Excess Molar Enthalpies of *p*-Cymene + α-Pinene + *β*-Pinene at (298.15, 308.15, and 318.15) K and at Atmospheric Pressure

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Excess molar enthalpies ($H^E_{m,123}$), measured at (298.15, 308.15, and 318.15) K in a Calvet low-temperature microcalorimeter, are reported for the ternary mixture of *p*-cymene (1) + α-pinene (2) + *β*-pinene (3) at atmospheric pressure. The results serve to show that all the experimental $H^E_{m,123}$ values are positive and that they increase with an increase in temperature. In all cases, maximum values were observed in the vicinity of $x_1 \approx 0.5$. Furthermore, at constant x_1, the measured $H^E_{m,123}$ values increase as the ratio of x_2/x_3 increases.

Introduction

Turpentine oil, obtained by steam distillation of pine resins, is one of the richest resources of refined oil in the world. Its main constituents include α-pinene, *β*-pinene, and *p*-cymene.[1] Turpentine oil is widely used in the synthesis of a variety of fine chemicals such as synthetic resins and terpenic surfactants and in the pharmaceutical and cosmetic industries.[2,3] Furthermore, α-pinene proved to be exceptionally successful in the asymmetric reduction for synthesizing optical pure materials via chiral organoboranes.[4]

The usefulness of relevant thermodynamic data with the consideration of separating these components is evident. Vapor−liquid equilibrium (VLE) and excess molar enthalpies are primary thermodynamic properties for indicating the nonideal behavior of the mixtures. These basic mixture properties are useful for further processing of turpentine.[5] Francesconi et al.[6,7] and Berlin and Comelli[8] reported the measurement of physical properties, excess molar enthalpies using an LKB flow microcalorimeter. VLE values for some binary mixtures containing pine resins have been reported.[9−11] Recently, Liao et al.[12] presented excess enthalpies for binary systems of α-pinene + *p*-cymene and *β*-pinene + *p*-cymene using a low-temperature Calvet microcalorimeter. The purpose of this work is to provide excess molar enthalpies for ternary mixtures of *p*-cymene (1) + α-pinene (2) + *β*-pinene (3) at three temperatures [(298.15, 308.15, and 318.15) K].

Experimental Section

The chemicals α-pinene and *β*-pinene, both mole fraction minimum purities $x = 99$ %, were obtained from Fluka, and *p*-cymene was obtained from Aldrich Chemical Co. All chemicals were dried with zeolite type 5A. Subsequently, the purities of the chemicals were determined by means of a gas chromatograph (Agilent 6890), using a DB-5 capillary column.

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Table 1. Densities and Refractive Indices of Experiment Chemical Reagents at (298.15, 308.15, and 318.15) K

	T	ρ/g·cm^{-3}			n_D		
component	K	this work	lit.[13]	lit.[14]	this work	lit.[13]	lit.[14]
α-pinene	298.15	0.85403	0.8539	0.8548	1.4626	1.4632	1.4621
	308.15	0.84579		0.8469	1.4581		1.4579
	318.15	0.83864		0.8394	1.4542		1.4540
β-pinene	298.15	0.86706	0.8667	0.8655	1.4760	1.4768	1.4748
	308.15	0.85908		0.8580	1.4714		1.4705
	318.15	0.85208		0.8505	1.4665		1.4660
p-cymene	298.15	0.85347	0.8533	0.8521	1.4878	1.4885	1.4865
	308.15	0.84559		0.8445	1.4831		1.4824
	318.15	0.83846		0.8374	1.4789		1.4782

Table 2. Excess Enthalpies for the Binary System α-Pinene (2) + *β*-Pinene (3)

T/K = 298.15		T/K = 308.15		T/K = 318.15	
x_2	$H^E_{m,23}$/J·mol^{-1}	x_2	$H^E_{m,23}$/J·mol^{-1}	x_2	$H^E_{m,23}$/J·mol^{-1}
0.2012	7.0	0.2000	7.5	0.2003	8.8
0.3013	9.2	0.3004	10.1	0.3001	11.2
0.3987	10.6	0.3999	11.4	0.4034	12.7
0.4993	11.1	0.5006	11.9	0.5000	13.2
0.5994	10.3	0.6013	10.9	0.6004	12.6
0.7023	9.5	0.6972	9.9	0.7012	11.4
0.7998	7.3	0.7998	7.4	0.7975	8.4

The mole fraction purities were found to be (99.77, 99.58, and 99.17) % for α-pinene, *β*-pinene, and *p*-cymene, respectively.

The densities (ρ) of the chemicals were determined using a densimeter (Sartorius, model YDK01). The refractive indices (n_D) were measured using an Abbe refractometer (Shanghai Scientific Instruments, model WZS-I830688). A comparison of the measured ρ and n_D of the chemicals at the three temperatures with those available in the literature[13,14] is presented in Table 1. The good agreement shown in the table further verified the purities of the chemicals used in this work.

The treated pure liquids were kept in dark bottles. α-Pinene was placed in a freezer at 4 °C as recommended by the supplier.

Excess molar enthalpies (H^E_m) were determined in a low-temperature Calvet microcalorimeter (model BT2.15, SETARAM Co., France). Details of this equipment and its

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Table 3. Coefficients h_k and Standard Deviations s for the Representation of $H^E_{m,ij}$ ($i < j$) by Equation 2 for the Three Binary Mixtures at Three Temperatures

T/K	h_1	h_2	h_3	h_4	s/J·mol^{-1}
		p-Cymene (1) + α-Pinene (2)[12]			
298.15	769.36	−5.53	−127.14	−24.52	1.33
308.15	853.75	4.31	−53.05	−24.11	1.89
318.15	888.40	29.62	−77.76	−79.91	2.13
		p-Cymene (1) + *β*-Pinene (3)[12]			
298.15	417.60	−0.50	−53.86	13.98	0.77
308.15	440.55	1.09	−65.44	7.63	0.76
318.15	449.71	−6.08	−53.14	−2.55	1.42
		α-Pinene (2) + *β*-Pinene (3)			
298.15	43.94	−0.40	2.36	6.60	0.20
308.15	47.11	−4.17	−1.54	10.91	0.17
318.15	52.88	1.41	2.73	−8.49	0.20

performance have been described previously.[12] The temperature of the microcalorimeter was kept constant to ± 0.005 K.

In studying the ternary system, the excess molar enthalpy of $H^E_{m,1+23}$ was determined for several pseudo-binary mixtures in which component 1 (*p*-cymene) was added to several binary mixtures of components 2 (α-pinene) + 3 (*β*-pinene). For this purpose, binaries with fixed mole ratios x_2/x_3 were prepared by mass. The excess molar enthalpy of the ternary system was then obtained from the relation[15]

$$H^E_{m,123} = H^E_{m,1+23} + (1 - x_1)H^E_{m,23} \quad (1)$$

where $H^E_{m,23}$ is the excess molar enthalpy of the particular binary mixture of α-pinene + *β*-pinene. Over most of the mole fraction range of component 1, the uncertainties of the excess enthalpies for ternary system, the mole fraction of the mixtures, and the temperature are estimated to be ± 0.006 $H^E_{m,1+23}$, 5 × 10^{-4}, and ± 0.005 K, respectively.

Results and Discussion

Excess molar enthalpies $H^E_{m,ij}$ ($i < j$), measured at T = (298.15, 308.15, and 318.15) K for two of the constituent binary systems of present interest, *p*-cymene (1) + α-pinene (2) and *p*-cymene (1) + *β*-pinene (3) have been reported previously.[12] The experimental results for the excess enthalpies of α-pinene (2) + *β*-pinene (3) at the three temperatures are listed in Table 2. The smoothing function

$$H^E_{m,ij}/\text{J·mol}^{-1} = x_i(1 - x_i)\sum_{k=1}^{n} h_k(1 - 2x_i)^{k-1} \quad (2)$$

where $i < j$ was fitted to the results by a least-square method, with all points weighted equally. Values of the coefficients h_k are listed in Table 3 along with the standard deviation s for the

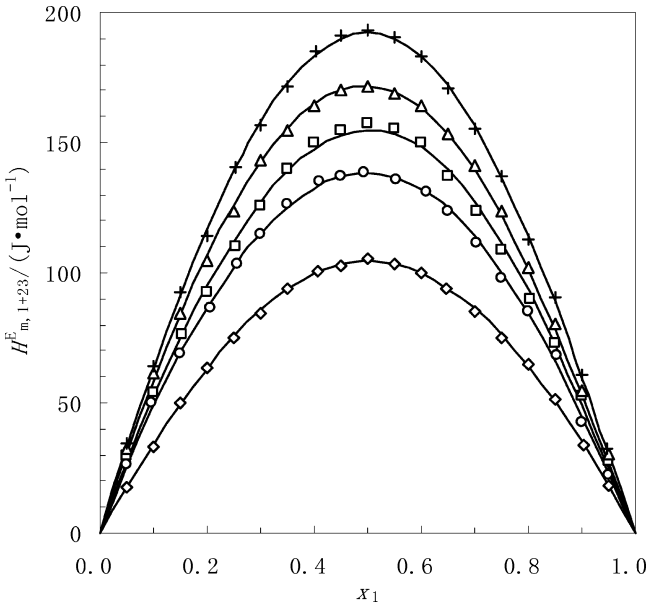

Figure 1. Excess molar enthalpies, $H^E_{m,1+23}$, for the ternary system *p*-cymene (1) + α-pinene (2) + *β*-pinene (3) at 298.15 K. Experimental results: ◇, $x_2 = 0$ from ref 12; ○, $x_2/x_3 = 0.5028$; □, $x_2/x_3 = 1.0000$; Δ, $x_2/x_3 = 1.9876$; +, $x_3 = 0$ from ref 12. Solid curves were calculated from eq 3.

Table 4. Experimental Excess Molar Enthalpies for the Ternary System *p*-Cymene (1) + α-Pinene (2) + *β*-Pinene (3) at 298.15 K and Values of $H^E_{m,123}$ from Equation 1

x_1	$H^E_{m,1+23}$/J·mol^{-1}	$H^E_{m,123}$/J·mol^{-1}	x_1	$H^E_{m,1+23}$/J·mol^{-1}	$H^E_{m,123}$/J·mol^{-1}	x_1	$H^E_{m,1+23}$/J·mol^{-1}	$H^E_{m,123}$/J·mol^{-1}
			$x_2/x_3 = 0.5028$, $H^E_{m,23}$/J·mol^{-1} = 9.6					
0.0500	26.2	35.4	0.4093	135.0	140.6	0.7516	98.0	100.4
0.0983	49.8	58.4	0.4506	137.2	142.5	0.7995	85.5	87.4
0.1506	69.2	77.4	0.4939	138.7	143.5	0.8519	68.4	69.8
0.2052	86.6	94.3	0.5543	136.0	140.3	0.9000	42.8	43.8
0.2573	103.6	110.5	0.6089	131.1	134.9	0.9499	22.6	23.1
0.2997	115.0	121.8	0.6490	123.5	126.9			
0.3500	126.0	132.3	0.7024	111.3	114.2			
			$x_2/x_3 = 1.0000$, $H^E_{m,23}$/J·mol^{-1} = 11.2					
0.0501	30.0	40.7	0.3999	149.9	156.7	0.7512	108.9	111.7
0.1016	54.1	64.2	0.4490	154.8	161.0	0.8017	90.0	92.2
0.1529	76.5	86.0	0.5004	157.2	162.8	0.8499	72.9	74.6
0.2003	92.6	101.5	0.5489	155.4	160.5	0.8995	52.6	53.7
0.2520	110.1	118.5	0.5994	150.2	154.7	0.9500	27.5	28.1
0.3007	125.7	133.6	0.6500	137.2	141.1			
0.3500	140.1	147.4	0.7026	124.0	127.3			
			$x_2/x_3 = 1.9876$, $H^E_{m,23}$/J·mol^{-1} = 9.8					
0.0500	32.6	41.9	0.4003	164.5	170.4	0.7500	123.4	125.8
0.0996	61.5	70.4	0.4501	170.1	175.5	0.8006	101.9	103.9
0.1500	84.2	92.5	0.5000	171.4	176.3	0.8504	80.1	81.6
0.2000	104.76	112.5	0.5498	168.6	173.0	0.9000	54.6	55.5
0.2491	123.6	131.0	0.5993	164.0	168.0	0.9499	30.4	30.9
0.3005	143.4	150.2	0.6505	153.1	156.6			
0.3500	155.0	161.4	0.6993	141.5	144.5			

Table 5. Experimental Excess Molar Enthalpies for the Ternary System *p*-Cymene (1) + α-Pinene (2) + *β*-Pinene (3) at 308.15 K and Values of $H^E_{m,123}$ from Equation 1

x_1	$H^E_{m,1+23}$ / J·mol^{-1}	$H^E_{m,123}$ / J·mol^{-1}	x_1	$H^E_{m,1+23}$ / J·mol^{-1}	$H^E_{m,123}$ / J·mol^{-1}	x_1	$H^E_{m,1+23}$ / J·mol^{-1}	$H^E_{m,123}$ / J·mol^{-1}
			$x_2/x_3 = 0.5028$, $H^E_{m,23}$/J·mol^{-1} = 10.6					
0.0503	29.2	39.3	0.4001	140.7	147.1	0.7499	104.6	107.3
0.1011	52.8	62.3	0.4503	143.9	149.8	0.7996	91.2	93.3
0.1500	75.2	84.2	0.5007	145.9	151.2	0.8501	72.7	74.3
0.2001	94.9	103.4	0.5500	143.3	148.1	0.9006	48.9	49.9
0.2503	110.0	118.0	0.5995	138.8	143.1	0.9499	27.9	28.5
0.3001	123.1	130.5	0.6500	130.1	133.8			
0.3500	133.2	140.1	0.6999	121.4	124.5			
			$x_2/x_3 = 1.0000$, $H^E_{m,23}$/J·mol^{-1} = 12.0					
0.0505	32.6	44.0	0.4001	161.2	168.5	0.7496	116.2	119.2
0.1008	64.2	75.0	0.4500	164.6	171.2	0.7994	98.9	101.3
0.1502	89.6	99.8	0.5002	166.9	172.9	0.8500	77.4	79.2
0.2002	106.1	115.7	0.5497	163.9	169.4	0.9002	57.2	58.4
0.2504	124.2	133.2	0.5987	157.2	162.1	0.9495	29.1	29.8
0.2997	137.3	145.7	0.6500	146.9	151.0			
0.3500	153.2	161.1	0.6995	134.6	138.2			
			$x_2/x_3 = 1.9876$, $H^E_{m,23}$/J·mol^{-1} = 10.4					
0.0502	35.9	45.8	0.3998	176.4	182.7	0.7499	135.2	137.8
0.1004	69.5	78.9	0.4504	181.7	187.4	0.7994	114.9	117.0
0.1500	95.2	104.1	0.5003	182.7	187.9	0.8500	86.6	88.2
0.2005	118.5	126.9	0.5500	180.3	185.0	0.9000	60.7	61.7
0.2504	137.5	145.3	0.5999	174.1	178.3	0.9495	33.0	33.5
0.2999	156.4	163.7	0.6500	163.1	166.8			
0.3500	167.5	174.3	0.7001	153.7	156.9			

Table 6. Experimental Excess Molar Enthalpies for the Ternary System *p*-Cymene (1) + α-Pinene (2) + *β*-Pinene (3) at 318.15 K and Values of $H^E_{m,123}$ from Equation 1

x_1	$H^E_{m,1+23}$ / J·mol^{-1}	$H^E_{m,123}$ / J·mol^{-1}	x_1	$H^E_{m,1+23}$ / J·mol^{-1}	$H^E_{m,123}$ / J·mol^{-1}	x_1	$H^E_{m,1+23}$ / J·mol^{-1}	$H^E_{m,123}$ / J·mol^{-1}
			$x_2/x_3 = 0.5028$, $H^E_{m,23}$/J·mol^{-1} = 11.7					
0.0500	29.9	41.0	0.3998	148.1	155.1	0.7495	109.0	111.9
0.1000	56.3	66.9	0.4503	150.0	156.5	0.8000	92.2	94.5
0.1500	77.2	87.2	0.4999	151.1	157.0	0.8500	71.9	73.6
0.2000	96.5	105.8	0.5498	149.6	154.9	0.9005	50.4	51.6
0.2498	112.1	120.8	0.5996	143.7	148.4	0.9500	27.4	28.0
0.2997	127.3	135.5	0.6500	135.2	139.3			
0.3500	138.0	145.6	0.6996	124.1	127.6			
			$x_2/x_3 = 1.0000$, $H^E_{m,23}$/J·mol^{-1} = 13.1					
0.0506	33.2	45.7	0.4003	165.1	172.9	0.7497	121.4	124.7
0.1006	64.1	76.0	0.4501	170.0	177.2	0.7996	101.9	104.6
0.1500	90.1	101.3	0.4996	171.1	177.7	0.8500	83.6	85.6
0.2001	109.7	120.2	0.5499	168.4	174.3	0.9000	59.6	60.9
0.2502	128.7	138.5	0.5998	162.9	168.1	0.9499	29.2	29.8
0.3001	142.2	151.4	0.6500	151.2	155.8			
0.3500	156.6	165.1	0.6998	138.4	142.3			
			$x_2/x_3 = 1.9876$, $H^E_{m,23}$/J·mol^{-1} = 11.9					
0.0500	34.2	45.5	0.3996	183.4	190.5	0.7500	135.2	138.2
0.0997	71.6	82.3	0.4498	188.0	194.5	0.7996	115.0	117.4
0.1500	98.2	108.3	0.4998	189.6	195.6	0.8500	91.6	93.4
0.1999	121.2	130.7	0.5503	187.9	193.2	0.8995	64.9	66.1
0.2497	141.29	150.1	0.6003	179.7	184.4	0.9500	32.8	33.4
0.3000	161.3	169.6	0.6500	170.1	174.3			
0.3500	174.9	182.6	0.6997	155.3	158.9			

Table 7. Coefficients of Equation 4 for the Ternary System *p*-Cymene (1) + α-Pinene (2) + *β*-Pinene (3) and Standard Deviations, *s*

T / K	c_0	c_1	c_2	c_3	c_4	c_5	s / J·mol^{-1}
298.15	1404.80	−3739.62	−4656.68	3016.69	6278.16	4765.98	1.82
308.15	−25.58	−901.83	1785.13	1211.12	−582.83	−1597.38	1.63
318.15	477.07	−1617.43	−371.74	1369.46	1143.80	541.72	1.40

representation. For convenience, the coefficients and standard deviations for the representation of the other two binary mixtures at the same three temperatures are also included in Table 3.

The experimental results for $H^E_{m,1+23}$ and the corresponding values of $H^E_{m,123}$ calculated from eq 1 are listed in Tables 4 to 6 for the temperatures of (298.15, 308.15, and 318.15) K, respectively. The results for $H^E_{m,1+23}$ are plotted in Figure 1 for the temperature of 298.15 K.

Following Tsao and Smith,[16] the values of $H^E_{m,1+23}$ were represented as a sum of binary terms with an added ternary contribution

$$H^E_{m,1+23} = \frac{x_2}{1-x_1} H^E_{m,12} + \frac{x_3}{1-x_1} H^E_{m,13} + H^E_{m,T} \qquad (3)$$

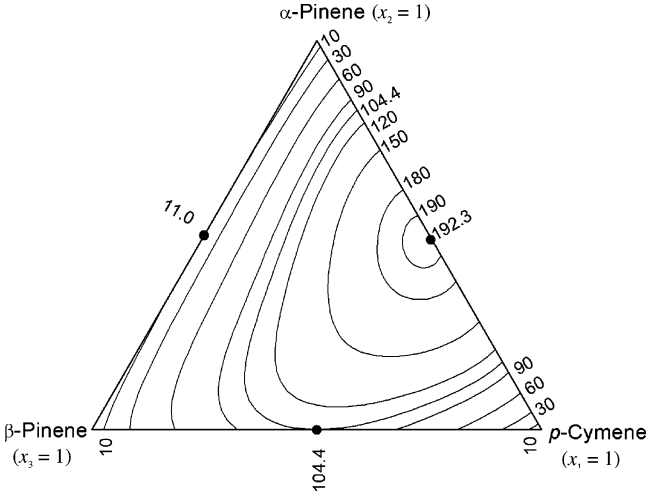

Figure 2. Contours for constant values of $H^E_{m,123}$/J· mol^{-1} for the *p*-cymene (1) + α-pinene (2) + *β*-pinene (3) ternary system at 298.15 K.

where the values of $H^E_{m,ij}$ were calculated from the appropriate smoothing functions. The form

$$H^E_{m,T} = x_1x_2x_3(c_0 + c_1x_1 + c_2x_2 + c_3x_1^2 + c_4x_1x_2 + c_5x_2^2 + c_6x_1^3 + ...) \quad (4)$$

which was adopted for the ternary contribution, is similar to that employed by Morris et al.[17] Values of the coefficients c_i were obtained from least-squares analyses in which eqs 3 and 4 were fitted to the experimental values of $H^E_{m,1+23}$ in Tables 4 to 6. The coefficients of eq 4 for the ternary system *p*-cymene (1) + α-pinene (2) + *β*-pinene (3) at the three temperatures are presented in Table 7 together with the standard deviations of the representation. The solid curves for $H^E_{m,1+23}$ in Figure 1 were calculated from eqs 3 and 4 using these representations.

In addition, eqs 1 to 4 were used to calculate the constant $H^E_{m,123}$ contours plotted on the Roozeboom diagram in Figure 2. The general characteristics illustrated for the ternary system at the three temperatures are very similar.

The excess molar enthalpies of the ternary system are positive and increase somewhat with the temperature. The maximum values of $H^E_{m,1+23}$ occur near $x_1 = 0.5$ as shown in Figure 1. The maxima observed at the three temperatures in Figure 2 are identical to those observed for the *p*-cymene (1) + α-pinene (2) binary system.

The results reported in this paper are a reflection of the physical properties of the components of the mixture. Additional research needs to be conducted to gain a fundamental understanding of such trends as $H^E_{m,21} > H^E_{m,31} > H^E_{m,23}$ that may be a consequence of intramolecular interaction between the different *π*-bonding regions on the different components.

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