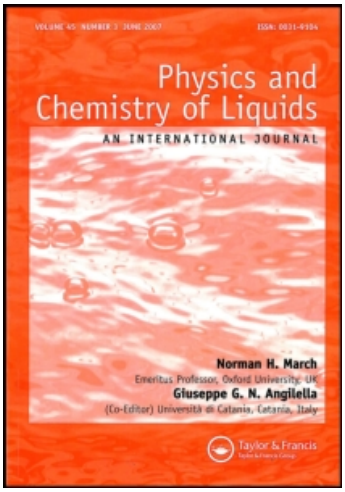

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Refractive indices and molar refractions of binary mixtures formed by some cyclic ethers and isomeric chlorobutanes

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Refractive indices and molar refractions of binary mixtures formed by some cyclic ethers and isomeric chlorobutanes

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Refractive indices of binary mixtures formed by a cyclic ether (tetrahydrofuran or tetrahydropyran) and each of the isomeric chlorobutanes (1-chlorobutane, 2-chlorobutane, 1-chloro-2-methylpropane and 2-chloro-2-methylpropane) have been measured at two temperatures, 298.15 K and 313.15 K. From experimental data, refractive index deviations and molar refractions have been calculated. Furthermore, several common mixing rules have been used to predict refractive indices of the mixtures from their experimental densities reported previously.

Keywords: Refractive index; Tetrahydrofuran; Tetrahydropyran; Chlorobutane

1. Introduction

The study of excess and mixture properties is normally used to get information about molecular processes that can occur during the mixing phenomena. In this respect, refractive index deviations defined on a volume fraction basis [1–3] can be related with excess volume and from its analysis interesting information about molecular mixing phenomena can be extracted.

Following previous studies of liquid mixtures involving cyclic ethers and chloroalkanes [4–11], we show here refractive indices of binary mixtures formed by tetrahydrofuran or tetrahydropyran and each one of the isomeric chlorobutanes measured at two temperatures, 298.15 K and 313.15 K. From experimental data, we have calculated refractive index deviations and molar refractions. Results have been discussed in terms of the type and nature of components and the specific interactions between them.

It is common to relate refractive index to thermodynamic properties like density, speed of sound or surface tension. In this article, we have used several common mixing rules: Lorentz–Lorenz [12,13], Gladstone–Dale [14], Laplace [15] and Eykman [16] to

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Table 1. Experimental and literature data of refractive indices, n_D, of pure components.

	n_D $T = 298.15$ K		n_D $T = 313.15$ K
	Exp.	Lit.	Exp.
Tetrahydrofuran	1.404636	1.40496[a]	1.396617
Tetrahydropyran	1.418743	1.41862[a]	1.410969
1-chlorobutane	1.399499	1.4001[b]	1.391332
2-chlorobutane	1.394043	1.3941[b]	1.385735
1-chloro-2-methyl propane	1.395284	1.3951[b]	1.386856
2-chloro-2-methyl propane	1.382242	1.3828[b]	1.373120

[a]Ref. [19].
[b]Ref. [20].

predict refractive indices of the mixtures from their experimental densities reported previously [17,18] and refractive index and density of the pure components.

2. Experimental section

Aldrich (1-chlorobutane and 2-chloro-2-methylpropane have a purity of 99%, tetrahydrofuran and 2-chlorobutane with purity better than 99.5% and 99% respectively) provided some of the chemicals used on experimental measurements, 1-chloro-2-methylpropane was purchased from Fluka (purity better than 99%) and tetrahydropyran, with a purity of 99%, was provided by Acros Organics. Additional purification has not been made. A comparison between experimental and literature data of refractive index of pure compounds [19,20] is reported in table 1.

Refractive indices corresponding to 589.3 nm sodium D wavelength were measured using a high precision automatic refractometer Abemat-HP DR. Kernchen. The temperature of the sample is controlled within $\pm 2 \cdot 10^{-3}$ K by a built-in Peltier device while another Peltier thermostat is used to kept constant the temperature of the internal refractometer components. The reproducibility of the measurement is $\pm 1 \cdot 10^{-6}$ and the corresponding accuracy is $\pm 2 \cdot 10^{-5}$. Details of the equipment and measure procedure have been described elsewhere [21].

The composition of each mixture was determined by using a mass balance Mettler H20T. The maximum estimated error in the mole fraction is $\pm 1 \cdot 10^{-4}$.

3. Results and discussion

From experimental results of refractive indices we have calculated refractive index deviations defined on a volume fraction basis [1,2], Δn_D, as follows:

$$\Delta n_D = n_D - \phi_1 n_{D1} - \phi_2 n_{D2} \quad (1)$$

where n_D is the refractive index of the mixture, ϕ_i and n_Di are the volume fraction referred to the unmixed state and the refractive index of component i, respectively.

Molar refraction, R_m, has been calculated by means of the Lorentz–Lorenz relation:

$$R_m = \frac{n_D^2 - 1}{n_D^2 + 2} V_m \tag{2}$$

where V_m is the molar volume of the mixture.

In table 2 appear experimental values of n_D as well as calculated values of Δn_D, and R_m for the studied mixtures. Refractive index deviations are graphically represented in figures 1–4.

Refractive index deviations and excess volumes at each temperature were correlated with a Redlich–Kister polynomial equation:

$$\Delta n_D = \phi_1(1 - \phi_1) \sum_i A_i (2\phi_1 - 1)^i \tag{3}$$

A_i being the adjustable parameters that are shown together with the standard deviations $\sigma(Y)$ in table 3.

According to Nakata and Sakurai [3] the sign of Δn_D is opposite to that of V^E if the behaviour of refractive index is not too non-linear between n_{D1} and n_{D2}. Revising values of excess volume reported before [17,18], we can conclude that in our mixtures, this rule is truly fulfilled in all the cases. Previous studies have revealed that other similar mixtures formed by some cyclic ethers containing two oxygen atoms like 1,3-dioxolane or 1,4-dioxane, and isomeric chlorobutanes also follow this rule [22,23].

Experimental results for the refractive index deviation of the mixtures containing tetrahydrofuran or tetrahydropyran are quite similar. All the values are positive and the sequence followed for a given cyclic ether at both temperatures is also the same: 1-chlorobutane < 1-chloro-2-methylpropane < 2-chlorobutane < 2-chloro-2-methylpropane. Another characteristic for these mixtures is that the ones containing 2-chlorobutane or 1-chloro-2-methylpropane show a similar behaviour, especially at the temperature of 313.15 K.

The effect of the temperature is not very significant, values of Δn_D being a little bigger if the temperature increases.

The values of the thermodynamic properties of solutions are the result of several energetic and structural effects. For the mixtures studied here, the energetic effects, i.e. the molecular interactions between the components of the mixture, are in one hand the disruption of dipole–dipole interactions in cyclic ethers and isomeric chlorobutanes, and on the other hand specific interactions between the oxygen and chloride atoms. Furthermore, structural effects like interstitial accommodation or changes in free volume also play an important role in the explanation of refractive behaviour.

Attending to the results obtained for other thermodynamic properties of the mixtures studied here [17,18,24,25], and paying attention to the experimental Δn_D values, we can conclude that the new specific interactions between the oxygen and chloride atoms are slightly more important than the weakening of the interactions of the pure compounds.

Differences regarding the isomeric chlorobutane can be explained in terms of structural effects. The linear isomer, 1-chlorobutane, is the chlorobutane that in, the pure state, has the smallest reduced molar free volume, i.e. the unoccupied part of the molar volume [26], as his values of molar refraction and isothermal compressibility show [27]. Therefore the possibility of interstitial accommodation of cyclic ether molecules is reduced. On other hand, while the 2-chloro-2-methylpropane has the

Table 2. Refractive indices, n_D, refractive index deviations, Δn_D, and molar refractions, R_m, for the studied mixtures.

ϕ_1	n_D	Δn_D	R_m ($cm^3 \cdot mol^{-1}$)	ϕ_1	n_D	Δn_D	R_m ($cm^3 \cdot mol^{-1}$)
Tetrahydrofuran (1) + 1-chlorobutane (2) at 298.15 K							
0.0531	1.399795	0.000023	25.088	0.6210	1.403010	0.000321	21.776
0.0890	1.399999	0.000043	24.847	0.7522	1.403622	0.000259	21.135
0.2591	1.400998	0.000168	23.768	0.8605	1.404085	0.000165	20.634
0.3544	1.401561	0.000242	23.207	0.9104	1.404283	0.000107	20.411
0.4772	1.402264	0.000314	22.525	0.1762	1.400504	0.000100	24.281
0.5235	1.402520	0.000332	22.278				
Tetrahydrofuran (1) + 2-chlorobutane (2) at 298.15 K							
0.0210	1.394340	0.000075	25.381	0.5381	1.400625	0.000882	22.207
0.0739	1.395070	0.000244	25.011	0.5882	1.401124	0.000851	21.944
0.1333	1.395870	0.000415	24.610	0.7151	1.402320	0.000702	21.310
0.2846	1.397790	0.000733	23.649	0.8851	1.403759	0.000341	20.522
0.3563	1.398642	0.000825	23.221	0.9189	1.404022	0.000245	20.373
0.4683	1.399899	0.000895	22.584				
Tetrahydrofuran (1) + 1-chloro-2-methylpropane (2) at 298.15 K							
0.0648	1.396082	0.000192	25.040	0.5719	1.401310	0.000677	22.020
0.1086	1.396602	0.000302	24.744	0.6880	1.402300	0.000582	21.435
0.1603	1.397202	0.000419	24.405	0.7727	1.402974	0.000464	21.029
0.2786	1.398489	0.000600	23.665	0.8885	1.403851	0.000258	20.502
0.3579	1.399300	0.000669	23.194	0.9871	1.404549	0.000034	20.078
0.4805	1.400484	0.000706	22.506				
Tetrahydrofuran (1) + 2-chloro-2-methylpropane (2) at 298.15 K							
0.0668	1.384330	0.000591	25.261	0.4091	1.393612	0.002209	22.992
0.0783	1.384680	0.000685	25.176	0.5119	1.395910	0.002204	22.399
0.1393	1.386514	0.001152	24.737	0.6487	1.398650	0.001880	21.664
0.2668	1.390068	0.001852	23.875	0.7680	1.400840	0.001399	21.070
0.3288	1.391670	0.002065	23.480	0.9227	1.403440	0.000534	20.357
Tetrahydrofuran (1) + 1-chlorobutane (2) at 313.15 K							
0.0470	1.391619	0.000038	25.153	0.5656	1.394565	0.000244	22.076
0.0741	1.391782	0.000058	24.968	0.6429	1.394949	0.000219	21.683
0.1614	1.392315	0.000130	24.397	0.7414	1.395423	0.000173	21.204
0.3202	1.393246	0.000222	23.426	0.9079	1.396196	0.000066	20.445
0.4175	1.393787	0.000248	22.871	0.9593	1.396433	0.000031	20.222
0.4506	1.393965	0.000251	22.688				
Tetrahydrofuran (1) + 2-chlorobutane (2) at 313.15 K							
0.0528	1.386430	0.000121	25.180	0.5655	1.392690	0.000801	22.085
0.1101	1.387223	0.000290	24.786	0.6884	1.393890	0.000664	21.462
0.1779	1.388140	0.000469	24.338	0.7832	1.394760	0.000502	21.007
0.2784	1.389444	0.000679	23.708	0.8879	1.395675	0.000278	20.532
0.3907	1.390800	0.000814	23.044	0.9412	1.396130	0.000153	20.299
0.4804	1.391800	0.000838	22.541				
Tetrahydrofuran (1) + 1-chloro-2-methylpropane (2) at 313.15 K							
0.0450	1.387431	0.000136	25.205	0.6153	1.393510	0.000648	21.819
0.0804	1.387875	0.000234	24.960	0.6734	1.394011	0.000581	21.529
0.1679	1.388934	0.000439	24.378	0.7675	1.394795	0.000447	21.077
0.2700	1.390110	0.000619	23.739	0.8714	1.395632	0.000270	20.603
0.3788	1.391272	0.000719	23.097	0.9836	1.396490	0.000033	20.118
0.5102	1.392562	0.000726	22.368				
Tetrahydrofuran (1) + 2-chloro-2-methylpropane (2) at 313.15 K							
0.0318	1.374206	0.000338	25.536	0.5307	1.387960	0.002371	22.311
0.0941	1.376253	0.000922	25.074	0.6281	1.389987	0.002108	21.789
0.1435	1.377792	0.001301	24.721	0.7395	1.392163	0.001666	21.229
0.2275	1.380276	0.001810	24.144	0.8563	1.394243	0.001002	20.679

(*Continued*)

Table 2. Continued.

ϕ_1	n_D	Δn_D	R_m ($cm^3 \cdot mol^{-1}$)	ϕ_1	n_D	Δn_D	R_m ($cm^3 \cdot mol^{-1}$)
0.3157	1.382785	0.002246	23.578	0.9262	1.395313	0.000430	20.362
0.4274	1.385637	0.002475	22.901				
Tetrahydropyran (1) + 1-chlorobutane (2) at 298.15 K							
0.0465	1.400430	0.000036	25.418	0.6095	1.411373	0.000144	25.002
0.1033	1.401561	0.000074	25.375	0.6694	1.412510	0.000130	24.961
0.1910	1.403291	0.000116	25.308	0.8066	1.415111	0.000089	24.867
0.3095	1.405617	0.000161	25.219	0.8814	1.416518	0.000058	24.817
0.3877	1.407127	0.000168	25.161	0.9610	1.418010	0.000018	24.763
0.5026	1.409332	0.000162	25.077				
Tetrahydropyran (1) + 2-chlorobutane (2) at 298.15 K							
0.0369	1.395040	0.000086	25.501	0.5948	1.409407	0.000673	25.040
0.0979	1.396690	0.000230	25.449	0.6966	1.411825	0.000577	24.957
0.1993	1.399407	0.000441	25.363	0.7963	1.414141	0.000430	24.877
0.3013	1.402075	0.000590	25.278	0.9391	1.417385	0.000145	24.773
0.3960	1.404505	0.000681	25.201	0.9832	1.418358	0.000031	24.746
0.4975	1.407045	0.000713	25.119				
Tetrahydropyran (1) + 1-chloro-2-methylpropane (2) at 298.15 K							
0.0390	1.396288	0.000090	25.454	0.6673	1.411453	0.000514	24.967
0.1008	1.397889	0.000239	25.399	0.8269	1.414962	0.000280	24.853
0.2040	1.400508	0.000439	25.317	0.9004	1.416549	0.000143	24.803
0.4901	1.407423	0.000641	25.100	0.5974	1.409894	0.000595	25.020
0.7312	1.412870	0.000433	24.921	0.9480	1.417590	0.000067	24.771
0.2948	1.402764	0.000563	25.248				
Tetrahydropyran (1) + 2-chloro-2-methylpropane (2) at 298.15 K							
0.0246	1.383383	0.000242	25.745	0.5084	1.403037	0.002238	25.223
0.0908	1.386368	0.000813	25.674	0.5859	1.40575	0.002121	25.143
0.1836	1.390358	0.001415	25.570	0.7003	1.409579	0.001774	25.029
0.2866	1.394631	0.001928	25.457	0.8057	1.412943	0.001291	24.925
0.3830	1.398423	0.002200	25.354	0.8876	1.415394	0.000753	24.842
Tetrahydropyran (1) + 1-chlorobutane (2) at 313.15 K							
0.0444	1.392260	0.000056	25.443	0.5840	1.403000	0.000200	25.035
0.1061	1.393535	0.000120	25.393	0.6889	1.405040	0.000180	24.960
0.2067	1.395590	0.000199	25.315	0.7776	1.406749	0.000147	24.898
0.2948	1.397335	0.000213	25.247	0.8958	1.409025	0.000101	24.821
0.3872	1.399164	0.000228	25.179	0.9772	1.410543	0.000021	24.772
0.4986	1.401334	0.000211	25.097				
Tetrahydropyran (1) + 2-chlorobutane (2) at 313.15 K							
0.0326	1.386658	0.000099	25.533	0.5717	1.401000	0.000838	25.086
0.0976	1.388498	0.000299	25.477	0.7012	1.404117	0.000687	24.981
0.2031	1.391440	0.000581	25.389	0.8020	1.406459	0.000487	24.901
0.3004	1.394066	0.000750	25.308	0.8979	1.408680	0.000287	24.831
0.4029	1.396765	0.000862	25.224	0.9902	1.410755	0.000033	24.766
0.4946	1.399085	0.000869	25.149				
Tetrahydropyran (1) + 1-chloro-2-methylpropane (2) at 313.15 K							
0.0417	1.388031	0.000168	25.494	0.5729	1.401429	0.000758	25.066
0.0883	1.389316	0.000331	25.457	0.6801	1.403892	0.000636	24.981
0.1664	1.391374	0.000505	25.391	0.7852	1.406248	0.000458	24.900
0.2703	1.394056	0.000682	25.306	0.8719	1.408166	0.000285	24.838
0.3788	1.396771	0.000781	25.219	0.9358	1.409562	0.000142	24.796
0.4615	1.398792	0.000807	25.154				
Tetrahydropyran (1) + 2-chloro-2-methylpropane (2) at 313.15 K							
0.0653	1.376146	0.000555	25.735	0.5816	1.397524	0.002390	25.284
0.0927	1.377580	0.000953	25.726	0.6790	1.400921	0.002103	25.170
0.1892	1.382049	0.001767	25.663	0.7957	1.404738	0.001503	25.022
0.2903	1.386344	0.002238	25.578	0.8887	1.407604	0.000848	24.899
0.4056	1.391010	0.002538	25.473	0.9469	1.409536	0.000576	24.833

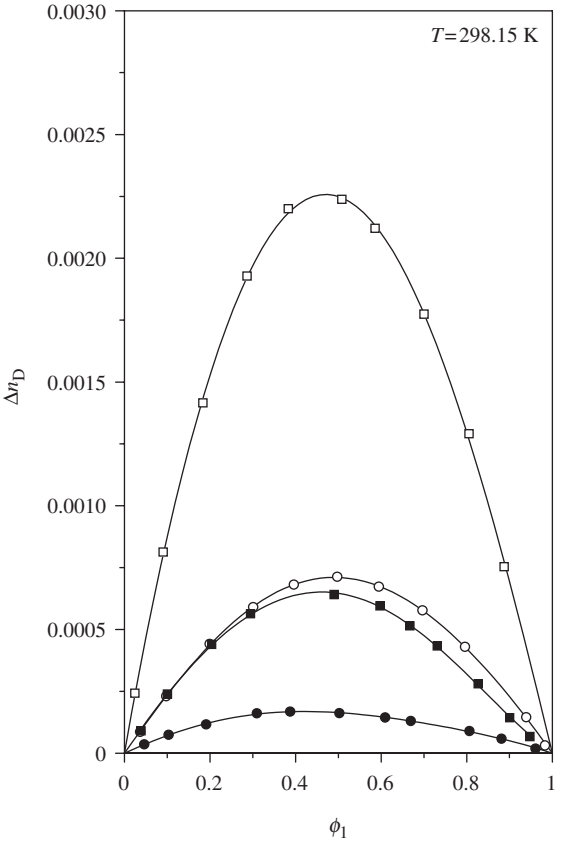

Figure 1. Refractive index deviations for tetrahydrofuran (1) + isomeric chlorobutane (2) at 298.15 K as a function of volume fraction, ϕ_1: 1-chlorobutane (●), 2-chlorobutane (○), 1-chloro-2-methylpropane (■), 2-chloro-2-methylpropane (□); (——) Redlich–Kister equation.

biggest reduced molar free volume, therefore tetrahydrofuran or tetrahydropyran can be easily accommodated leading to bigger Δn_D values. Refractive index deviations for 2-chlorobutane and 1-chloro-2-methylpropane show intermediate values between 1-chlorobutane and 2-chloro-2-methylpropane, according to the pure state properties of these isomers.

4. Refractive index mixing rules

The refractive index of a liquid mixture can be predicted from his density together with the refractive indices and densities of the pure components by using a mixing rule:

$$\left(\frac{1}{\rho}\right) f(n_D) = \left(\frac{w_1}{\rho_1}\right) f(n_{D1}) + \left(\frac{w_2}{\rho_2}\right) f(n_{D2}) \tag{4}$$

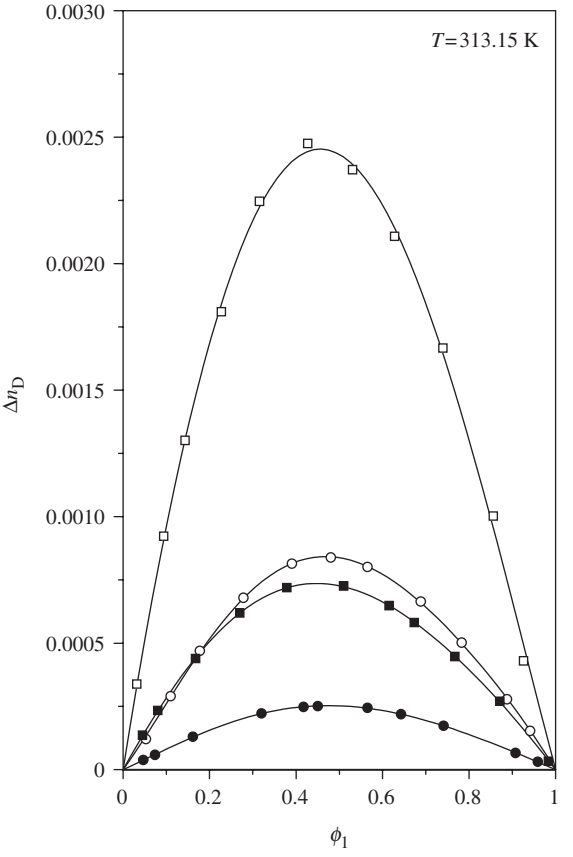

Figure 2. Refractive index deviations for tetrahydrofuran (1) + isomeric chlorobutane (2) at 313.15 K as a function of volume fraction, ϕ_1: 1-chlorobutane (●), 2-chlorobutane (○), 1-chloro-2-methylpropane (■), 2-chloro-2-methylpropane (□); (——) Redlich–Kister equation.

where w_i is the weight fraction of component i and $f(n_D)$ denotes a function of the refractive index. The mixing rules used in this article were [12–16]:

$$\text{Lorentz–Lorenz (LL)}: \quad f(n_D) = \frac{n_D^2 - 1}{n_D^2 + 2} \tag{5}$$

$$\text{Gladstone–Dale (GD)}: \quad f(n_D) = n_D - 1 \tag{6}$$

$$\text{Laplace (LP)}: \quad f(n_D) = n_D^2 - 1 \tag{7}$$

$$\text{Eykman (EK)}: \quad f(n_D) = \frac{n_D^2 - 1}{n_D + 0.4} \tag{8}$$

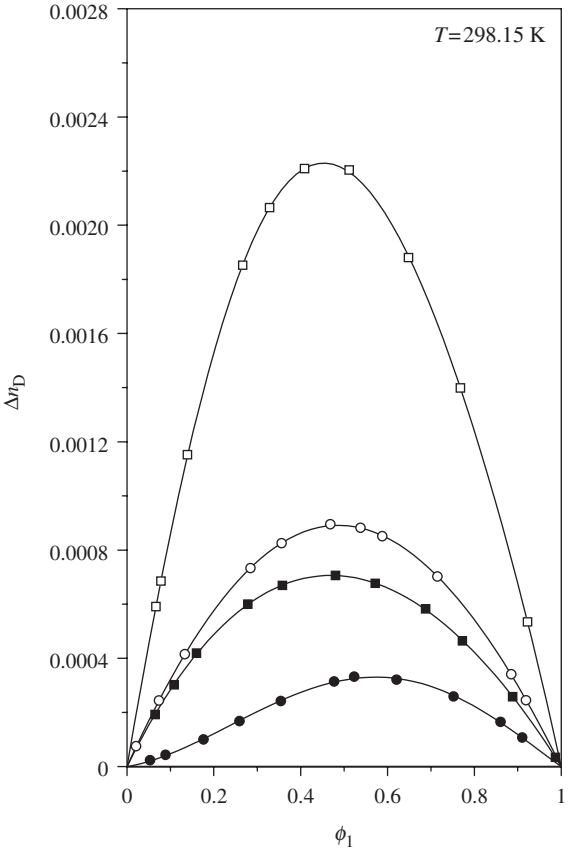

Figure 3. Refractive index deviations for tetrahydropyran (1) + isomeric chlorobutane (2) at 298.15 K as a function of volume fraction, ϕ_1: 1-chlorobutane (●), 2-chlorobutane (○), 1-chloro-2-methylpropane (■), 2-chloro-2-methylpropane (□); (——) Redlich–Kister equation.

Experimental refractive indices were compared with those predicted by these mixing rules. The root mean square deviations, RMSD, between experimental and predicted n_D defined as:

$$\mathrm{RMSD} = \left(\frac{1}{N} \left(\sum_i n_{D,\mathrm{exp}} - n_{D,\mathrm{pred}} \right)^2 \right)^{1/2} \tag{9}$$

where N is the number of experimental data, are given in table 4.

As RMSD values indicate, refractive indices are predicted with high accuracy for all the mixtures studied in this work. Furthermore, if temperature increases, RMSD values also increase. We should remark that all the mixing rules provide similar predictions, being results slightly worse for the mixing rule suggested by Laplace.

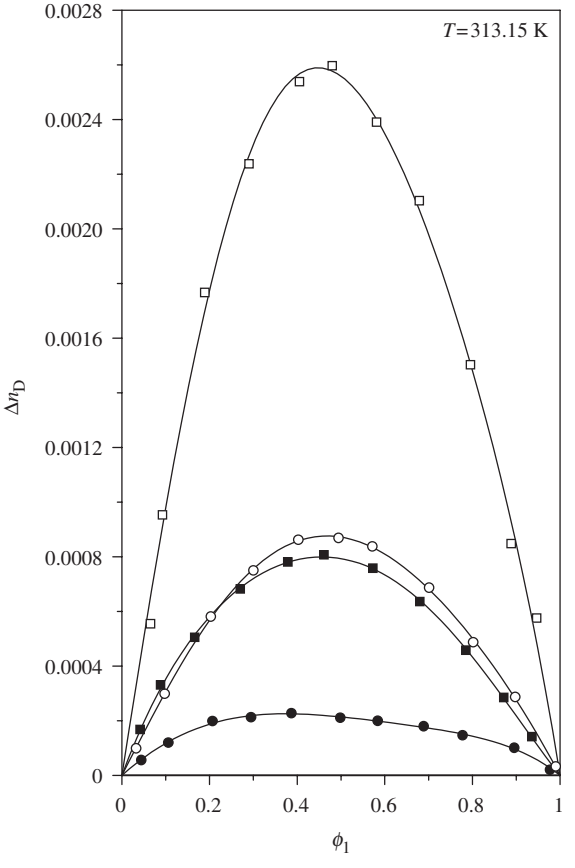

Figure 4. Refractive index deviations for tetrahydropyran (1) + isomeric chlorobutane (2) at 313.15 K as a function of volume fraction, ϕ_1: 1-chlorobutane (●), 2-chlorobutane (○), 1-chloro-2-methylpropane (■), 2-chloro-2-methylpropane (□); (——) Redlich–Kister equation.

Table 3. Parameters, A_i, and standard deviations, σ, for the Redlich–Kister equation.

T (K)	A_0	A_1	A_2	A_3	σ
Tetrahydrofuran (1) + 1-chlorobutane (2)					
298.15	0.001279	0.000576	−0.000565	−0.000131	0.000005
313.15	0.001010	−0.000130	−0.000240	0.000110	0.000003
Tetrahydrofuran (1) + 2-chlorobutane (2)					
298.15	0.003563	−0.000195	−0.000181	0.000054	0.000003
313.15	0.003350	−0.000540	−0.000770	0.000770	0.000006
Tetrahydrofuran (1) + 1-chloro-2-methylpropane (2)					
298.15	0.002814	−0.000339	0.000092	−0.000010	0.000004
313.15	0.002910	−0.000690	−0.000250	0.000290	0.000005
Tetrahydrofuran (1) + 2-chloro-2-methylpropane (2)					
298.15	0.008834	−0.001814	−0.000490	0.000845	0.000004
313.15	0.009728	−0.001921	−0.001071	−0.000184	0.000039

(*Continued*)

Table 3. Continued.

T (K)	A_0	A_1	A_2	A_3	σ
Tetrahydropyran (1) + 1-chlorobutane (2)					
298.15	0.000655	−0.000224	0.000026	0.000123	0.000004
313.15	0.000856	−0.000316	0.000500	0.000242	0.000006
Tetrahydropyran (1) + 2-chlorobutane (2)					
298.15	0.002840	−0.000131	−0.000417	0.000130	0.000004
313.15	0.003489	−0.000508	−0.000400	0.000458	0.000007
Tetrahydropyran (1) + 1-chloro-2-methylpropane (2)					
298.15	0.002585	−0.000511	−0.000682	−0.000170	0.000008
313.15	0.003174	−0.000552	−0.000007	−0.000593	0.000009
Tetrahydropyran (1) + 2-chloro-2-methylpropane (2)					
298.15	0.008999	−0.001122	−0.000469	−0.000218	0.000017
313.15	0.010235	−0.002310	−0.000150	0.002184	0.000070

Table 4. Root mean square deviations, RMSD, for the Lorentz–Lorentz (LL), Gladstone–Dale (GD), Laplace (LP) and Eykman (EK) equations.

T (K)	LL	GD	LP	EK
Tetrahydrofuran (1) + 1-chlorobutane (2)				
298.15	0.00004	0.00005	0.00007	0.00005
313.15	0.00008	0.00006	0.00003	0.00007
Tetrahydrofuran (1) + 2-chlorobutane (2)				
298.15	0.00002	0.00004	0.00011	0.00002
313.15	0.00011	0.00004	0.00005	0.00007
Tetrahydrofuran (1) + 1-chloro-2-methylpropane (2)				
298.15	0.00003	0.00003	0.00007	0.00003
313.15	0.00012	0.00006	0.00003	0.00009
Tetrahydrofuran (1) + 2-chloro-2-methylpropane (2)				
298.15	0.00002	0.00013	0.00029	0.00013
313.15	0.00004	0.00015	0.00030	0.00009
Tetrahydropyran (1) + 1-chlorobutane (2)				
298.15	0.00003	0.00003	0.00003	0.00003
313.15	0.00016	0.00014	0.00012	0.00015
Tetrahydropyran (1) + 2-chlorobutane (2)				
298.15	0.00005	0.00005	0.00005	0.00005
313.15	0.00004	0.00005	0.00007	0.00005
Tetrahydropyran (1) + 1-chloro-2-methylpropane (2)				
298.15	0.00009	0.00008	0.00006	0.00008
313.15	0.00011	0.00009	0.00009	0.00009
Tetrahydropyran (1) + 2-chloro-2-methylpropane (2)				
298.15	0.00013	0.00022	0.00032	0.00017
313.15	0.00013	0.00020	0.00030	0.00016

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