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Phase Transition Studies and Thermodynamic Parameters of Four Members of nO.m Series—A Dilatometric Study

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Density measurements are carried out on four alkoxy benzylidene alkyl anilines, viz., N-(p-n-methoxy/ethoxybenzylidene)-p-n-dodecyl and tetradecylanilines, 1O.12, 1O.14, 2O.12, and 2O.14. The first three compounds exhibit monovariant nematic phase while the last compound exhibits nematic and smectic-A phase with variable thermal ranges. The density measurements infer the first order nature for Isotropic to nematic (Iso-N) phase transformation while the nematic to smetic-A (N-SmA) transition as weak first order. Thermal expansion coefficient α estimated from density ρ is utilized for the computation of thermodynamic parameters of above liquid crystalline compounds. The temperature variation of different thermodynamic parameters like Moelwyn-Hughes parameter, reduced volume, reduced compressibility, isothermal, isochoric, and isobaric Gruneisan parameters, etc., are estimated. The results are compared with the available data in literature of other compounds.

Keywords Density; *n*O.*m* compounds; phase transitions; thermodynamic parameters

Introduction

Alkoxybenzylidene alkylanilines with varying alkoxy and alkyl chain number form a family of liquid crystals (LC) and are extensively studied for their ability to exhibit different liquid crystalline variants showing from single variant to hexavariant [1] and for understanding the nature of different phase transformations [2–6]. Dilatometric studies are carried out on a number of compounds at different mesomorphic interfaces [7,8]. Further, it has been observed that the formation as well as the thermal span of the nematic phase depends on the chain number [1]. The four compounds, N-(p-n-methoxy/ethoxybenzylidene)-p-n-dodecyl and tetradecyl anilines, 1O.12, 1O.14, 2O.12, and 2O.14, possess short alkoxy chain with long alkyl chain. The density studies are carried out on all the compounds to understand the nature of the transition and pretransitional effects.

As a part of ongoing research program on thermodynamic, acoustic properties of LC, the authors here report the results of the density measurements on four LC, viz., N-(p-n-methoxy/ethoxybenzylidene)-p-n-dodecyl and tetradecylanilines, 1O12, 1O.14, 2O.12,

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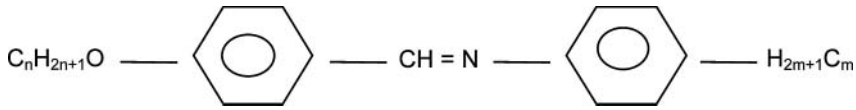
and 2O.14. Further, the thermodynamic parameters such as Moelwyn-Hughes parameter, reduced volume, reduced compressibility, isothermal, isochoric, and isobaric Gruneisan parameters [9], etc., are estimated using the thermal expansion coefficient derived from the density results. The derived parameters offer a convenient method for the study of thermodynamic properties of compounds, not easily obtained by other means. The results are compared with the data available on other LC compounds.

Experimental

The liquid crystalline compounds used in the present study are synthesized according to standard procedures [4]. The synthesized compounds are subjected to repeated crystallization in methanol/ethanol until the transition temperatures are in agreement with the reported data. A U-shaped bi-capillary pyknometer in conjunction with the cathetometer was used for the density measurements. The absolute error in the measurement of density is $\pm 10^{-4}$ g*cm^{-3}. The cooling rate during the measurement was 0.5°C h^{-1}. The molecular structure of the compounds is given below.



with $n = 1$ and m =12—N-(*p*-*n*-methoxybenzylidene)-*p*-*n*-dodecyl aniline, 1O.12,
with $n = 2$ and $m = 12$—N-(*p*-*n*-ethoxybenzylidene)-*p*-*n*-dodecyl aniline, 2O.12,
with $n = 1$ and $m = 14$—N-(*p*-*n*-methoxybenzylidene)-*p*-*n*-tetradecyl aniline, 1O.14, and
with $n = 2$ and $m = 14$—N-(*p*-*n*-ethoxybenzylidene)-*p*-*n*-dodecyl aniline, 2O.14.

Thermodynamic Parameters

The theoretical procedure for the evaluation of different thermoacoustic parameters using α is described below [9]. All the parameters are interrelated through one and only one parameter, viz., the Moelwyn-Hughes parameter C_1 [9]. The other parameters related to C_1 (which is related to α) are given below.

The Moelwyn-Hughes parameter C_1 and reduced molar volume $V^{\sim}$ may be expressed as

$$C_1 = \left(\frac{13}{3}\right) + \left(\frac{1}{\alpha T}\right) + \left(\frac{4\alpha T}{3}\right), \tag{1}$$

$$V^{\sim} = \left\{ \frac{\left(\frac{\alpha \mathrm{T}}{3}\right)}{(1+\alpha \mathrm{T})} + 1 \right\}^3. \tag{2}$$

Bayer's nonlinear asymmetry parameter *B*/*A* is given by

$$B/A = C_1 - 1. \tag{3}$$

Sharma and Reddy demonstrated the following interrelationship between n and C_1 (magnitude of repulsive forces) as

$$n = (3C_1 - 12). \tag{4}$$

The isothermal Grunesien parameter is $\Gamma_{ith} = (C_1 - 1)/2$, (5)

and the isochoric Grunesien parameter is $\Gamma_{ich} = (E - F)/F$, (6)

where $E = [2 + (\alpha T)^{-1}(-2\alpha)V^{\sim C1}]^{-1}$, (7)

and $F = -2\alpha T$. (8)

Using the values of Γ_{ith} and Γ_{ich}, the isobaric Gruneisan parameter Γ_{iba} can be evaluated from the equation

$$\Gamma_{ith} = \Gamma_{iba} + \Gamma_{ich}. \quad (9)$$

The isothermal Anderson-Gruneisan parameter δ is known to be an important parameter in the theory of temperature dependence of bulk modulus in solids. It is defined as

$$\delta = 2\ \Gamma_{iba}. \quad (10)$$

However, the Anderson-Gruneisan parameter δ is distinguished from the Moelwyn-Hughes parameter as

$$\theta = (C_1 - \delta) = 2(\Gamma_{ith} - \Gamma_{iba}). \quad (11)$$

Further, the available volume V_a can also be estimated [9] from *B*/*A* also as

$$V_a = V(1/2(B/A) + 1)^{-1}. \quad (12)$$

Results and Discussion

Dilatometric Studies

The compounds 1O.12, 2O.12, 1O.14 exhibit single phase variant nematic while the compound 2O.14 exhibits nematic and SmA phases. The phase variants and the transition temperatures are given in Table 1 and are in good agreement with the reported values [1] except in the case of 2O.14, where the density measurements exhibited the shrinkage of nematic thermal range.

The density, ρ (gm*cm^{-3}) increases with the decrease of temperature. However, at the phase interface it exhibits step increase before stabilizing in the equilibrium phase as expected. The density jumps, the molar volume at T_{IN+5}, the density slopes in different phases, and the thermal expansion coefficient maxima at the phase transformation are estimated from the density data. The percentage of density jumps, the density slopes, and

Table 1. The transition temperatures (°C) across different phase transitions in 1O.12, 2O.12, 1O.14, and 2O.14

Compound	Phase variant	Isotropic–Nematic	Nematic–smectic-A	Nematic/smectic-A–crystal
1O.12	N	59.9	–	54.5
2O.12	N	76.2	–	49.0
1O.14	N	61.3	–	55.2
2O.14	NA	70.2	58.4	–

Table 2. The percentage of density jumps, the thermal expansion coefficient, and the slopes of density in different phases of 1O.12, 2O.12,1O.14, and 2O.14

	I-N transition		N-A Transition				
Compound	% of $\Delta\rho/\rho$	α ($10^{-4\circ}$C)	% of $\Delta\rho/\rho$	α ($10^{-4\circ}$C)	$(d\rho/dT)_{Iso}$ $\times 10^{-4}$	$(d\rho/dT)_{N}$ $\times 10^{-4}$	$(d\rho/dT)_{A}$ $\times 10^{-4}$
1O.12	0.35	74.70	—	—	8.8	12.6	—
2O.12	0.16	37.80	—	—	7.9	11.2	—
1O.14	0.07	34.00	—	—	6.8	8.0	—
2O.14	0.31	60.00	0.11	49.40	8.9	10.0	10.7

the thermal expansion coefficient maxima for all these compounds are presented in Table 2. The temperature variation of density ρ and thermal expansion coefficient α for all the compounds is shown in Figs. 1–4.

Isotropic–Nematic (IN) Transition

The IN transition is accompanied by density jumps of 0.35, 0.16, 0.07, and 0.31 and thermal expansion coefficient maxima of 74.7, 37.8, 34.0, and 60.0 $\times$ $10^{-4\circ}$C for the compounds 1O.12, 2O.12, 1O.14, and 2O.14, respectively. The values suggest the nature of this transition as first order. However, the density jump values in the case of 2O.12 and 1O.14 are small compared to the values obtained for other compounds which exhibit the same transition. Generally, the density jumps are of the order of 0.20–0.40. However, the exhibition of small values in some compounds is common.

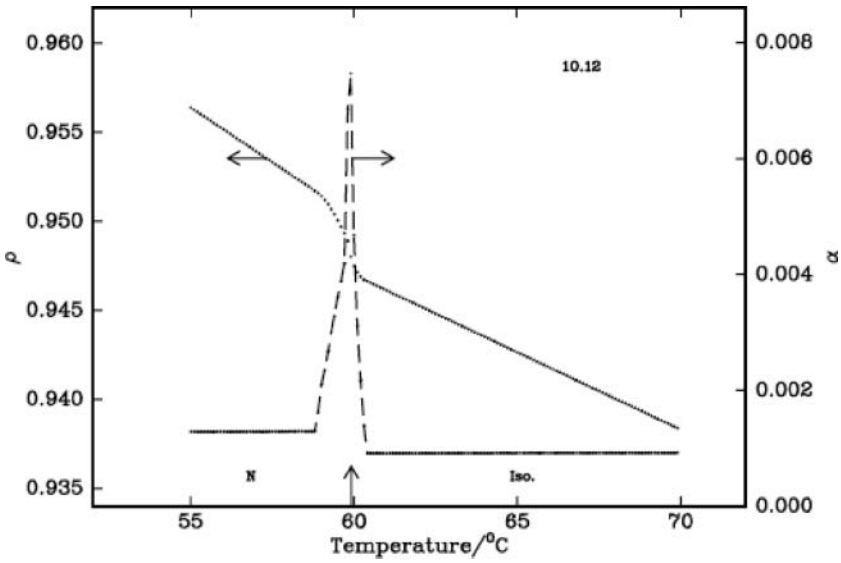


Figure 1. Variation density, ρ, and thermal expansion coefficient, α, with temperature in 1O.12.

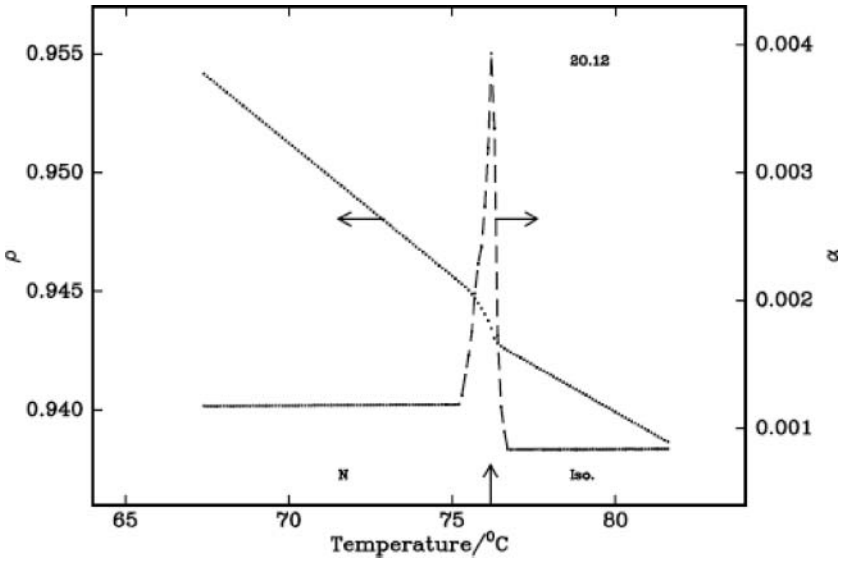


Figure 2. Variation density, ρ, and thermal expansion coefficient, α, with temperature in 2O.12.

Nematic–Smectic-A (N-SmA) Phase Transitions

The nematic to SmA transition is observed only in 2O.14, the density jump and the thermal expansion coefficient maxima suggest it to be first order. The nO.m compounds are a classic example to exhibit tricritical point (TCP), where a second order transition transforms into first order depending on the value of McMillan parameter, and the alkoxy/alkyl chain length. The McMillan [10] theory predicts a first order transition for $M = (T_{NA}/T_{NI}) = 0.87$. It

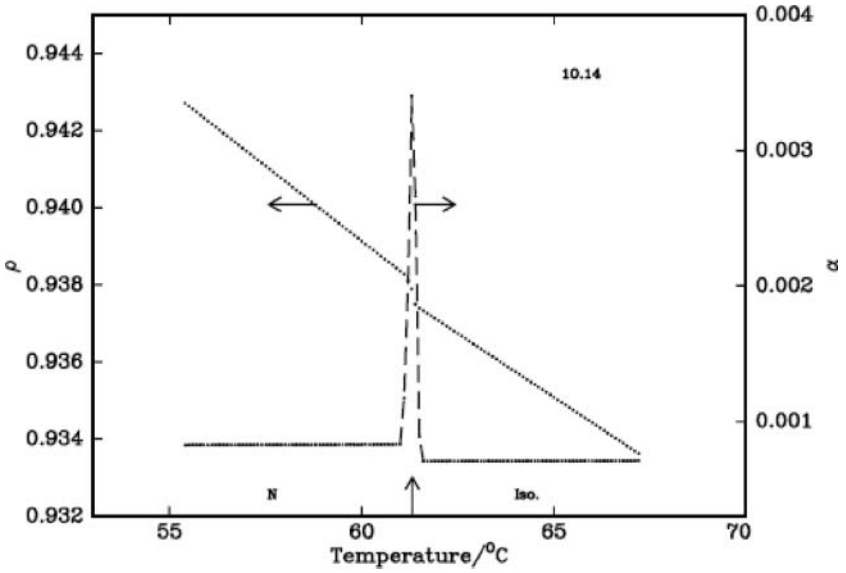


Figure 3. Variation density, ρ, and thermal expansion coefficient, α, with temperature in 1O.14.

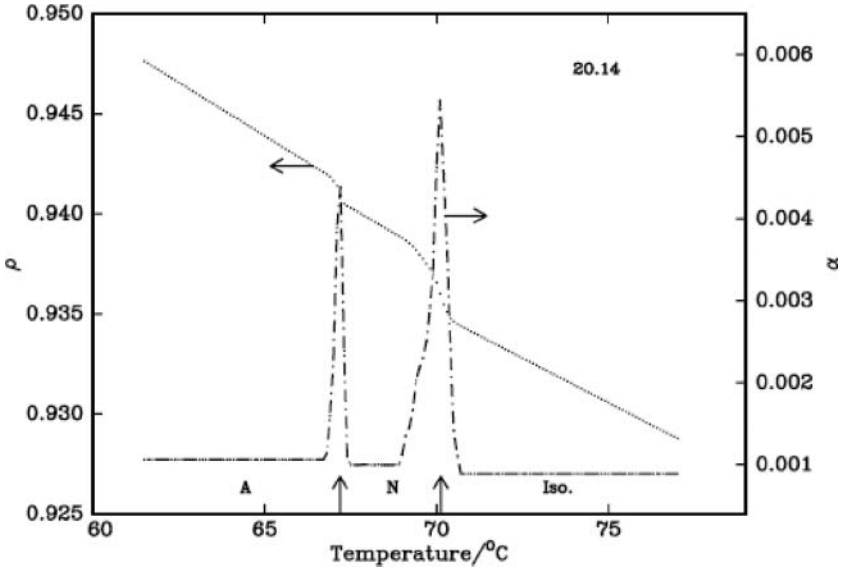


Figure 4. Variation density, ρ, and thermal expansion coefficient, α, with temperature in 2O.14.

must be pointed out that the McMillan predicted heat of transition involves pretransitional effects and has to be modified for true total enthalpy. Hence, the above predicted value is low compared to the value arrived at by different experimental techniques which is $\geq$0.959 [11]. The systematic studies on nO.m compounds found a possible TCP (where the second order transition changes to first order) to exist [11] around the value given above. The McMillan parameter ($M = T_{NA}/T_{NI}$) value obtained for this compound is 0.966 which is $>$0.959.

The value of slope of the variation of density with temperature in the equilibrium SmA phase $(d\rho/dT)_A$ in 2O.14 is 10.7×10^{-4} g cm^{-3}°C. The higher slope of density variation with temperature ($d\rho/dT$) in SmA phase than the slope of density in nematic/isotropic phase suggests an additional packing with positional and translational order. The salient features observed through this study are

1. As expected the IN transition is found to be first order and the density jumps agree with the literature data. However, in the case of 1O.14 the jump observed is low compared to many compounds in this series, and
2. The N-SmA transition in the 2O.14 is found to be first order with McMillan parameter 0.966 and is $>$0.959, a value reported as TCP in nO.m compounds [11].

Thermodynamic Parameters

Employing the relevant equations, the thermoacoustic parameters, Aderson-Grunesian parameter θ, isothermal Grunesian parameter Γ_{itc}, isobaric Grunesian parameter Γ_{iba}, isochoric Grunesian parameter Γ_{ich}, the reduced bulk modulus $B^{\sim}$, isothermal Aderson-Grunesian parameter δ, magnitude of repulsive forces n, available volume V_a are calculated. The variation of all these parameters with temperature for the compound 1O.14 are given in Tables 3 and 4 depicts the variation of parameters in isotropic and liquid crystalline phases in all the compounds.

Table 3. Variation of Moelwyn-Hughes parameter, $C_{1,}$ reduced molar volume, $V,^{\sim}$ reduced compressibility, $V^{\sim C1}$, reduced bulk modulus, $B^{\sim}$, isothermal Grunesian parameter, Γ_{ith}, isochoric Grunesian parameter, Γ_{ich}, isobaric Grunesian parameter, Γ_{iba}, isothermal–Anderson Grunesian parameter, δ, magnitude of repulsive force, n, available volume,V_a in 1O.14

$T^{O}C$	C_1	$V^{\sim}$	$V^{\sim C1}$	$B^{\sim}$	Γ_{ith}	Γ_{ich}	Γ_{iba}	δ	θ	n	Y	Va
64.7	8.835	1.205	5.212	0.192	3.917	2.063	1.854	3.709	5.126	14.504	0.100	88.50
64.1	8.845	1.205	5.204	0.192	3.923	2.066	1.857	3.713	5.132	14.535	0.100	88.37
63.5	8.854	1.205	5.198	0.192	3.927	2.068	1.859	3.717	5.136	14.561	0.100	88.25
63.0	8.861	1.204	5.192	0.193	3.930	2.070	1.860	3.720	5.140	14.582	0.100	88.16
62.6	8.865	1.204	5.189	0.193	3.932	2.071	1.861	3.722	5.142	14.594	0.100	88.10
62.5	8.868	1.204	5.187	0.193	3.934	2.072	1.862	3.724	5.144	14.604	0.100	88.06
62.1	8.874	1.204	5.183	0.193	3.937	2.074	1.863	3.726	5.147	14.621	0.100	87.99
61.8	8.878	1.204	5.180	0.193	3.939	2.075	1.864	3.728	5.150	14.634	0.100	87.93
61.6	8.881	1.203	5.177	0.193	3.940	2.076	1.865	3.729	5.151	14.642	0.100	87.89
61.5	8.095	1.247	5.962	0.168	3.547	1.858	1.690	3.380	4.715	12.285	0.099	95.48
61.3 (I-N)	6.726	1.632	26.958	0.037	2.863	1.239	1.624	3.247	3.478	8.177	0.086	112.34
61.1	7.407	1.308	7.326	0.136	3.203	1.653	1.550	3.101	4.306	10.221	0.096	103.19
60.9	8.311	1.233	5.697	0.176	3.656	1.919	1.737	3.474	4.837	12.933	0.099	93.16
60.6	8.315	1.233	5.693	0.176	3.657	1.920	1.738	3.475	4.839	12.944	0.099	93.10
60.3	8.318	1.232	5.689	0.176	3.659	1.921	1.738	3.477	4.841	12.955	0.099	93.04
60.0	8.322	1.232	5.685	0.176	3.661	1.922	1.739	3.479	4.844	12.967	0.099	92.98
59.6	8.327	1.232	5.679	0.176	3.664	1.923	1.740	3.481	4.846	12.982	0.099	92.90

(Continued on next page)

Table 3. Variation of Moelwyn-Hughes parameter, C_1, reduced molar volume, $V^{\sim}$ reduced compressibility, $V^{\sim C1}$, reduced bulk modulus, $B^{\sim}$, isothermal Grunesian parameter, Γ_{ith}, isochoric Grunesian parameter, Γ_{ich}, isobaric Grunesian parameter, Γ_{iba}, isothermal–Anderson Grunesian parameter, δ, magnitude of repulsive force, n, available volume, V_a in 1O.14 *(Continued)*

$T^{O}C$	C_1	$V^{\sim}$	$V^{\sim C1}$	$B^{\sim}$	Γ_{ith}	Γ_{ich}	Γ_{iba}	δ	θ	n	Y	Va
59.3	8.332	1.232	5.674	0.176	3.666	1.925	1.741	3.483	4.849	12.996	0.099	92.82
59.0	8.335	1.231	5.671	0.176	3.667	1.925	1.742	3.484	4.851	13.004	0.099	92.78
58.7	8.340	1.231	5.665	0.177	3.670	1.927	1.743	3.486	4.854	13.019	0.099	92.70
58.5	8.340	1.231	5.665	0.177	3.670	1.927	1.743	3.486	4.854	13.019	0.099	92.69
58.4	8.342	1.231	5.662	0.177	3.671	1.928	1.744	3.487	4.855	13.027	0.099	92.65
58.1	8.347	1.231	5.657	0.177	3.674	1.929	1.745	3.489	4.858	13.042	0.099	92.58
57.7	8.351	1.230	5.653	0.177	3.676	1.930	1.746	3.491	4.860	13.053	0.099	92.51
57.3	8.355	1.230	5.649	0.177	3.677	1.931	1.746	3.493	4.862	13.065	0.099	92.45
57.1	8.359	1.230	5.645	0.177	3.679	1.932	1.747	3.494	4.864	13.076	0.099	92.39
56.8	8.361	1.230	5.642	0.177	3.681	1.933	1.748	3.495	4.866	13.084	0.099	92.34
56.5	8.366	1.230	5.636	0.177	3.683	1.934	1.749	3.498	4.869	13.099	0.099	92.27
56.3	8.369	1.229	5.634	0.178	3.684	1.935	1.749	3.499	4.870	13.107	0.099	92.23
56.1	8.372	1.229	5.631	0.178	3.686	1.936	1.750	3.500	4.872	13.115	0.099	92.19
55.9	8.374	1.229	5.628	0.178	3.687	1.937	1.751	3.501	4.873	13.122	0.099	92.15

Table 4. Variation of Moelwyn-Hughes parameter, $C_{1,}$ reduced molar volume, $V,^{\sim}$ reduced compressibility, $V^{\sim C1}$, reduced bulk modulus, $B^{\sim}$, isothermal Grunesian parameter, Γ_{ith}, isochoric Grunesian parameter, Γ_{ich}, isobaric Grunesian parameter, Γ_{iba}, isothermal–Anderson Grunesian parameter, δ, magnitude of repulsive force, n, available volume, V_a in different liquid crystalline phases of 1O.12, 2O.12,1O.14, and 2O.14

Compound	$T^{O}C$	C_1	$V^{\sim}$	$V^{\sim C1}$	$B^{\sim}$	Γ_{ith}	Γ_{ich}	Γ_{iba}	δ	θ	n	Y	Va
1O.12 Iso	64.5	7.961	1.256	6.154	0.162	3.481	1.819	1.661	3.323	4.638	11.883	0.098	89.69
N	58.0	7.239	1.330	7.897	0.127	3.120	1.599	1.520	3.041	4.198	9.717	0.095	96.57
2O.12 Iso	80.0	8.248	1.237	5.769	0.173	3.624	1.900	1.724	3.448	4.800	12.74	0.099	90.63
N	74.8	7.381	1.312	7.405	0.135	3.190	1.644	1.546	3.092	4.289	10.14	0.096	99.38
1O.14 Iso	64.1	8.845	1.205	5.204	0.192	3.923	2.066	1.857	3.713	5.132	14.535	0.100	88.37
N	57.3	8.355	1.230	5.649	0.177	3.677	1.931	1.746	3.493	4.862	13.065	0.099	92.45
2O.14 Iso	74.0	7.782	1.271	6.455	0.155	3.391	1.766	1.624	3.249	4.533	11.345	0.878	102.94
N	68.0	7.573	1.290	6.889	0.145	3.286	1.704	1.582	3.165	4.408	10.718	0.871	104.51

Table 5. Density jumps, the order parameter jumps due to Maier and Saupe and from birefringence of all the four compounds

Compound	Density jumps	Order parameter jumps[a]	Order parameter jumps[b]
1O.12	0.35	0.422	0.393
2O.12	0.16	0.398	0.445
1O.14	0.07	0.445	0.270
2O.14	0.31	0.426	0.369

[a]from Maier and Saupe equation.
[b]from birefringence.

The salient features observed from the tables are

1. The available volume V_a obtained from nonlinearity parameter *B*/*A* is well in agreement with the data reported earlier [12] with exhibiting a higher value in liquid crystalline phase than in isotropic phase.
2. It is observed that the magnitude of these parameters show a decreasing trend with the decrease of temperature and the values are lower in liquid crystalline phases.
3. The magnitude of repulsive exponent exhibits a high value in isotropic phase than in the liquid crystalline phase with a variation around 7–15 which is evident from Tables 3 and 4, whereas the parameter n varies from 16 to 56 in polymers [13].
4. The isobaric Gruneisan parameter is found to be exhibiting low values than that of isothermal Gruneisan parameter [14].

Density and Order Parameter Jumps

The density jumps estimated are given in Table 2 for the IN transition. However, the order parameter variation with the temperature can be obtained by different techniques. Maier and Saupe [15] arrived at the variation of order parameter with the temperature with the following relation using density data:

$$S = \{1 - 0.98(T/T_{\mathrm{IN}})(V_n/V_{n(\mathrm{IN})})^2\}^{0.22},$$

where T and T_{IN} are the temperature and the isotropic nematic transition temperature, respectively, while V_n and $V_{n(\mathrm{IN})}$ are the molar volume at a temperature T and at IN transition. However, the same can be obtained from birefringence studies in conjunction with the density results also. Table 5 exhibits the density and the order parameter jumps at the IN transition derived from different methods. The table reveals that the order parameter starts from generally around 0.4 while it is not the case with that calculated from birefringence. More details of the order parameter estimation from polarizability data and its variation with temperature are described in detail in the reference [16].

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