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Activity Coefficients at Infinite Dilution and Excess Molar Volumes in Binary Mixtures Containing Normal Alkanes (Nonane, Decane, Undecane, or Dodecane) and Cresols (2-Methylphenol or 3-Methylphenol)

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The differential ebulliometry method was used to measure activity coefficients at infinite dilution for cresols (2-methylphenol or 3-mehtylphenol) with nonane, decane, or undecane a in a range of temperature from (373 to 433) K. Temperature-dependent parameters were determined for the UNIQUAC model. Densities of binary mixtures of systems containing 2-cresol + nonane or decane or undecane and 3-cresol + nonane or undecane or dodecane have been measured at 308.15 K under atmospheric pressure. With these data, excess molar volumes (V^E) have been calculated. The excess molar volumes were fitted to the Redlich−Kister polynomial equation.

Introduction

In previous papers, isothermal vapor−liquid equilibria in binary mixtures of phenol or cresols and normal alkanes were reported.[1,2] In the present paper, activity coefficients at infinite dilution at different temperatures in the range from (373 to 433) K for 2-cresol + alkanes and 3-cresol + alkanes are presented. By using the VLE data[1,2] and the activity coefficients at infinite dilution, UNIQUAC[3] model parameters were estimated. Furthermore, excess molar volumes of such binary mixtures were measured at 308.15 K. Cresol + alkanes mixtures are important in process engineering design for coal chemistry, and there is interest in the resulting thermophysical data for environmental rehabilitation.

Experimental Section

Chemicals. 2-Methylphenol (2-cresol, Aldrich Chemical Co., Steinheim, Germany, purity >98%) and 3-methylphenol (3-cresol, Merck-Schuchardt, Hohenbrunn, Germany, purity >99%) were fractionally distilled twice in a Vigreux column at reduced pressure under an N_2 atmosphere and dried over Na_2SO_4. Nonane (Merck-Schuchardt, Hohenbrunn, Germany, purity >99%), decane and undecane (Petrolchemisches Kombinat, Schwedt, Germany, purity >98%), and dodecane (J. T. Baker B. V., Deventer, Netherlands, purity >98%) were also fractionally distilled twice in a Vigreux column at reduced pressure and dried over type 4A molecular sieves. The mass-fraction purities of the substances were 2-cresol (99.1%), 3-cresol (99.9%), nonane (99.9%), decane (99.4%), undecane (99.9%), and dodecane (99.7%), as determined by gas−liquid chromatography. The purity was also verified by comparing experimental values of the refractive index and density with those reported in the literature (Table 1). The experimental and the literature values match well; only decane showed larger deviations from the literature values of density. The purity of decane is 99.4%, which could explain the size of the deviation.

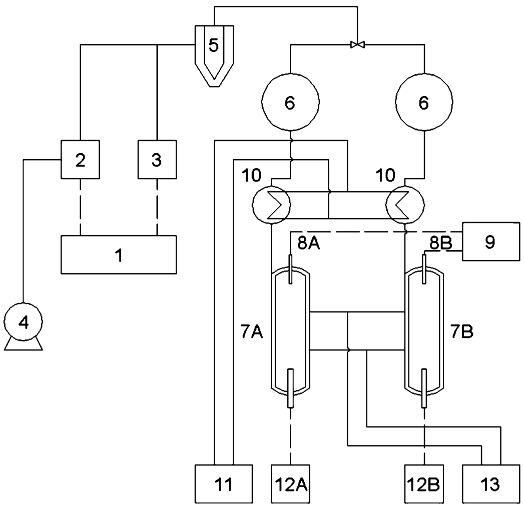

Figure 1. Experimental setup and equipment for comparative ebulliometry: (1) PC, (2) pressure controller, (3) pressure indicator, (4) vacuum pump, (5) cold trap, (6) buffer volumes, (7) ebulliometer, (8) Pt-100 resistance thermometer, (9) digital readout for resistance thermometer, (10) reflux condenser, (11) thermostat, (12) heating units, (13) thermostat.

Table 1. Comparison of the Experimental Refractive Index, n_D, and Density, ρ, of Pure Liquids with Literature Values

		n_D		ρ/g cm^{-3}	
liquid	*T*/K	exptl	lit	exptl	lit
2-cresol	308.15	1.5386	1.5386[8]	1.03273	1.0327[8]
3-cresol	293.15	1.5401	1.5401[8]	1.03385	1.0339[8]
n-nonane	293.15	1.4058	1.4058[8]	0.71916	0.7192[8]
n-decane	308.15	1.4055	1.4052[9]	0.72026	0.71884[9]
n-undecane	293.15	1.4164	1.4164[8]	0.74026	0.7402[8]
n-dodecane	293.15	1.4210	1.4210[8]	0.74945	0.7495[8]

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Table 2. Isothermal VLE Data[1,2] That Were Fitted Simultaneously with the Activity Coefficients at Infinite Dilution to the UNIQUAC Model

system	temperatures/K, number of data points
2-cresol/*n*-nonane	373.15 (15), 393.15 (16), 413.15 (15)
2-cresol/*n*-decane	393.15 (24), 413.15 (24), 433. 15 (23)
2-cresol/n-undecane	393.15 (18), 413.15 (19), 433. 15 (19)
3-cresol/*n*-nonane	393.15 (14), 413.15 (14), 433. 15 (15)
3-cresol/*n*-decane	373.15 (13), 433. 15 (12)
3-cresol/*n*-undecane	373.15 (15), 393.15 (14), 413.15 (15)

Apparatus and Procedure. The differential ebulliometry method for determining activity coefficients at infinite dilution is based on the measurement of the boiling temperature at constant pressure as a function of composition. Twin ebulliometers were used; the reference ebulliometer contained only the solvent, and the other one, solvent and a small amount of solute. The details of the entire apparatus used for this measurement and the additional equipment are shown in Figure 1. The technique provides the boiling temperature difference between both of the ebulliometers. The equation for the activity coefficient at infinite dilution was derived by Dohnal and Novotná[4] and is given by

$$\gamma_i^{\infty} = \frac{\epsilon_i^{\infty} P_j^{\mathrm{sat}}}{P_i^{\mathrm{S}}}\left[1 - \beta\left(\frac{\partial T}{\partial x_i}\right)_P^{\infty}\right] \qquad (1)$$

where

$$\epsilon_i^{\infty} = \exp\left[\frac{(B_{ii} - V_i^{\mathrm{L}})(P_j^{\mathrm{sat}} - P_i^{\mathrm{sat}}) + \delta_{ij} P_j^{\mathrm{sat}}}{RT}\right]$$

$$\beta = \left(1 + P_j^{\mathrm{sat}} \frac{B_{jj} - V_j^{\mathrm{L}}}{RT}\right)\frac{\mathrm{d}\ln P_j^{\mathrm{sat}}}{\mathrm{d}T}$$

$$\delta_{ij} = 2B_{ij} - B_{ii} - B_{jj}$$

P_i^{sat} and V_i^{L} are the pure-component vapor pressure and liquid molar volumes, B_{ii} and B_{ij} are the second virial coefficients, R is the gas constant, and T is the absolute temperature. The equation includes no assumption about the nonideality of the liquid phase. Experimental data are used to determine the limiting slope at infinite dilution $(\partial T/\partial x_i)_P^{\infty}$. Furthermore, the difference between the gravimetrically determined overall composition and the equilibrium liquid composition is corrected as shown by Dohnal and Novotná.[4] They introduced ebulliometer constants that were used in the material balance equations for the correction. Calibration measurements in the ebulliometers, used for this work, for several systems for which reliable and accurate activity coefficients at infinite dilution data were available from the literature have shown that the same ebulliometer constants proposed by Dohnal and Novotná[4] can be used. This is a result of the fact that the constants depend on the interior surface of the ebulliometer on which a film of equilibrium liquid is present. The ebulliometer of Dohnal and those used for this work are similar in design and this mentioned interior surface. Because of this correction, only systems with a relative volatility α_{ij} (eq 2) between 0.2 and 8 can be measured. In systems with a relative volatility beyond these limits, the error of the correction becomes unacceptable.

$$\alpha_{ij}^{\infty} = \frac{\gamma_i^{\infty} P_i^{\mathrm{sat}}}{P_j^{\mathrm{sat}}} \qquad (2)$$

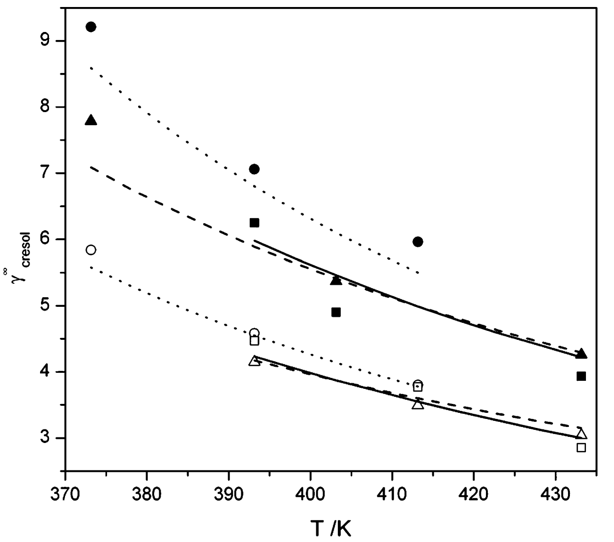

Figure 2. Experimental activity coefficients at infinite dilution and correlations of the UNIQUAC model with parameters fit to activity coefficients at infinite dilution with VLE data for 2-cresol + alkanes and 3-cresol + alkanes. Experimental results: ○, 2-cresol + *n*-nonane; △, *n*-decane; □, *n*-undecane; ●, 3-cresol + *n*-nonane; ▲, *n*-decane; ■, *n*-undecane. UNIQUAC results: ···, cresols + *n*-nonane; - - -, cresols + *n*-decane; −, cresols + *n*-undecane.

The ebulliometers are connected by a manifold to a high-accuracy pressure controller (Druck Limited, DPI 520) and a digital output pressure transducer (Druck Limited, RTP 301) with an accuracy of 0.06 kPa. The boiling temperature is measured with a Pt-100 resistance thermometer (Automatic Systems Laboratories Ltd., F250MkII) with an absolute accuracy of ±0.03 K, and the measurement of the boiling temperature difference is stable to within ±0.002 K. The ebulliometers are designed with Cottrell pumps, which increase the vapor−liquid contact and assist in a quicker approach to the steady state. The ebulliometer was gravimetrically filled, and after achieving the boiling temperature by adjusting the pressure controller, a small amount of the solute was weighed and injected. After equilibrium was achieved, the temperature difference was recorded. The procedure of adding solute was repeated until the concentration of solute was between (0.03 and 0.04) mole fraction.

The limiting slope was obtained by linear regression. A higher-order polynomial did not give a better result when fitting the data. The limiting slopes were used to calculate the activity coefficients at infinite dilution, and the required virial coefficients were estimated by the method of Hayden and O'Connell.[5] The accuracy of weighing is ±0.5 mg; with respect to the loadings therewith, the uncertainty in x is ±0.0001. Experimental errors in the activity coefficients at infinite dilution are less than 7%.

For the determination of excess molar volumes, series of mixtures were prepared by mass and measured in a vibrating tube density meter (model DMA 58, Anton Paar, Graz, Austria) with a precision of 0.00002 g/cm^3. The experimental uncertainty in T is ±0.01 K, and for x_i, it is ±0.0002. Therewith, the total error estimated for these systems in V^{E} is ±0.018 cm^3 mol^{-1}.

Results and Discussions

The measured activity coefficients at infinite dilution were fitted simultaneously over the entire range of VLE data[1,2] (Table 2) using the UNIQUAC model with linear

Table 3. Experimental and UNIQUAC Activity Coefficients at Infinite Dilution

component 1 in component 2	T/K	γ_1^∞	γ_1^∞ UNIQUAC
2-cresol(1)/n-nonane (2)	373.15	5.84	5.58
	393.15	4.58	4.55
	413.15	3.8	3.78
2-cresol(1)/n-decane (2)	393.15	4.15	4.50
	413.15	3.49	3.60
	433.15	3.04	3.15
2-cresol(1)/n-undecane (2)	393.15	4.47	4.23
	413.15	3.77	3.55
	433.15	2.85	3.00
3-cresol(1)/n-nonane (2)	373.15	9.21	8.59
	393.15	7.06	6.81
	413.15	5.96	5.50
3-cresol(1)/n-decane (2)	373.15	7.79	7.09
	403.15	5.37	5.41
	433.15	4.26	4.29
3-cresol(1)/n-undecane (2)	393.15	6.25	5.98
	413.15	4.90	4.99
	433.15	3.93	4.22

temperature-dependent C_{ij} parameters given by eq 3. The results are shown in Figure 2.

$$\frac{C_{ij}}{R} = C_{ij}^{\mathrm{C}} + C_{ij}^{\mathrm{T}}(T - 273.15) \qquad C_{ij} = u_{ij} - u_{jj} \tag{3}$$

Coefficients C_{ij}^{C} and C_{ij}^{T} were evaluated by minimizing the objective function Q of Renon,[6] given by

$$Q = \Pi_1 \sum_i \left(\frac{100}{P_{\mathrm{exptl}}}\right)_i^2 (P_{\mathrm{calcd}} - P_{\mathrm{exptl}})_i^2 + \Pi_2 \sum_i (100)_i^2 (y_{i,\mathrm{calcd}} - y_{i,\mathrm{exptl}})_i^2 + \Pi_3 \sum_i \left(\frac{10}{\gamma_{\mathrm{exptl}}^\infty}\right)_i^2 (\gamma_{\mathrm{calcd}}^\infty - \gamma_{\mathrm{exptl}}^\infty)_i^2 \tag{4}$$

where the summation is made over all data points, Π_i is a weighting factor, P is the pressure, y is the equilibrium vapor-phase mole fraction, and γ^∞ is the activity coefficient at infinite dilution.

The results for the measurements and the deviations of the fitting are reported in Tables 3 and 4. The deviations for the pressure and vapor-phase composition of the VLE data are altogether small, and for the activity coefficients at infinite dilution, they are of the same magnitude as the experimental error. The mixtures with alkanes as solutes and cresol as the solvent are indeed just within the limits of the relative volatility but the behavior of boiling was very poor, and differences in boiling temperatures appeared between the series of measurements. The arising error was intolerable in its magnitude, so the obtained data must be neglected.

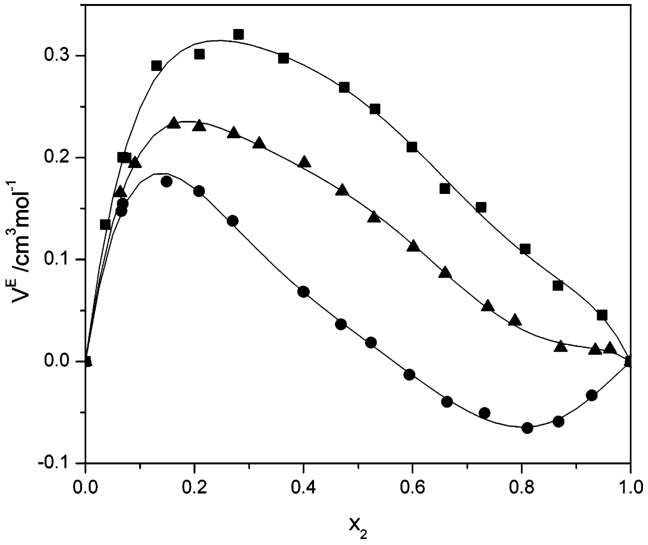

Figure 3. Excess molar volumes V^{E} for 2-cresol (2) + alkanes (1) at 308.15 K. Experimental results: ●, n-nonane; ▲, n-decane; ■, n-undecane. Calculated values from the Redlich−Kister equation (eq 6).

The activity coefficients at infinite dilution decrease with increasing temperature and the number of C atoms in systems of 2-cresol and 3-cresol. These activity coefficients show the typical behavior of positive deviations from Raoult's law, which become smaller with increasing temperature. The system 2-cresol + undecane does not follow this systematic trend. However, this could be explained by experimental errors. Generally, the activity coefficients at infinite dilution of 3-cresol are larger than the 2-cresol values because the methyl group is further away from the hydroxyl group and therefore the self-association effects have a stronger influence.

The excess molar volumes (V^{E}) were calculated using eq 5 with the densities ρ of the mixtures, densities ρ_i of pure components i, their molar masses M_i, and mole fractions x_i of components i:

$$V^{\mathrm{E}}/\mathrm{cm}^3\,\mathrm{mol}^{-1} = \frac{x_1 M_1 + x_2 M_2}{\rho} - \frac{x_1 M_1}{\rho_1} - \frac{x_2 M_2}{\rho_2} \tag{5}$$

The experimental ρ and V^{E} values are listed in Table 5 and are graphically presented in Figures 3 and 4.

The V^{E} data were fitted to the Redlich−Kister polynomial equation.[7]

$$V_{\mathrm{calcd}}^{\mathrm{E}}/\mathrm{cm}^3\,\mathrm{mol}^{-1} = x_1 x_2 \sum_{i=1}^{k} A_i (x_1 - x_2)^{i-1} \tag{6}$$

All coefficients A_i of the six systems and the standard deviations are listed in Table 6.

The excess molar volumes increase within the homologous series of the alkanes with the number of C atoms. The excess molar volumes of mixtures with 3-cresol are more negative than those with 2-cresol. Mixtures with 3-cresol give characteristic S-shaped V^{E} curves with nonane and undecane, whereas 2-cresol systems with nonane only give these curves. Comparable results were presented by

Table 4. UNIQUAC Parameters and Deviations

component 1 in component 2	C_{12}^{C}/K	C_{21}^{C}/K	C_{12}^{T}/K	C_{21}^{T}/K	ΔP^a/%	Δy^b/mol %	$\Delta\gamma_1^{\infty a}$/%
2-cresol(1)/n-nonane(2)	−83.02	363.07	0.0893	−0.8398	1.27	2.30	3.3
2-cresol(1)/n-decane(2)	−36.75	275.03	−0.0748	−0.4133	1.03	1.18	3.5
2-cresol(1)/n-undecane(2)	−195.63	498.11	0.8368	−1.7193	0.96	0.94	6.8
3-cresol(1)/n-nonane(2)	−129.51	478.61	0.2340	−1.1013	1.93	1.19	6.5
3-cresol(1)/n-decane(2)	−60.23	346.38	−0.1395	−0.3557	2.54	1.40	5.4
3-cresol(1)/n-undecane(2)	−155.37	486.13	0.3720	−1.1030	1.26	1.22	5.1

[a] $\Delta Z = 100(1/n_{\mathrm{p}} \sum((Z_{\mathrm{calcd}} - Z_{\mathrm{exptl}})/Z_{\mathrm{exptl}})^2)^{1/2}$, where n_{p} is the number of data points and Z represents P or γ_1^∞. [b] $\Delta y = (\sum(|y_{\mathrm{calcd}} - y_{\mathrm{exptl}}|)^2/n_{\mathrm{p}})^{1/2}$.

Table 5. Density, ρ, and Excess Molar Volume, V^E, for Alkanes (1) + 2-Cresol (2) and Alkanes (1) + 3-Cresol (2) at 308.15 K

x_2	ρ/g cm^{-3}	V^E/cm^3 mol^{-1}	x_2	ρ/g cm^{-3}	V^E/cm^3 mol^{-1}	x_2	ρ/g cm^{-3}	V^E/cm^3 mol^{-1}	x_2	ρ/g cm^{-3}	V^E/cm^3 mol^{-1}
					n-Nonane (1) + 2-Cresol (2)						
0.0000	0.70741	0.000	0.2703	0.76408	0.138	0.5237	0.83366	0.019	0.8106	0.93953	−0.065
0.0661	0.71958	0.148	0.3994	0.79732	0.069	0.5941	0.85651	−0.013	0.8674	0.96512	−0.059
0.0684	0.72002	0.155	0.4008	0.79769	0.068	0.6631	0.88076	−0.040	0.9285	0.99476	−0.033
0.1489	0.73650	0.177	0.4688	0.81704	0.037	0.7323	0.90698	−0.051	1.0000	1.03273	0.000
0.2085	0.74960	0.167									
					n-Decane (1) + 2-Cresol (2)						
0.0000	0.72024	0.000	0.2720	0.77090	0.223	0.6014	0.85840	0.112	0.8714	0.96458	0.014
0.0637	0.73049	0.166	0.3186	0.78132	0.214	0.6596	0.87802	0.087	0.9348	0.99628	0.011
0.0908	0.73520	0.194	0.4014	0.80121	0.195	0.7379	0.90695	0.054	0.9617	1.01078	0.012
0.1620	0.74833	0.233	0.4707	0.81947	0.167	0.7876	0.92704	0.040	1.0000	1.03273	0.000
0.2086	0.75757	0.230	0.5290	0.83606	0.141						
					n-Undecane (1) + 2-Cresol (2)						
0.0000	0.72913	0.000	0.2094	0.76270	0.301	0.5311	0.83593	0.248	0.8065	0.93192	0.110
0.0367	0.73421	0.134	0.2809	0.77641	0.321	0.5989	0.85595	0.210	0.8662	0.95918	0.075
0.0684	0.73892	0.200	0.3636	0.79401	0.298	0.6593	0.87561	0.170	0.9476	1.00143	0.046
0.0746	0.73992	0.199	0.4749	0.82079	0.269	0.7261	0.89941	0.151	1.0000	1.03273	0.000
0.1306	0.74880	0.290									
					n-Nonane (1) + 3-Cresol (2)						
0.0000	0.70741	0.000	0.3020	0.77094	−0.022	0.5000	0.82500	−0.287	0.9000	0.97443	−0.318
0.1021	0.72654	0.107	0.4021	0.79676	−0.128	0.7000	0.89222	−0.491	1.0000	1.02202	0.000
0.1998	0.74724	0.041									
					n-Undecane (1) + 3-Cresol (2)						
0.0000	0.72913	0.000	0.3395	0.78784	0.126	0.6023	0.85421	0.036	0.9000	0.96861	−0.049
0.1287	0.74852	0.137	0.3890	0.79863	0.114	0.6949	0.88419	−0.009	1.0000	1.02202	0.000
0.1853	0.75811	0.149	0.5024	0.82611	0.084	0.8000	0.92373	−0.026			
					n-Dodecane (1) + 3-Cresol (2)						
0.0000	0.73863	0.000	0.3009	0.78451	0.210	0.6976	0.88371	0.104	0.8950	0.96390	0.051
0.1325	0.75667	0.147	0.4009	0.80435	0.191	0.7987	0.92129	0.036	1.0000	1.02202	0.000
0.2055	0.76797	0.193									

Table 6. Redlich−Kister Coefficients and Standard Deviations

system	A_1/cm^3 mol^{-1}	A_2/cm^3 mol^{-1}	A_3/cm^3 mol^{-1}	A_4/cm^3 mol^{-1}	A_5/cm^3 mol^{-1}	s^a/cm^3 mol^{-1}
2-cresol + *n*-nonane	0.1044	0.8063	0.1926	1.1485	1.1983	0.0035
2-cresol + *n*-decane	0.6257	0.7365	0.1442	0.9099	1.2102	0.0033
2-cresol + *n*-undecane	1.0322	0.8029	0.3077	0.7106	1.2944	0.0073
3-cresol + *n*-nonane	−1.0857	2.7709	−1.2857	0.1437	1.6950	0.0125
3-cresol + *n*-undecane	0.3114	0.7300	0.0243	0.6597		0.0080
3-cresol + *n*-dodecane	0.6516	0.7658	0.3402			0.0160

[a] $s = (1/(n_p - 1)\Sigma(V^E_{calcd} - V^E_{exptl})^2)^{1/2}$, where n_p is the number of data points.

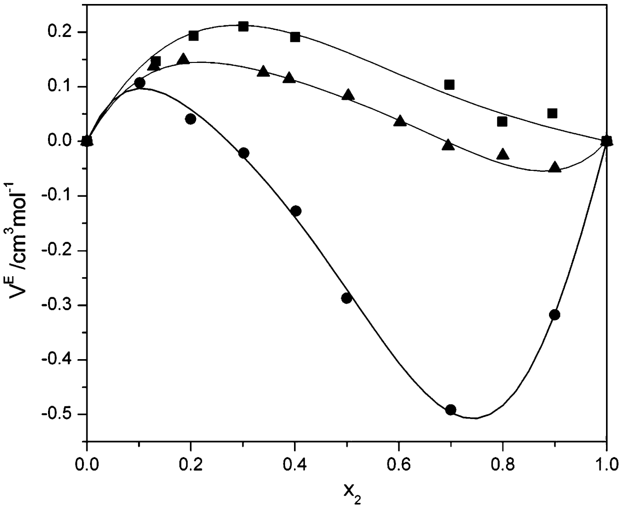

Figure 4. Excess molar volumes V^E for 3-cresol (2) + alkanes (1) at 308.15 K. Experimental results: ●, *n*-nonane; ▲, *n*-undecane; ■, *n*-dodecane. Calculated values from the Redlich−Kister equation (eq 6).

Treszczanowicz and Benson[10] in mixtures of *n*-hexan-1-ol + *n*-alkanes. There, the mixture with *n*-decane shows also only positive V^E values that decrease with decreasing chain length of the alkanes. Furthermore, the V^E curves become S-shaped. Treszczanowicz[10] explained this behavior by assuming that V^E is the resultant of opposing effects; this can be adapted to the mixtures of this work. Changes in free volume lead to decreasing volume at large concentrations of cresols as a result of the 3D structures that are formed by highly directed hydrogen bonds. Conversely, between alkanes and cresols there exist only slight interactions; therefore, the volume increases when a cresol is added to a solution of alkanes. The other systems (3-cresol + *n*-dodecane, 2-cresol + *n*-decane, and 2-cresol + *n*-undecane) have positive V^E curves over the entire range of composition.

A successful fit to the V^E curves was obtained from the Redlich−Kister equation. The deviations between the V^E values calculated from experimental densities and the fitted values using the Redlich−Kister equation are of the same order of magnitude as the inaccuracy caused by the experimental uncertainties.

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