mPa·s	−0.6233	0.1624	−0.0202	0.0396	0.0006
Tetrahydropyran + Chlorocyclohexane at 298.15 K					
V^E/cm^3·mol^{-1}	−0.0897	0.0809	0.0833	0.2370	0.0007
$\Delta\eta$/mPa·s	−0.2820	0.0578	0.0157	0.0143	0.0003
2-Methyltetrahydrofuran + Chlorocyclohexane at 298.15 K					
V^E/cm^3·mol^{-1}	−0.3535	−0.0562	−0.0633	0.2128	0.0008
$\Delta\eta$/mPa·s	−0.7803	0.2607	−0.0800	0.0410	0.0003
2,5-Dimethyltetrahydrofuran + Chlorocyclohexane at 298.15 K					
V^E/cm^3·mol^{-1}	−1.3327	−0.1255	−0.2459	−0.0436	0.0045
$\Delta\eta$/mPa·s	−0.8389	0.3063	−0.0954	0.0319	0.0003
Tetrahydrofuran + Chlorocyclohexane at 313.15 K					
V^E/cm^3·mol^{-1}	−0.1728	−0.1074	0.0990	−0.0121	0.0022
$\Delta\eta$/mPa·s	−0.3904	0.0943	−0.0110	0.0038	0.0005
Tetrahydropyran + Chlorocyclohexane at 313.15 K					
V^E/cm^3·mol^{-1}	−0.1144	0.0609	0.1021	0.0274	0.0012
$\Delta\eta$/mPa·s	−0.1823	0.0380	0.0383	−0.0557	0.0007
2-Methyltetrahydrofuran + Chlorocyclohexane at 313.15 K					
V^E/cm^3·mol^{-1}	−0.3521	−0.0671	−0.0793	0.1075	0.0011
$\Delta\eta$/mPa·s	−0.5134	0.1599	−0.0469	0.0056	0.0003
2,5-Dimethyltetrahydrofuran + Chlorocyclohexane at 313.15 K					
V^E/cm^3·mol^{-1}	−1.5782	−0.3487	−0.4715	−0.4511	0.0110
$\Delta\eta$/mPa·s	−0.5615	0.2231	−0.0307	−0.0783	0.0005

Viscosity deviations are negative over the whole composition range for all the mixtures and decrease in absolute value as the temperature is increased. Equimolar $\Delta\eta$, in absolute value, increases in the sequence: tetrahydropyran < tetrahydrofuran < 2,5-dimethyltetrahydrofuran < 2-methyltetrahydrofuran.

The experimental viscosity behavior indicates, on one hand, that effects that lead to negative viscosity deviations, like the weakening of the dipole–dipole interactions between the pure components, are slightly predominant and, on the other hand, that the Cl–O interaction has a minor importance, as is shown by the similar viscosity behavior of the mixtures tetrahydrofuran with cyclohexane (Geier and Bittrich, 1974) and tetrahydrofuran with chlorocyclohexane.

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