

# Phase Transitions by Dilatometry, Part I: Synthesis, Characterisation and Isotropic to Smectic-A and Smectic-A to Smectic-B Transitions in Two Liquid Crystalline Higher Homologues of N-(*p*-*n*-nonyloxybenzylidene) *p*-*n*-alkylanilines

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Two higher homologues of N(*p*-*n*-nonyloxybenzylidene)*p*-*n*-alkylanilines, viz. the 9O.*m* series with *m* = 12 and 16, are synthesised and characterised by thermal microscopy, differential scanning calorimetry and density studies. The compounds exhibit the phase variants smectic-A, smectic-B and smectic-G. Density studies reveal the first order nature of the isotropic to smectic-A and smectic-A to smectic-B transitions. An estimate of the pressure dependence of the phase transition temperature, using volume and enthalpy data, is presented. A comparison of these results with those reported on *n*O.*m* and other liquid crystalline compounds is presented.

**Key words:** Phase-transitions in Liquid Crystals; Experimental Determination of Smectic Phases; Mass and Density; Pressure Effects.

## 1. Introduction

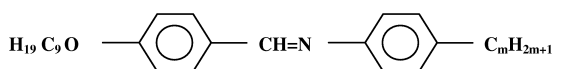
The N-(*p*-*n*-alkyloxybenzylidene)*p*-*n*-alkylaniline series (known as *n*O.*m* series) shows rich and complex polymesomorphism [1–5]. Measurement of the density variation with temperature [6–9] allows to determine the order of the phase transitions, the pre-transitional [10–11] behaviour and the critical exponent [12]. The dilatometric technique gives complementary results [13–15] to other techniques, such as differential scanning calorimetry (DSC) and thermal microscopy (TM). The thermal stability of the smectic-A phase is important, because this phase is used in modern liquid crystal displays [16].

This paper presents the synthesis and characterisation of two members of the *n*O.*m* series, viz. 9O.*m* with *m* = 12 and *m* = 16. Their phase variants, transition temperatures, transition enthalpies, and the nature of their isotropic to smectic-A and smectic-A to smectic-B transitions are given. The pressure dependence of the transition temperatures is estimated from the measured volume jumps and enthalpy data available in the literature. The results are compared with reported data

[10, 15] of the *n*O.*m* series and other liquid crystalline compounds.

## 2. Experimental

The mentioned two members of *n*O.*m* series are prepared [17] by condensation of *p*-*n*-nonyloxybenzaldehyde (0.1 mole) with appropriate *p*-*n*-alkylanilines (*n* = 12 and 16, each 0.1 mole) by refluxing in absolute ethanol in the presence of a few drops of glacial acetic acid. After refluxing the mixture for four hours, the solvent is removed by distillation under reduced pressure. The pure compound is recrystallised from cold ethanol, until the transition temperatures become reproducible. The molecular formula for 9O.*m* compounds is



A Hertel-Reuss (super pan II) polarising microscope and a personal computer (PC) monitoring the Instec mk-1 heating stage with  $\pm 0.1$  °C accuracy, was used for the textural identification of the LC phases and the determination of the phase transition temperatures.

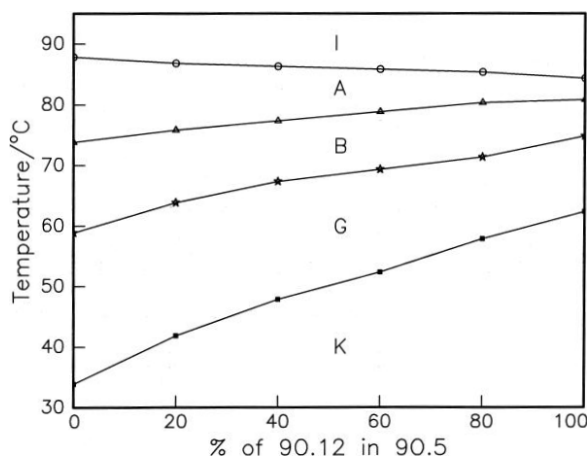


Fig. 1. Miscibility diagram for 9O.12 + 9O.5.

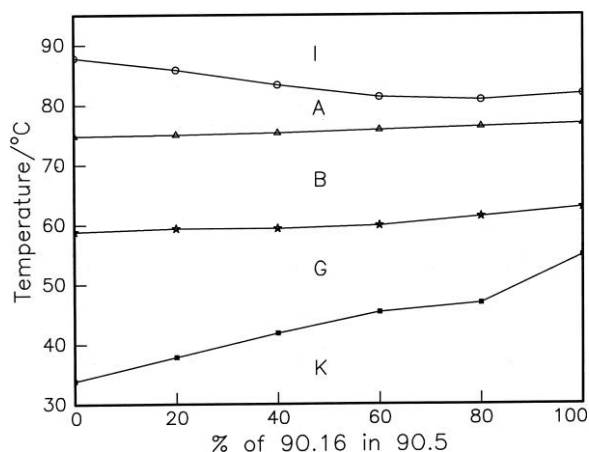


Fig. 2. Miscibility diagram for 9O.16 + 9O.5.

A Perkin Elmer DSC-7 system was also used for the determination of the transition temperatures and the relevant enthalpies of transitions. An U-shaped bicapillary pycnometer and a cathetometer were used to study the temperature variation of the density. The error in the density investigations was  $\pm 10^{-4}$  g/cm<sup>3</sup>. The cooling rate, during the density experiments was 0.5 °C per hour.

### 3. Results and Discussion

Our preliminary textural studies with a TM showed that on cooling the samples from the isotropic liquid, a mesomorphic phase  $S_1$  appears in the form of batonnets (at 86.3 °C for 9O.12 and 79.7 °C for 9O.16), and these batonnets coalesce into a focal conic fan texture. This phase also exhibits a homeotropic texture (or pseudo-isotropic texture), indicating it as an orthogonal phase. The focal conic fan texture observed in the two compounds is similar to the texture obtained in [18] for the smectic-A ( $S_A$ ) phase in 9O.5. These observations indicate that the phase may be  $S_A$ . On further cooling, the  $S_A$  phase transforms into another smectic phase  $S_2$  (at 80.4 °C for 9O.12 and 75.7 °C for 9O.16). The transition is indicated by transient transition bars across the focal conic fans of the  $S_A$  phase, which disappeared after the completion of the transition, leading to a smooth focal conic fan texture. The smooth focal conic fan texture and the appearance of transient transition bars across the phase boundary characterise the  $S_2$  phase as smectic-B ( $S_B$ ) phase. The transient transition bars across  $S_A$ - $S_B$  and the smooth focal conic fan texture of the  $S_2$  phase are

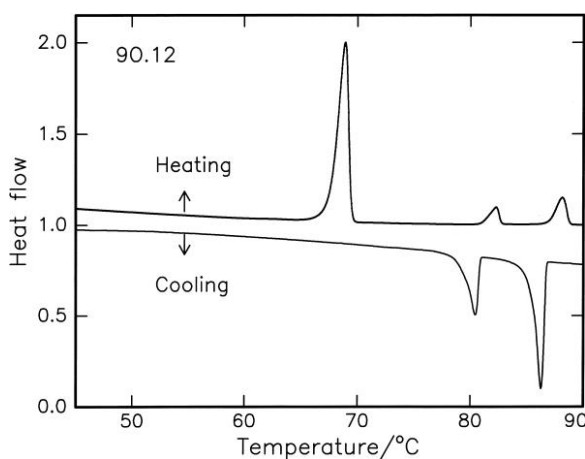


Fig. 3. The DSC thermogram of 9O.12.

similar to the textures obtained [18] in the  $S_B$  phase in 9O.5 and 5O.9 compounds. On further cooling (77 °C for 9O.12 and 77.2 °C for 9O.16) the compounds exhibit a striped-broken focal conic fan texture characterising the phase as smectic-G ( $S_G$ ). On further cooling, the smectic-G phase of 9O.12 and 9O.16 is transformed to crystal at 65 and 55 °C, respectively. The ABG phase variant observed in 9O.12 and 9O.16 is further confirmed by miscibility studies, using N-(*p*-nonyloxybenzylidene)*p*-*n*-pentylaniline, (9O.5) as the standard compound which also exhibits an ABG phase variant, as these three phases are continuously miscible in the binary system. The phase diagrams of the [9O.5 + 9O.12] and [9O.5 + 9O.16] systems are illustrated in Figs. 1 and 2. The DSC thermogram of 9O.12 is shown in Figure 3.

Table 1. Transition temperatures in °C, obtained from Thermal Microscopy (TM), Differential Scanning Calorimetry (DSC) and Density measurements, along with enthalpy values ( $\Delta H$ , in J/g), for the compounds 9O.12 and 9O.16.

Compound (Phase Variant)	Method	Isotropic to Smectic-A	Smectic-A to Smectic-B	Smectic-B to Smectic-G	Smectic-G to Crystal	Ref.
9O.12 (ABG)	TM	86.3	80.4	77	65	a
	DSC	86.25	80.42	—	—	
	Density	86.3	80.4	—	—	
	Enthalpy:	16.47	8.83	—	—	
9O.16 (ABG)	TM	79.7	75.7	63.5	55	a
	DSC	79.62	75.67	—	45.36	
	Density	79.7	75.7	—	—	
	Enthalpy:	1.61	4.22	—	106.52	

<sup>a</sup> Present work.

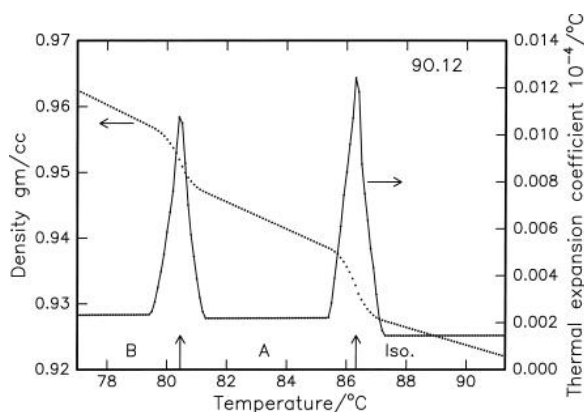


Fig. 4. Temperature variation of the density,  $\rho(T)$  and thermal expansion coefficient,  $\alpha(T)$  for the compound 9O.12.

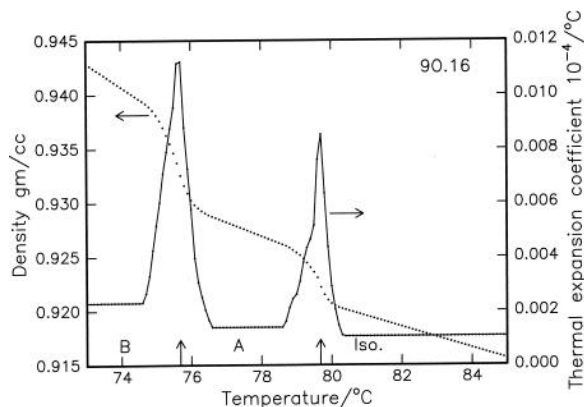


Fig. 5. Temperature variation of the density,  $\rho(T)$  and thermal expansion coefficient,  $\alpha(T)$  for the compound 9O.16.

The average increment of the molar volume contributed by an extra methylene group estimated from the observed molar volumes,  $M_V$  (mol.wt/density) in the equilibrium isotropic liquid (i.e. at  $T_{IA} + 5$  °C) is found to be  $16 \cdot 10^{-6} \text{ cm}^3 \text{ mol}^{-1}$  in the homologues

of the 9O.*m* series and agrees with the reported values for 8O.*m* [19] and other Schiff based [20–21] liquid crystalline systems. This agrees also with the reported value for the methylene unit contribution [22] in normal isotropic liquids. The variation of the density and volume expansion coefficient for the compounds 9O.12 and 9O.16 are presented in Figs. 4 and 5. The transition temperatures for the compounds 9O.12 and 9O.16 obtained from TM, DSC, and dilatometry along with the enthalpy values are presented in Table 1.

#### 4. Isotropic to Smectic-A Transition (IA)

The isotropic to smectic-A (IA) transition brings about a layered arrangement of a few molecules, and is expected to be of first order. The IA transition is accompanied by a large density jump in these compounds. It amounts to 0.9% and 0.42% in the 9O.12 and 9O.16 compounds, respectively. The density jumps across the IA transition, the thermal expansion coefficient maxima ( $\alpha_{\max}$ ) and the enthalpy values ( $\Delta H$ ) are presented in Table 2. They fall between the maximum and minimum values reported in [23–30] of the *nO.m*'s, terephthalylidene-bis-*p-n*-alkylanilines (TbnA), *n*(4-*n*-phenylbenzylidene) 4-*n*-alkylaniline (PBnA), 5-*n*-alkyl-2-(4-*n*-alkyloxy-phenyl)-pyrimidines (PYP *nO.m*),  $\alpha$ ,  $\omega$ -Bis(4-*n*-alkylaniline-benzylidene-4'-oxy) alkanes (*m.OnO.m*) and other liquid crystalline compounds. The magnitude of the density jumps and the thermal expansion coefficient maxima in these compounds indicate the first order nature of the IA phase transition.

The dilatometric studies did show that the IA transitions are accompanied by regions of large temperature-fluctuations of maximal  $\approx 1.5$  °C. It is observed that the translucent smectic-A phase grows at the bottom of the pyknometer bulb, while a transparent isotropic liquid floats above it. The smectic-A phase with a clear separation boundary is found to permeate from the isotropic liquid all through the pyknometer with the decrease of the temperature when the smectic-A phase is thermally stabilized. The formation of smectic-A embryos at the bottom of the bulb, and their growth clearly indicate the nucleation type of the growth at the IA phase transition. Further, the density jump observed during the IA transition in 9O.16 is at least by a factor of two smaller than that of all other members of the series. It is also found that the density jumps in 9O.14 and 9O.16 across the IA transition decrease with increasing num-

Table 2. Density jumps  $\Delta\rho/\rho$ , Thermal expansion coefficient maxima  $\alpha_{\max}$ , Enthalpy values  $\Delta H$ , Pressure dependence of transition temperatures  $dT_i/dP$  across the IA phase transition in *nOm* and other compounds.

Compound		$\Delta\rho/\rho$ -100	$\alpha_{\max}$ ( $10^{-4} \text{ }^\circ\text{C}^{-1}$ )	$\Delta H$ (J/g)	$dT_i/dP$ (K/kbar)	Ref.
70.6		1.07	222	16.11	24.6	[10]
70.8		1.04	225	14.40	27.3	[10]
70.9		0.8	205	—	—	[10]
70.10		1.08	259	—	—	[10]
70.14		0.99	124	13.54	10.4	[24]
80.4		1.11	122	15.53	26.5	[21]
80.5		1.24	144	14.31	32.7	[6]
80.6		1.37	114	17.00	30.6	[6]
80.7		1.32	146	16.02	31.5	[6]
80.8		0.73	122	—	—	[34]
80.9		0.81	66	17.74	17.8	[6]
80.10		0.9	239	—	—	[19]
80.14		1.1	154	15.56	24.4	[24]
90.4		1.23	390	16.6	39.5	[35]
90.5		1.18	280	9.14	50.0	[18]
90.6		1.51	440	22.38	25.5	[23]
90.8		1.95	490	17.53	43.6	[23]
90.12		0.90	123	16.47	21.1	<sup>a</sup>
90.14		1.6	156	9.75	40.6	[24]
90.16		0.42	85	1.61	100.7	<sup>a</sup>
100.6		1.32	344	20.35	—	[25]
100.8		1.5	629	18.65	32.4	[36]
100.9		1.4	480	19.08	29.2	[36]
100.10		0.87	282	9.44	17.6	[36]
100.12		0.82	113	11.09	29.7	[36]
100.16		1.6	173	13.17	47.7	[36]
130.1		0.57	98.7	18.37	14.86	[26]
140.1		0.93	133.7	21.62	15.85	[26]
di- <i>n</i> -Hexadecyl 4,4'-azoxy cinnamate		0.4	—	6.05	26.7	[23]
di- <i>n</i> -Decyl 4,4'-azoxy cinnamate		0.35	—	8.59	34.9	[23]
di- <i>n</i> -Decyl 4,4'-azoxy $\alpha$ - methylcinnamate		1.21	—	12.70	18.9	[23]
<i>n</i> -Amyl 4(4- <i>n</i> -dodecyloxy benzylidene) amino-cinnate		1.28	—	16.6	33.7	[23]
Diethyl 4-4'-azoxy benzoate		2.00	—	15.9	43.0	[23]
Terephthalidene bis(4'- <i>n</i> -octylaniline)	TBAA8	0.90	62.5	11.10	42.0	[32]
Terephthalidene bis(4'- <i>n</i> -nonylaniline)	TBAA9	1.00	77	12.44	41.5	[32]
Terephthalidene bis(4'- <i>n</i> -decylaniline)	TBAA10	1.82	183	12.53	72.2	[32]
<i>n</i> (4- <i>n</i> -phenylbenzylidene) 4- <i>n</i> -decylaniline	PB10A	0.70	84	6.77	44.3	[31]
<i>n</i> (4- <i>n</i> -phenylbenzylidene) 4- <i>n</i> -dodecylaniline	PB12A	1.17	96	13.58	36.6	[31]
<i>n</i> (4- <i>n</i> -phenylbenzylidene) 4- <i>n</i> -tetradecylaniline	PB14A	1.10	88	9.1	44.2	[31]
<i>n</i> (4- <i>n</i> -phenylbenzylidene) 4- <i>n</i> -hexadecylaniline	PB16A	0.36	58	6.48	23.2	[31]
5- <i>n</i> -alkyl-2-(4- <i>n</i> -alkyloxy-phenyl)-pyrimidines PYP <i>nOm</i> for <i>n</i> = 9 & <i>m</i> = 9 (i.e. PYP 9O9)		1.37	791	—	—	[27]
5- <i>n</i> -alkyl-2-(4- <i>n</i> -alkyloxy-phenyl)-pyrimidines PYP <i>nOm</i> for <i>n</i> = 9 & <i>m</i> = 7 (i.e. PYP 9O7)		1.35	644	—	—	[27]
N( <i>p</i> - <i>n</i> -Decyloxybenzylidene) <i>p</i> -amino 2-methylbutyl cinnamate (DOBAMBC)		0.81	155	10.58	31.0	[28]
$\alpha$ , $\omega$ -Bis(4- <i>n</i> -heptylanilinebenzylidene-4'-oxy) butane (7.O4O.7)		1.57	428	27.39	29.08	[29]
$\alpha$ , $\omega$ -Bis(4- <i>n</i> -heptylanilinebenzylidene-4'-oxy) pentane (7.O5O.7)		0.95	182	13.73	30.07	[29]

<sup>a</sup> Present work.

ber of methylene units of the flexible alkyl-end chain. A similar trend was reported for the IA transition [31] in the (PBnA) series (14 to 16), for the isotropic to smectic-C transition [11] in the (TBnA) series (14 to 16), and for the isotropic to smectic-F transition [8] in the (12O*m*) series (12 to 16). The trend of a decreasing density jump with increasing length of the flexible end chain from the isotropic liquid signifies a de-

creasing thermal potential barrier across the isotropic to smectic-A interface.

The density variations with temperature in the equilibrium isotropic phase  $(d\rho/dT)_{\text{iso}}$ , in 9O.12 and 9O.16 are  $13.4 \cdot 10^{-4} \text{ }^\circ\text{C}^{-1}$  and  $9.8 \cdot 10^{-4} \text{ }^\circ\text{C}^{-1}$ , respectively. They agree with those observed [19] for the equilibrium isotropic liquid state of other *nOm* liquid crystalline compounds. The values of  $(d\rho/dT)_A$  for

the equilibrium smectic-A phase in 9O.12 and 9O.16 are  $20.66 \cdot 10^{-4} \text{ }^{\circ}\text{C}^{-1}$  and  $13 \cdot 10^{-4} \text{ }^{\circ}\text{C}^{-1}$ , respectively, and these agree with the observed [19] data on the smectic-A phase of other [10, 18, 21, 24] *nO.m* liquid crystalline compounds. The higher slopes of  $d\rho/dT$  in the smectic-A phase than in the isotropic phase suggest an additional packing with positional and translational order.

The pressure dependence of the IA transition temperature, estimated in [23], using the Clausius-Clapeyron equation, from the data of calorimetry and dilatometry, along with the values reported for other *nO.m* compounds, is presented in Table 2. Generally, the observed poor agreement in the pressure dependence of the IA transition temperature among various [23] compounds, as presented in Table 2, which shows the thermal stability of the mesophase in the chemical environment, may be due to the parameters being obtained under different experimental conditions. However it is useful to note that the higher value of 100.7 K/kbar obtained for the 9O.16 compound is due to the small value of the transition enthalpy measured by DSC (Table 1). The obtained values of pressure dependence of the phase transition temperatures for the 9O.12 compound (as well for 8O.9) show that the smectic-A temperature range decreases under pressure. However, the values of 9O.16 (as well as those 8O.4, 8O.5 and 9O.5) exhibit an opposite behaviour, i.e. the smectic-A mesophase is stabilized by pressure.

### 5. Smectic-A to Crystalline Smectic-B Transition (AB)

This smectic-A to crystalline smectic-B transition is associated with the development of long range three dimensional order and molecular positional correlation of hexagonal inplane stacking with its layered structures. The density jumps of 0.68% and 0.74% and the volume expansion coefficient maxima ( $\alpha_{\max}$ ) of  $107.32 \cdot 10^{-4} \text{ }^{\circ}\text{C}^{-1}$  and  $112.4 \cdot 10^{-4} \text{ }^{\circ}\text{C}^{-1}$  for 9O.12 and 9O.16, respectively, indicate the first order nature of the AB transition, as expected [33], with the growth of orthogonal 3-D hexagonal ordering of the molecules as observed [33] in other *nO.m* compounds. The sticky nature of the substance (to the inner walls of the pycnometer) did not allow us to further study the temperature dependence of the density (deep into the smectic-B phase or across the smectic-B to smectic-G transitions). The density jumps, thermal expansion coefficient maxima and the pressure temperature on de-

Table 3. Thermal ranges of the smectic-A and smectic-B phases of *nO.m* compounds, Density jumps, Thermal expansion coefficient maxima, Enthalpy values, Pressure dependence of transition temperatures across the AB phase transition.

Com- pound	S <sub>A</sub> -Thermal Range (°C)	S <sub>B</sub> -Thermal Range (°C)	$\Delta\rho/\rho$ ·100	$\alpha_{\max}$ ( $10^{-4} \text{ }^{\circ}\text{C}^{-1}$ )	$\Delta H$ (J/g)	$dT_i/dP$ (K/kbar)	Ref.
4O.4	0.7	—	0.57	386	—	19.7	[33]
4O.5	3.4	—	0.53	346	9.28	17.8	[33]
4O.6	7.4	—	0.34	188	—	16.6	[33]
4O.8	14	16.0	0.62	340	—	41.0	[33]
4O.9	16.7	—	0.71	215	5.43	45.3	[33]
4O.10	17.9	2.5	1.08	509	4.79	—	[33]
4O.12	17.6	12.9	0.90	585	8.83	33.8	[33]
5O.8	15.8	24.2	0.79	150	5.95	49.8	[37]
5O.9	17.2	6.6	0.76	145	6.93	33.7	[18]
5O.10	13.0	13.0	0.76	150	—	—	[38]
5O.12	17.1	23.8	1.40	250	16.01	93.4	[36]
5O.14	17.6	8.7	0.53	191	5.34	34.3	[36]
6O.4	11.1	2.0	—	30	—	—	[17]
6O.5	13.7	16.3	0.62	180	7.80	26.9	[10]
6O.6	14.7	26.8	0.78	124	6.43	36.6	[10]
6O.7	14.5	—	1.43	598	8.05	61.8	[10]
6O.8	15.7	51.3	0.51	—	—	25.8	[39]
7O.2	6.0	2.0	1.59	401	12.47	42.5	[10]
7O.3	8.5	29.0	1.41	362	13.74	35.2	[10]
7O.10	12.2	15.6	1.37	357	—	—	[10]
7O.14	9.9	1.5	0.99	124	13.54	—	[24]
8O.4	13.2	3.2	0.40	85	—	15	[21]
8O.5	14.6	18.7	0.54	92	9.01	21.1	[6]
8O.8	8.7	19.3	0.24	71	—	—	[34]
8O.9	10.9	29.5	0.60	60	8.53	25.9	[6]
8O.10	9.6	34.5	1.41	376	—	—	[19]
8O.14	5.4	1.8	1.10	154	7.79	—	[24]
9O.5	13	14.5	1.36	288	16.03	30.2	[18]
9O.12	5.9	3.4	0.68	107	8.83	28.7	<sup>a</sup>
9O.14	3.2	8.4	1.60	156	6.54	—	[24]
9O.16	4.0	12.2	0.74	112	4.22	65.4	<sup>a</sup>

<sup>a</sup> Present work.

pendence of the AB transition, estimated [23] using Clausius-Clapeyron equation from the data of calorimetry and dilatometry observed for the compounds 9O.12 and 9O.16 along with the values reported in literature for other *nO.m* compounds, are presented in Table 3. The values are found to agree well with the values reported in literature for other *nO.m* compounds.

### 6. Summary

The transition temperatures of the compounds found from TM, DSC and density are found to agree well with each other. The compounds 9O.12 and 9O.16 exhibit ABG phase variants. Density studies reveal a first order nature of the IA and AB transitions. An estimate of the pressure dependence of the phase transition temperatures, using volume and enthalpy data, are found to be consistent with other reported results in *nO.m* and other compounds.

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