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Induced smectic phases. Densities of binary mixtures of 4,4'-dialkylazoxybenzenes with 4-pentyl-4'-cyanobiphenyl (PCB) (*)

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Résumé. — Les densités des mélanges binaires de 4,4'-dipentylazoxybenzène (DPAB) et 4-éthyl-4'-pentylazoxybenzène (EPAB) avec 4-pentyl-4'-cyanobiphényle (PCB) pour les concentrations moyennes et dans l'intervalle des températures 25-80 °C sont mesurées.

Les volumes isothermes de mélange des solutions V^s (égaux aux volumes d'excès V^E , en cas d'existence des deux composants dans la même phase que la solution) sont aussi calculés à partir des interpolations de volumes molaires. Les coefficients isobares de dilatation thermique, pour un mélange de EPAB + PCB sont aussi calculés. La présence de complexes molaires de type A-B est confirmée.

Abstract. — Densities of binary mixtures of 4,4'-dipentylazoxybenzene (DPAB) and 4-ethyl-4'-pentylazoxybenzene (EPAB) with 4-pentyl-4'-cyanobiphenyl (PCB) were measured over the temperature range of 25 to 80 °C and in the middle area of concentrations. Isothermal volumes of mixing V^s (equal to the excess volume V^E when both pure components and solution are in the same phase) were also calculated from interpolated molar volumes. Isobaric thermal expansion coefficients were also calculated for one of the mixture concentrations EPAB + PCB. The presence of a molecular A-B complex has been confirmed.

1. **Introduction.** — In many binary mixtures composed of mesogenic compounds a formation of smectic A phase is observed in the middle area of concentrations with a maximum of clearing temperatures, even if the pure components do not exhibit any smectic phase.

For the substances in which smectic phases occur their stability in mixtures is strongly enhanced.

These smectic phases existing in the middle area of concentrations of the phase diagram are called induced smectic phases and have been of great interest recently [1-14].

To date one finds mostly smectic A phases, but binary systems with induced S_B , S_E phases are also known [14].

Some binary mixtures composed of substances

with different chemical character have been investigated recently by Domon and Billard [12]. They have presented a classification of phase diagrams of induced smectic phases.

Induced smectics are mostly formed in the mixtures of compounds, one of which possess in a terminal position an electron acceptor substituent with a large dipole moment e.g. CN, NO₂, Cl, CF₃, COCH₃, NCS.

The other component is a polar derivative of biphenyl or a derivative of biphenyl with an electron donor group NH₂, NHR, or a substance with a central bond of zero or of a small dipole moment, such as HC=CH, N=N, CH=N, COO or similar bond.

In spite of an accumulation of large experimental material the true mechanism of formation of induced smectic phases and factors determining their stability are not yet satisfactorily understood.

(*) This work was carried out with the Research Project 03.10.

Many authors have expressed the opinion that the formation of a complex of an A-B type plays an important role.

Labes [11], Sharma [13], Park [8] had suggested that the interaction responsible for the complex formation can be of a donor-acceptor character, although others have also drawn attention to that of dipole-induced dipole forces.

Schneider [14] explained the formation of induced smectic phases by separation of polar molecules by the non-polar components counteracting in this manner the formation of bilayer structures. Thus monolayer structures are favoured with an effective side-interaction favourable for smectic phase formation.

We believe that an investigation of density variations with temperature and composition of solutions may be of interest in this context.

In the case of interactions of molecules leading to the formation of A-B complexes a large contraction must be observed on formation of a mixture from pure compounds.

In this paper we present our results for density as a function of composition and temperature for the mixtures composed of 4,4'-dipentylazoxybenzene (DPAB) and 4-pentyl-4'-cyanobiphenyl (PCB) and 4-ethyl-4'-pentylazoxybenzene (EPAB) and 4-pentyl-4'-cyanobiphenyl (PCB). The phase diagrams of these two systems are different. In the first system, in the middle area of concentrations, a nematic phase exists in a quite narrow range of temperature, in the other the nematic phase exists in a larger range of temperatures.

Properties of diluted solutions of these compounds were recently investigated and also in cyclohexane solutions the existence of stable 1 : 1 A-B complexes was confirmed [16].

2. Experimental. — The measurements of density were made with the aid of a digital precision density meter of Anton Paar (type DMA 602 HT) with vibrating tube.

The vibrating tube containing about 1 cm³ of the liquid, was thermostated by an external water circuit from a thermostat bath (about 40 liters).

The temperature of water in this thermostat bath was controlled with temperature controller PID type 650 (Unipan-Poland) with stability better than ± 1 mK from 25 °C to about 50 °C and ± 1.5 mK up to 85 °C.

Temperature stability of the sample was probably better than 0.01 °C.

The temperature measurement was made by using a Hewlett-Packard quartz thermometer with the sensor placed in the reflux water of the thermostating jacket of the measuring cell and inserted as close as possible to the vibrating tube. The sensor of the quartz thermometer was tested immediately before the beginning of measurements with Pt-resistance thermometer.

The total error due to the calibration uncertainties was less than the differences between successive runs. Calibration constants were fixed for the whole temperature range. A calibration run (with 5 °C temperature intervals) was made before and after almost every series of measurements.

The systematic error was about $\pm 7 \times 10^{-5}$ g cm⁻³.

Calibration of instrument was made using air and deionized and distilled water, degassed immediately before measurements.

Density of air was calculated from data in the doctorate thesis of A. Koźdoń [20] and density of water from that of G. S. Kell [21].

Calibration constants and sample density were calculated from data supplied by the manufacturer of densimeter [22].

Components of both mixtures were made in the laboratory by one of us (R. Dąbrowski). 4,4'-dipentylazoxybenzene (DPAB) was synthesized according to the description [19] and its temperatures of phase transitions were : $t_{cN} = 24.5$ °C, $t_{NI} = 67.8-68.0$ °C, 4-ethyl-4'-pentylazoxybenzene (EPAB) was made according to [17] with its phase transition temperatures : $t_{cN} = 5$ °C, $t_{NI} = 38.9$ °C. 4-pentyl-4'-cyanobiphenyl (PCB) obtained according to the description of [18] has its phase transition temperatures for the first sample $t_{cN} = 22.5$ °C, $t_{NI} = 35.7$ °C, the second one $t_{cN} = 22.5$ °C, $t_{NI} = 36.7$ °C.

These components were used without further purification. Immediately before each filling of the apparatus the heated samples of pure compounds and prepared mixtures were degassed under vacuum.

Transition temperatures and the kind of the smectic phase were also estimated prior to the measurements under a polarizing microscope with a heated stage.

The use of the Paar densimeter is quite competitive with the traditional methods of volume determination.

3. Results and discussion. — The results of density measurements in the temperature range between 25 and 80 °C are represented in figures 1 and 2.

The tables with the data points are available on request.

In figures 1 and 2 most curves were shifted for sake of clarity. From our figures with a bigger scale and from the respective density tables we can see that all the mixtures of the second series (EPAB + PCB) are denser than of the first one (DPAB + PCB).

This is of course the consequence of the differences between the densities of DPAB and EPAB and perhaps to a minor degree also of the fact that the stability constants of a complex for compounds with longer alkyl substitution are mostly smaller [16].

In table I we represent densities and molar volumes of DPAB and EPAB for chosen temperatures. We can see that in both phases, nematic and isotropic, EPAB is denser than DPAB. The difference in their structure is the group (CH₂)₃ in one substituent. The

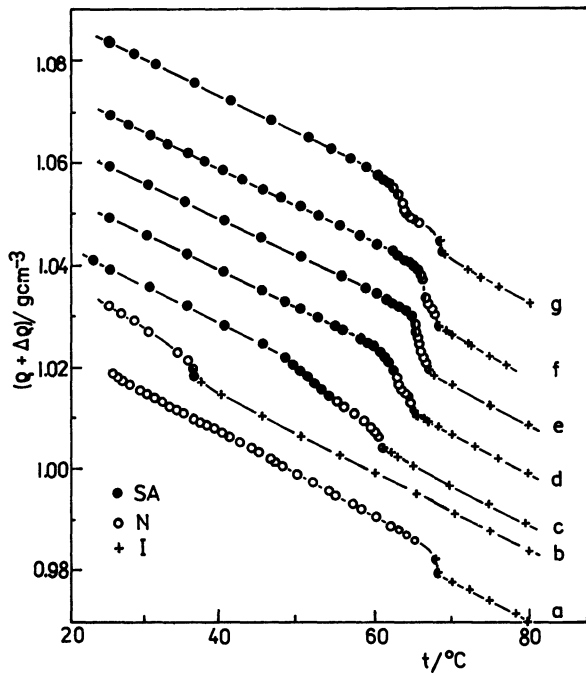


Fig. 1. — Densities of the system DPAB (x) + PCB (1-x) as a function of temperature (mixtures and pure components). The curves are shifted for sake of clarity by addition of different constants $\Delta\rho$: a) $x = 1.000\ 0$, $\Delta\rho = 0.000$; b) $x = 0.000\ 0$, $\Delta\rho = +0.010$; c) $x = 0.356\ 4$, $\Delta\rho = +0.015$; d) $x = 0.452\ 2$, $\Delta\rho = +0.025$; e) $x = 0.504\ 0$, $\Delta\rho = +0.035$; f) $x = 0.605\ 7$, $\Delta\rho = +0.045$; g) $x = 0.692\ 9$, $\Delta\rho = +0.060$.

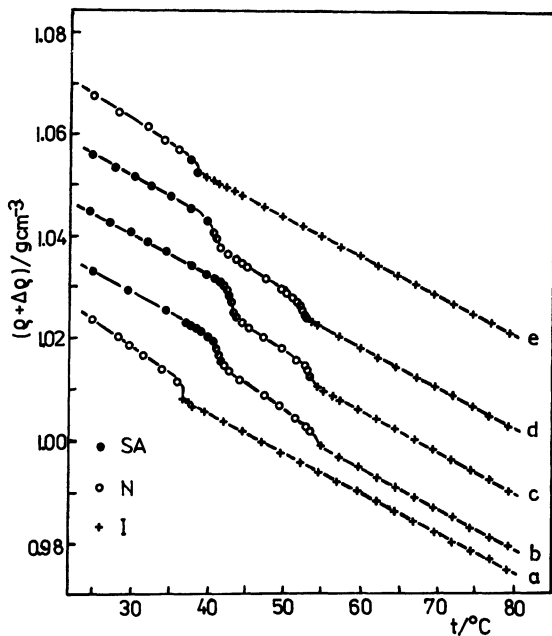


Fig. 2. — Densities of the system EPAB (x) + PCB (1-x) as a function of temperature (mixtures and pure components). The curves are shifted for sake of clarity by addition of different constants $\Delta\rho$: a) $x = 0.000\ 0$, $\Delta\rho = 0.000$; b) $x = 0.443\ 7$, $\Delta\rho = -0.010$; c) $x = 0.492\ 3$, $\Delta\rho = 0.000$; d) $x = 0.585\ 6$, $\Delta\rho = +0.010$; e) $x = 1.000\ 0$, $\Delta\rho = +0.020$.

Table I. — Molar volume and density for chosen temperatures of 4,4'-dipentylazoxybenzene and 4-ethyl-4'-pentylazoxybenzene.

t °C	DPAB M = 338.496		EPAB M = 296.398	
	V cm ³ mole ⁻¹	ρ cm ³ mole ⁻¹	V cm ³ mole ⁻¹	ρ cm ³ mole ⁻¹
25.00	332.200	1.018 95	283.174	1.046 70
30.00	333.385	1.015 33	284.331	1.042 44
35.00	334.625	1.011 57	285.568	1.037 92
70.00	345.821	0.978 82	293.885	1.008 55
75.00	347.249	0.974 79	294.971	1.004 84

same difference can be found between 4-octyl-4'-ethylazoxybenzene and EPAB.

We have calculated the volume of the group $(\text{CH}_2)_3$ in both cases for the temperature of 30.96 °C (temperature of one of the experimental points of 4-octyl-4'-ethylazoxybenzene) [17]. For the pair DPAB and EPAB this volume is 48.60 cm³ mole⁻¹ and for the pair 4-octyl-4'-ethylazoxybenzene and EPAB respectively 49.56 cm³ mole⁻¹ per group. Their difference is about 2%. DPAB being denser than 4-octyl-4'-ethylazoxybenzene.

The situation is quite different, when we made this calculation for the same reduced temperature

$$t_r = \frac{t_{\text{NI}} - t}{t_{\text{NI}}}$$

For the pair DPAB and EPAB the volume of the group $(\text{CH}_2)_3$ is 54.70 cm³ mole⁻¹ and for the pair with 4-octyl-4'-ethylazoxybenzene 50.45 cm³ mole⁻¹ respectively. Their difference is about 8%. 4-octyl-4'-ethylazoxybenzene being denser than DPAB.

In the mixtures of both series (DPAB + PCB and EPAB + PCB) we have observed strong pretransitional effects near the transition $S_A \rightarrow N$, on the side of the smectic A phase.

The pretransitional effects and the impossibility of observation of the first drop of the new phase or the vanishing of the last drop made it impossible to determine exactly the jump $\Delta\rho$ at the phase transition.

Extraordinarily strong pretransitional effects could be observed in the smectic phase of the mixture of DPAB + PCB series near the composition of $x_1 = 0.5$ (1), where the nematic phase exists only in a narrow temperature range (0.85 °C for $x_1 = 0.504\ 0$); the pretransitional effects and the two phase region being nearly in superposition.

We estimated graphically the transition volume jump (without pretransitional area) for EPAB + PCB series, this one of wider temperature ranges of nematic phase (10.24 °C for $x_1 = 0.492\ 3$).

(1) x_1 = mole fraction of DPAB or EPAB.

These results are respectively :

$$\begin{aligned} \text{PCB} : \Delta V &= 0.52 \pm 0.03 \text{ cm}^3 \text{ mole}^{-1} \\ \text{EPAB} : \Delta V &= 0.36 \pm 0.03 \text{ cm}^3 \text{ mole}^{-1} \end{aligned} \quad \text{transition N} \rightleftharpoons \text{I}$$

for mixtures of these components :

$$\begin{aligned} x_1 = 0.4437 \quad \Delta V_{\text{SAN}} &= 0.47 \pm 0.15 \text{ cm}^3 \text{ mole}^{-1} & \Delta V_{\text{NI}} &= 0.36 \pm 0.10 \text{ cm}^3 \text{ mole}^{-1} \\ x_1 = 0.4923 \quad \Delta V_{\text{SAN}} &= 1.08 \pm 0.15 \text{ cm}^3 \text{ mole}^{-1} & \Delta V_{\text{NI}} &= 0.40 \pm 0.10 \text{ cm}^3 \text{ mole}^{-1} \\ x_1 = 0.5856 \quad \Delta V_{\text{SAN}} &= 0.63 \pm 0.15 \text{ cm}^3 \text{ mole}^{-1} & \Delta V_{\text{NI}} &= 0.39 \pm 0.10 \text{ cm}^3 \text{ mole}^{-1} \end{aligned}$$

Table II. — Interpolated molar volumes and volumes of isothermal mixing of solution (V^s) for chosen temperatures of a serie : 4,4'-dipentylazoxybenzene (DPAB) + 4-pentyl-4'-cyanobiphenyl (PCB).

x	0.000 0	0.356 4		0.452 2		0.504 0		0.605 7		0.692 9		1.000 0
	V cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹
	nematic	smectic A										nematic
25.00	243.985	274.545	- 0.880	282.811	- 1.035	287.365	- 1.081	296.260	- 1.157	304.123	- 0.986	332.200
30.00	245.105	275.483	- 1.085	283.774	- 1.251	288.304	- 1.295	297.225	- 1.351	305.120	- 1.154	333.385
35.00	246.460	276.444	- 1.439	284.719	- 1.610	289.259	- 1.636	298.192	- 1.669	306.135	- 1.415	334.625
	all components and their mixtures in isotropic phase ($V^s = V^E$)											
70.00	254.061	286.299	- 0.466	294.955	- 0.600	299.703	- 0.605	308.987	- 0.653	317.002	- 0.640	345.821
75.00	255.027	287.398	- 0.498	296.146	- 0.584	300.942	- 0.565	310.282	- 0.604	318.355	- 0.573	347.249

x = mole fraction of DPAB.

Table III. — Interpolated molar volumes and volumes of isothermal mixing of solution (V^s) for chosen temperatures of a serie : 4-ethyl-4'-pentylazoxybenzene (EPAB) + 4-pentyl-4'-cyanobiphenyl (PCB).

x	0.000 0	0.443 7		0.492 3		0.585 6		1.000 0
	V cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹	V^s cm ³ mole ⁻¹	V cm ³ mole ⁻¹
	nematic	smectic A						nematic
25.00	243.640	259.148	- 2.032	261.033	- 2.070	264.846	- 1.945	283.174
30.00	244.764	260.094	- 2.226	261.964	- 2.279	265.800	- 2.134	284.331
35.00	246.056	261.075	- 2.513	262.920	- 2.588	266.796	- 2.398	285.568
40.00	isotropic	smectic A						isotropic
	247.910	262.163	- 3.246	263.958	- 3.369	268.088	- 2.918	287.351
	isotropic	nematic						isotropic
45.00	248.924	264.503	- 1.973	266.418	- 1.982	270.322	- 1.768	288.483
47.00	249.321	265.029	- 1.856	266.955	- 1.854	270.848	- 1.654	288.906
50.00	249.907	265.837	- 1.660	267.772	- 1.652	271.669	- 1.454	289.551
52.00	250.303	266.432	- 1.482	268.370	- 1.473	272.315	- 1.231	289.994
	all components and their mixtures in isotropic phase ($V^s = V^E$)							
56.00	251.089	268.052	- 0.686	269.983	- 0.689	273.760	- 0.623	290.867
57.00	251.283	268.282	- 0.660	270.205	- 0.671	273.984	- 0.605	291.082
60.00	251.858	268.936	- 0.584	270.859	- 0.625	274.638	- 0.565	291.724
65.00	252.827	270.002	- 0.564	271.931	- 0.579	275.713	- 0.527	292.809
70.00	253.787	271.051	- 0.527	272.987	- 0.540	276.771	- 0.497	293.885
75.00	254.754	272.094	- 0.504	274.038	- 0.514	277.830	- 0.474	294.971

x = mole fraction of EPAB.

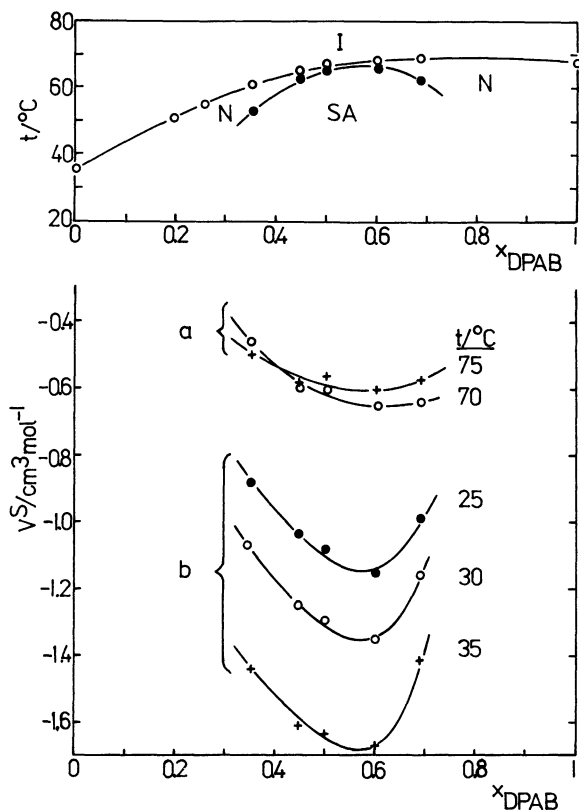


Fig. 3. — Phase diagram of the system DPAB + PCB and V^s versus mole fraction of DPAB (x): a) mixtures and components in isotropic phase ($V^s = V^E$); b) mixtures in smectic A phase; components in nematic phase.

Thus, we can see that $\frac{\Delta V_{SAN}}{\Delta V_{NI}} > 1$.

One of us [17] (K. W. Sadowska) found

$$\Delta V_{NI} \approx 2 \Delta V_{SAN}$$

for pure 4-ethyl-4'-decylazoxybenzene, one of the homologues exhibiting both a smectic A phase and a nematic one.

The density determined for the samples of the liquid-crystalline series DPAB + PCB and EPAB + PCB are used for calculations of molar volumes of pure components and their mixtures in temperature region 25 to 80 °C.

From interpolated molar volumes at rounded temperature values (see Table II and Table III) we calculated the volumes of isothermal mixing of solutions as composed from pure components at these temperatures. These volumes V^s are equal to the excess values V^E in the cases when both pure components and the solution are in the same phase.

The results and respective phase diagrams are shown in figures 3 and 4.

In all cases V^s is negative.

The negative sign of $V^s = V^E$ and their relatively large absolute values are an additional evidence in favour of the existence in mixtures of both series of a complex of the A-B type suggested earlier [16].

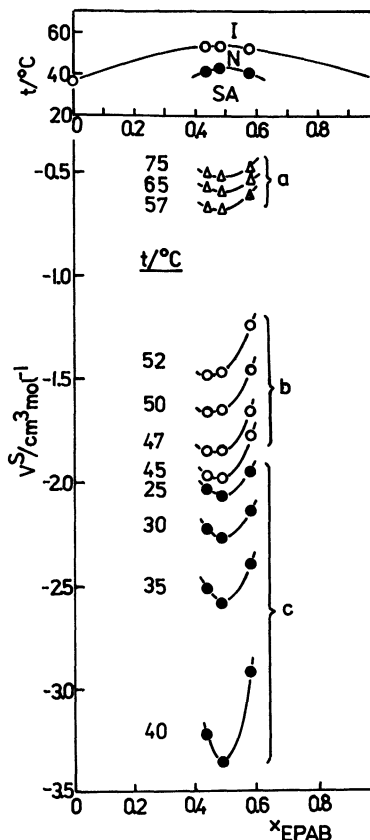


Fig. 4. — Phase diagram of the system EPAB (x) + PCB ($1 - x$)

and V^s versus mole fraction of EPAB (x): a) mixtures and components in isotropic phase ($V^s = V^E$); b) mixtures in nematic phase, components in isotropic phase; c) mixtures in smectic A phase, components in nematic phase.

In the cases when both components and the solution are in different phases the absolute values of V^s are bigger than they would be if the mixture and their components are in the same phase.

The sequences of V^s vs. t of the two series, for temperatures 25, 30 and 35 °C, are quite different than at other temperatures. The absolute value of V^s increases with increase of temperature. In the isotropic phase the absolute value of $V^s = V^E$ decreases with increase of temperature.

We calculated also the (isobaric) thermal expansion coefficients for a mixture of EPAB + PCB for $x_1 = 0.4923$ (Table IV) and the values of :

$$\left(\frac{dV^s}{dt}\right) = \left(\frac{dV}{dt}\right) - x_1 \left(\frac{dV_1}{dt}\right) - (1 - x_1) \left(\frac{dV_2}{dt}\right). \tag{1}$$

Table IV shows that dV^s/dt is negative for temperatures 25, 30 and 35 °C, because the mixture component of the sum in equation (1), dV/dt is smallest (0.19) in this case. The contribution of the pure components, taken with a negative sign (Eq. (1))

Table IV. — Isobaric thermal coefficients of expansivity for a mixture of EPAB + PCB for $x_1 = 0.4923$ and their components.

t °C	Mixture		Components				$\frac{\Delta V^s}{\Delta t} = \frac{\Delta V_{\text{mix}}}{\Delta t} - x_1 \frac{\Delta V_1}{\Delta t} - (1 - x_1) \frac{\Delta V_2}{\Delta t}$
	phase	$\frac{\Delta V}{\Delta t}$	EPAB		PCB		
			phase	$\frac{\Delta V_1}{\Delta t}$	phase	$\frac{\Delta V_2}{\Delta t}$	
		cm ³ mol ⁻¹ °C ⁻¹		cm ³ mol ⁻¹ °C ⁻¹		cm ³ mol ⁻¹ °C ⁻¹	cm ³ mol ⁻¹ °C ⁻¹
25	S _A	0.184	N	0.229	N	0.220	- 0.040
30	S _A	0.189	N	0.238	N	0.237	- 0.048
35	S _A	0.193	N	0.277	N	0.326	- 0.109
45	N	0.272	I	0.222	I	0.199	+ 0.062
47	N	0.268	I	0.222	I	0.199	+ 0.058
56	I	0.227	I	0.215	I	0.193	+ 0.023
60	I	0.216	I	0.218	I	0.193	+ 0.011
65	I	0.211	I	0.216	I	0.192	+ 0.007
70	I	0.210	I	0.217	I	0.192	+ 0.006
75	I	0.212	I	0.217	I	0.194	+ 0.007

exceeds the positive contribution of dV/dt . For these temperatures the mixtures are in a smectic phase, their pure compounds are in a nematic phase. For mixtures in nematic or isotropic phase dV^s/dt is positive.

The presence of an A-B complex, undoubtedly formed in all three phases, I, N, or S_A, has then little to do with the difference in behaviour of dV^s/dt ; rather it is likely that simply dV/dt is smaller in the smectic A phase as compared with the N or I phase

(see Table IV). This seems plausible as the structure of the smectic A phase is more constrained and hence the volume change with temperature is smaller. It could be interesting to compare dV/dt in induced smectic A phases in which the A-B complex is formed with dV/dt of the ordinary smectic A phases.

Incidentally, we also note from table IV that $V^s = V^E$ in the isotropic phase is negative and increases with temperature to a value of -0.514 at 75 °C.

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- $$\rho_{\text{air}} = \frac{0.0017047(p - 0.00378 \times p_n \times \eta)}{1000 \times (1 + 0.003673 \times t)} \text{ (g cm}^{-3}\text{)}$$
- p : atmospheric pressure in mmHg
 p_n : water saturated pressure
 η : related humidity in %.
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