Excess Volumes and Excess Heat Capacities of Some Mixtures with trans,trans,cis-1,5,9-Cyclododecatriene at 298.15 K[†]

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Excess molar volumes V_m^E and excess molar heat capacities C_{ρ}^{E} at constant pressure have been obtained, as a function of mole fraction x, at 298.15 K and atmospheric pressure for the binary liquid mixtures trans, trans, cis-1,5,9-cyclododecatriene (CDT, c-C₁₂H₁₈) + n-heptane, + n-dodecane, + n-hexadecane, + c yclohexane, + benzene, and + tetrachloromethane. The instruments used were a vibrating-tube densimeter and a Picker flow microcalorimeter, respectively. In the alkane series, excess volume is negative for CDT + *n*-heptane $(V_m^E(x=0.5) = -0.599 \text{ cm}^3 \cdot \text{mol}^{-1})$ but becomes positive for the mixtures with n-dodecane $(V_m^{E}(x=0.5) = 0.072 \text{ cm}^3 \cdot \text{mol}^{-1})$ and *n*-hexadecane $(V_m^E(x=0.5) = 0.322 \text{ cm}^3 \cdot \text{mol}^{-1})$. The $C_p^E_m$'s are all negative and decrease with increasing chain length of the *n*-alkane. $V_m^E/(\text{cm}^3 \cdot \text{mol}^{-1}) = 0.093$ for $\{\frac{1}{2}c - C_{12}H_{18} + \frac{1}{2}c - \frac{1}{2}H_{18} + \frac{1}{2}C + \frac$ $1/{_2C_6H_6}$ and -0.309 for $\{1/{_2C}-C_{12}H_{18} + 1/{_2CCI_4}\}$. Most surprisingly, V_m^E of $\{(x)c-C_{12}H_{18} + (1-x)c-C_{4}H_{12}\}$ exhibits two very shallow minima near the ends of the composition range, while $V_m^E_{mex} = 0.030 \text{ cm}^3 \cdot \text{mol}^{-1}$ at $x_{\text{max}} = 0.410$. The C_{ρ}^{E} is are negative for the mixtures of CDT with cyclohexane and with benzene and positive for the mixture with tetrachloromethane.

Introduction

Thermodynamic properties of binary liquid mixtures containing an *n*-alkane as one component, i.e., Y + an n-alkane, are currently attracting considerable interest. The focus is on the systematic study of the influence of chemical structure of the component Y as well as on the role of chain length of the n-alkane upon excess quantities (1-5). Recent experimental work on such mixtures has revealed surprisingly different and sometimes quite unexpected mixing behavior. For instance, order destruction or order creation has been indicated, depending on whether Y is a more or less globular molecule or a platelike molecule. For mixtures of n-alkanes with aliphatic or cyclic ethers (6, 7), α, ω -dichloroalkanes (8-10), or alkanones (11), $C_{\rho}^{E}(x)$ curves with two minima and one maximum (W-shaped curves) have been observed and discussed in terms of nonrandomness caused by strong orientational effects due to polar-polar interaction (12-16).

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There have been relatively few studies of hydrocarbon mixtures that contain an unsaturated hydrocarbon (17-20), and experimental work on mixtures of cycloalkanes with *n*-alkanes is also quite limited (21-23). The presence of three nonconjugated double bonds in the *trans*, *trans*, *cls*-1,5,9-cyclododecatriene (CDT, $c-C_{12}H_{18}$) molecule makes it a large cyclic compound with significantly restricted flexibility. We also note that CDT is a chemical of major technical importance in that it serves as the starting point for the synthesis of a variety of polymers (24). In this work we report excess molar volumes V_m^E and excess molar heat capacities $C_p^E_m$ at constant pressure, both at 298.15 K and atmospheric pressure, for binary mixtures of CDT with *n*-heptane, *n*-dodecane, and *n*-hexadecane, as well as with cyclohexane, benzene, and tetrachloromethane.

Experimental Section

All liquids were of the best quality available from Fluka: *n*-heptane, benzene, and tetrachloromethane were "puriss" with stated purities \geq 99.5 mol %, *n*-dodecane, *n*-hexadecane, and *trans*,*trans*,*cis*-1,5,9-cyclododecatriene were "purum" with purity >99 mol %. The liquids were carefully dried with a molecular sieve (Union Carbide Type 4A, beads, from Fluka) and used without further purification. Binary mixtures were prepared by mass with a possible error in the mole fraction estimated to be less than 10⁻⁴. All molar quantities are based on the relative atomic mass table of IUPAC, 1986 (*25*).

Densities ρ of the pure liquids and their mixtures were determined with a vibrating-tube densimeter from Sodev (Model 02D), which was operated under flow conditions (*26*). Before each series of measurements the instrument was calibrated with vacuum and doubly distilled and degassed water using density $\rho_{\rm H_20}$ (298.15 K) = 997.047 kg·m⁻³ as reported by Kell (*27*). Excess molar volumes $V_{\rm m}^{\rm E}$ were obtained from the density ρ of the mixture according to

$$V_{\rm m}^{\rm E} = V_{\rm m} - \{xV_1^{*} + (1-x)V_2^{*}\}$$
$$xM_1\{(1/\rho) - (1/\rho_1^{*})\} + (1-x)M_2\{(1/\rho) - (1/\rho_2^{*})\} \quad (1)$$

where V_i^* , M_i , and ρ_i^* denote, respectively, the molar volume, the molar mass, and the density of pure CDT (i = 1) or the other component (i = 2), and V_m is the molar volume of the mixture.

Heat capacities per unit volume, C_p/V , were measured with a Picker flow microcalorimeter from Setaram, using the stepwise procedure (26, 28, 29). For all measurements we used

Table I. Density, ρ^* , and Molar Heat Capacity, $C_{\rho}^*{}_m$, of the Pure Liquids at 298.15 K

	$\rho^*/(\text{kg}\cdot\text{m}^{-3})$		$C_p *_m / (J \cdot K^{-1} \cdot mol^{-1})$		
	this work	lit.	this work	lit.	
$n - C_7 H_{16}$	679.51	679.49ª	224	.78 ^d	
$n - C_{12}H_{28}$	746.31	745.14ª	372.12	374.82,° 374.93 [/]	
$n - C_{16}H_{34}$	770.07	769.96 ^ø	497.16	496.45, ^g 499.97 ^e	
$c-C_{6}H_{12}$	773.53	773.89°	156.90	156.75, ^h 156.15 ⁱ	
C ₆ H ₆	873.42	873.68°	135.62	135.60, ^j 135.71*	
CCl ₄	1584.58	1584.39°	133.35	131.82, ^h 131.36 ^l	
$c - C_{12}H_{18}$	889.19		287.76		

^aReference 30. ^bReference 31. ^cReference 32. ^dThis value from ref 33—was adopted for the *n*-heptane sample used in this work; it served as reference liquid in all our heat capacity measurements. ^eReference 34. ^fReference 35. ^gReference 8. ^hReference 36. ⁱReference 37. ^jReference 29. ^kReference 38. ^lReference 33.



Figure 1. Excess molar volumes V_m^E of $\{(x)c-C_{12}H_{18} + (1 - x)n-C_lH_{2l+2}\}$ at 298.15 K. O, \Box , Δ , experimental points; —, calculated from eq 3 with coefficients from Table III: O, l = 7; \Box , l = 12; Δ , l = 16.

a temperature increment of approximately 1 K centered on 298.15 K. Excess molar heat capacities were calculated from the molar heat capacities $C_{p,m}$ of the mixture and C_p^* , of the pure components from

$$C_{p}^{E}_{m} = C_{p,m} - \{xC_{p}^{*}_{1} + (1-x)C_{p}^{*}_{2}\}$$
(2)

For both densimeter and microcalorimeter, temperature control was better than ± 0.002 K as checked by a quartz thermometer (Hewlett-Packard, Model 2801 A). The imprecision of the values of the excess molar volumes and the excess molar heat capacities is estimated to be less than ± 0.005 cm⁻³-mol⁻¹ and ± 0.05 J·K⁻¹·mol⁻¹, respectively.

Results and Discussion

Experimental results for density and molar heat capacity at constant pressure of the pure liquids are summarized in Table



Figure 2. Excess molar volumes V_m^{E} for O, $\{(x)c-C_{12}H_{18} + (1 - x)c-C_{6}H_{12}\}; \Box$, $\{(x)c-C_{12}H_{18} + (1 - x)C_{6}H_{6}\}; \Delta$, $\{(x)c-C_{12}H_{18} + (1 - x)C_{12}H_{18} + (1 - x)C_{14}\}$. The curves were calculated from eq 3 with coefficients from Table III.



Figure 3. Excess molar heat capacities $C_p \stackrel{\text{E}}{}_m$ at constant pressure of $\{(x)c-C_{12}H_{18} + (1-x)n-C_1H_{2/+2}\}$ at 298.15 K. O, \Box , Δ , experimental points; ----, calculated from eq 3 with coefficients from Table III: O, l = 7; \Box , l = 12; Δ , l = 16.

I along with selected values from the literature. In general, agreement with the literature data is satisfactory. We note, however, the somewhat higher density of n-dodecane and the somewhat larger molar heat capacity of tetrachloromethane, indicative for slightly impure substances. No published values

Table II. Excess Molar Volumes, V_m^E , and Excess Molar Heat Capacities, $C_p^{E_m}$, for Some Binary Mixtures Containing *trans*, *trans*, *cis*-1,5,9-Cyclododecatriene ($c \cdot C_{12}H_{18}$) at 298.15 K

	$V_{\rm m}^{\rm E}/$	$C_{p}^{E}m/$		$V_{\rm m}^{\rm E}/$	$C_{p}^{\mathbf{E}}\mathbf{m}/$				
x	cm³•mol ^{−1}	J•K ⁻¹ •mol ⁻¹	x	cm³∙mol ⁻¹	J·K ⁻¹ ·mol ⁻¹				
$\frac{(x)c-C_{12}H_{18} + (1-x)n-C_{7}H_{16}}{(x)c-C_{12}H_{18} + (1-x)n-C_{7}H_{16}}$									
0.0781	-0.220	-0.449	0.6119	-0.540	-1.037				
0.1521	-0.375	-0.677	0.7013	-0.462	-0.887				
0.2227	-0.479	-0.878	0.7714	-0.373	-0.715				
0.2923	-0.557	-1.032	0.8472	-0.268	-0.521				
0.3951	-0.604	-1.144	0.9339	-0.125	-0.235				
0.4945	-0.599	-1.152							
$(x)c-C_{12}H_{18} + (1-x)n-C_{12}H_{28}$									
0.0836	0.016	-0.691	0.5972	0.073	-2.023				
0.1536	0.039	-1.130	0.6978	0.067	-1.743				
0.2286	0.047	-1.540	0.7720	0.059	-1.362				
0.3016	0.057	-1.818	0.8451	0.047	-1.014				
0.4022	0.068	-2.044	0.9348	0.028					
0.5025	0.074	-2.108							
	$(\mathbf{r})c$ -CueHue + $(1 - \mathbf{r})n$ -CueHau								
0.0893	0.098	-1.579	0.5967	0.322	-4.478				
0.1519	0.152	-2.465	0.6935	0.300	-3.943				
0.2273	0.216	-3.323	0.7700	0.255	~3.332				
0.2994	0.260	-3.987	0.8488	0.194	-2.461				
0.3942	0.298	-4.503	0.9134	0.132	-1.540				
		$(x)c-C_{12}H_{12} +$	(1 - x)c-0	CeH19					
0.1052	0.001	-0.307	0.6035	0.020	-1.009				
0.1958	0.013		0.7040	0.011	-0.825				
0.3039	0.028	-0.911	0.7912	0.002	-0.624				
0.4071	0.030	-1.070	0.8980	0.000	-0.322				
0.5026	0.024								
		(x)c-C ₁₀ H ₁₀ -	+(1-x)C	H.					
0.0720	0.051	-0.586	0.5999	0.074	-1.051				
0.1223	0.077	-0.872	0.6938	0.051	-0.834				
0.2180	0.103	-1.236	0.7698	0.034	-0.642				
0.2887	0.113	-1.358	0.8408	0.015	-0.455				
0.4000	0.104	-1.384	0.9106	0.005	-0.251				
0.4983	0.092	-1.265							
$(x)c-C_{1-1}H_{1-1} + (1 - x)CCL$									
0.0733	-0.134	0.482	0.5957	-0.292	1.247				
0.1513	-0.204	0.886	0.6967	-0.258	0.996				
0.2216	-0.257	1.105	0.7653	-0.217	0.797				
0.2988	-0.273	1.315	0.8407	-0.167	0.496				
0.3959	-0.310	1.367	0.9065	-0.107	0.285				
0.4982	-0.306	1.366							

for C_p^* or ρ^* of *trans*, *trans*, *cis*-1,5,9-cyclododecatriene could be found for comparison.

The experimental results for the excess molar quantities V_m^E and $C_p^E_m$ are given in Table II, and a graphical representation is provided by Figures 1–4. For each mixture, the excess quantities were smoothed by unweighted least-squares polynomial regression according to

$$Q^{\mathsf{E}} = x(1-x)\sum_{i=0}^{k} A_{i}(2x-1)^{i}$$
(3)



Figure 4. Excess molar heat capacities $C_p^{E}_{m}$ at constant pressure at 298.15 K for O, $\{(x)c-C_{12}H_{16} + (1-x)c-C_{6}H_{12}\}$; \Box , $\{(x)c-C_{12}H_{16} + (1-x)C_{6}H_{6}\}$; Δ , $\{(x)c-C_{12}H_{16} + (1-x)Cc_{14}\}$. The curves were calculated from eq 3 with coefficients from Table III.

where either $Q^{E} = V_{m}^{E}/(cm^{3} \cdot mol^{-1})$ or $Q^{E} = C_{p}^{E}m/(J \cdot K^{-1} \cdot mol^{-1})$ and x is the mole fraction of CDT. The coefficients A_{i} and the corresponding standard deviations $s(Q^{E})$ are shown in Table III. These coefficients were used to obtain the calculated curves in the figures. No literature results could be found for comparison.

For the series CDT + an *n*-alkane the dependence of V_m^E on the chain length of the *n*-alkane $n-C_lH_{2l+2}$ is as expected, i.e., V_m^E increases with increasing *l*. While little can be said concerning V_m^E of CDT + benzene and CDT + tetrachloromethane, the composition dependence of the excess molar volume of CDT + cyclohexane is most surprising: $V_m^E(x)$ exhibits *two* shallow *minima* situated near the ends of the composition range; that is, $x'_{min} = 0.043$ and $x''_{min} = 0.917$, while $V_m^E_{max} = 0.030 \text{ cm}^3 \cdot \text{mol}^{-1}$ is observed at $x_{max} = 0.410$. Such a behavior has never been reported before (*39*, *40*). Note that $V_m^E(x)$ with two maxima is also exceedingly rare, the only known example being tetrachloromethane + benzene between 295.4 and 296.2 K (*41*, *42*).

As concerns the excess molar heat capacities of CDT + an n-alkane, the dependence of $C_p^{E}{}_m$ on *i* is qualitatively similar to that observed for mixtures of cyclohexane with n-alkanes and may thus be discussed in terms of destruction of liquid order

Table III. Coefficients A_i and Standard Deviations $s(Q_m^E)$ for Least-Squares Representation by Eq 3 of $V_m^E/(\text{cm}^3 \cdot \text{mol}^{-1})$ and $C_{\rho \ m}^E/(J \cdot \text{K}^{-1} \cdot \text{mol}^{-1})$ at 298.15 K

	$Q^{\rm E}$	A ₀	A_1	A ₂	A_3	A_4	$s(Q_m^E)$	
$(x)c-C_{12}H_{18} + (1-x)n-C_7H_{16}$	V _m E	-2.3946	0.601	-0.201			0.0024	
	$C_{p}^{\mathbf{E}}$	-4.676	1.120				0.026	
$(x)c-C_{12}H_{18} + (1-x)n-C_{12}H_{28}$	V_m^E	0.2887	0.092	0.061			0.0036	
	C_{pm}^{TE}	-8.357	0.726				0.027	
$(x)c-C_{12}H_{18} + (1-x)n-C_{16}H_{34}$	V_m^{E}	1.2894	0.246	0.182			0.0039	
		-18.627	0.065	-1.125			0.026	
$(x)c-C_{12}H_{18} + (1-x)c-C_{6}H_{12}$	V_m^{E}	0.1094	-0.125	-0.167	0.187		0.0019	
	$C_{p}^{\mathbf{E}}$	-4.394	0.570	1.600	-1.184		0.003	
$(x)c-C_{12}H_{18} + (1-x)C_{6}H_{6}$	V _m E	0.3726	-0.350	0.049	-0.109		0.0017	
	$C_{p}^{\mathbf{E}}$ m	-5.030	3.285	-1.185			0.006	
$(x)c-C_{12}H_{18} + (1-x)CCl_4$	V _m E	-1.2369	0.075	-0.135	0.434	-0.548	0.0055	
· •- ·- ·	C.E.	5.365	-2.137				0.030	

(5). Of the remaining systems, the highly skewed $C_p^{E}(x)$ of CDT + benzene is to be noted.

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Registry No. CDT, 706-31-0; n-C7H18, 142-82-5; n-C12H28, 112-40-3; $n \cdot {\rm C_{16}H_{34}}, \ 544-76-3; \ c \cdot {\rm C_6H_{12}}, \ 110-82-7; \ {\rm C_6H_6}, \ 71-43-2; \ {\rm CCl_4}, \ 56-23-5.$

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Viscosity and Density of Ternary Mixtures for Toluene, Ethylbenzene, Bromobenzene, and 1-Hexanol

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Mixture viscosities and densities of the ternary mixtures of ethylbenzene, toluene, bromobenzene, and 1-hexanol were measured at 30, 40, 50, and 60 °C. The nonidealities reflected in mixture viscosities were expressed and discussed in terms of excess viscosities which were both positive and negative.

Introduction

In a continuation of our earlier work (1-9) on viscosities and dielectric constants of liquid mixtures, the present paper reports the viscosities and the densities for the ternary mixtures of toluene, ethylbenzene, bromobenzene, and 1-hexanol at 30, 40, 50, and 60 °C.

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Experimental Section

Materials. Toluene (BDH), ethylbenzene (E. Merck), and 1-hexanol (BDH) were purified by fractional distillation and drying, whereas bromobenzene (E. Merck) was purified by distillation and then fractional distillation, collecting the portion at 156 ± 0.5 °C before use. The mean of several repeat density, viscosity, and refractive index measurements compared favorably with the corresponding literature values within allowable limits (Table V). Redistilled, deionized, and degassed water that showed electrical conductivity of less than 7.0 \times 10^{-7} mhos cm⁻¹ was used for checking the instruments and calibrating the pycnometers for density measurements.

Experimental Measurements. Ternary mixtures were prepared by weight with an accuracy of 0.0001 g. Viscosities were measured with Ostwald viscometers to ascertain that the viscometer limbs coincided with the vertical within 0.5° and that the standard deviation for the time flow in each case did not exceed 0.1%. The densities were measured pycnometrically by using water with 0.997 07 g mL⁻¹ as its density at 25 °C for calibration. The experimental procedures adopted for viscosity

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