CHAPTER-IV

OPTICAL BIREFRINGENCE STUDIES ON

I) p-n-BUTOXYBENZYLIDENE-p'-n-BUTYLANILINE (BBBA in short).

II) p-n-PROPOXYBENZYLIDENE-p'-n-BUTYLANILINE (PBBA in short).

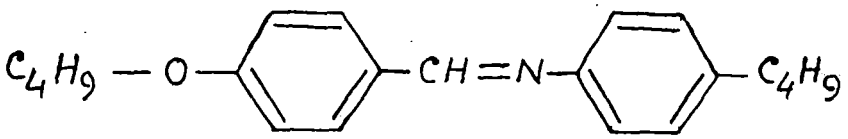
III) ANISYLIDENE-p-AMINOPHENYL ACETATE (APAPA in short).

4.1 Introduction

In this chapter I report refractive index measurements of three Schiff's base compounds. Due to paucity of the samples BBBA and PBBA and chemical decomposition of APAPA I could not measure the diamagnetic susceptibilities of these compounds. X-ray diffraction studies on these compounds had been carried out by my coworkers in our laboratory. They have calculated the orientational order parameters, apparent molecular length, average intermolecular distance and angular part of the mean field potential in the nematic phase of the aligned samples. They have also identified different phases specially of BBBA.

The names, structural formulae and transition temperatures of the three compounds are as follows :

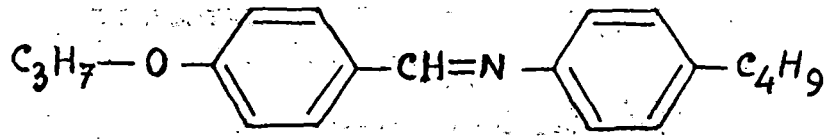
(i) p-n-Butoxybenzylidene-p'-n-butylaniline (BBBA in short)[1]



Transition temperatures,

$$\text{Solid} \underset{\sim 8^{\circ}C}{\rightleftharpoons} S_G(S_{G'}) \underset{41^{\circ}C}{\rightleftharpoons} S_B \underset{45^{\circ}C}{\rightleftharpoons} S_A \underset{45.5^{\circ}C}{\rightleftharpoons} N_C \underset{59^{\circ}C}{\rightleftharpoons} N_O \underset{74^{\circ}C}{\rightleftharpoons} I$$

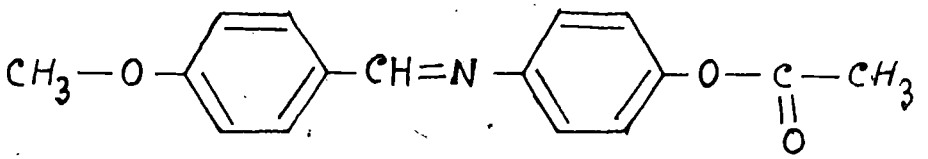
(ii) p-n-Propoxybenzylidene-p'-n-butylaniline (PBBA in short)[2]



Transition temperatures,

Solid $\underset{\sim 18.5^{\circ}C}{\overset{40.5^{\circ}C}{\rightleftharpoons}}$ Nematic $\overset{57.5^{\circ}C}{\rightleftharpoons}$ Isotropic

(iii) Anisylidene-p-aminophenyl acetate (APAPA in short)[3]



Transition temperatures,

Solid $\underset{\sim 74^{\circ}C}{\overset{82^{\circ}C}{\rightleftharpoons}}$ Nematic $\overset{110^{\circ}C}{\rightleftharpoons}$ Isotropic

The above three compounds were supplied by Late Prof. M.Wada of Tohoku University, Japan[4] in pure form. So I have performed experiments without further purification.

Mesomorphism of Schiff's base compounds is of much interest and a number of studies have been made on this series[5-13]. Orientational order parameters of EBBA, BBBA and PBBA were determined by Kirov et al[14] from infrared dichroism. Fujimura et al[15] measured the degree of order of these compounds by studying the NMR spectra. The compound BBBA was used by Rao and Murty[16] for ultrasonic study. Goodby et al[17] studied this compound by optical microscopy and miscibility method. The compound APAPA was studied extensively by several workers[4, 18-23].

4.2 Experimental methods

I checked the transition temperatures of these three compounds and no further purification of the samples

was felt necessary, since the observed transition temperatures agreed with the literature values. The compounds were examined using polarizing microscope with Metler hot stage (model FP 80/82). Observations were performed under crossed polarizers with a magnification 150X. Since Schiff's base compounds are very sensitive to atmospheric moisture, care was taken to minimise the exposure to atmosphere.

Refractive indices (n_o , n_e) for ordinary and extra-ordinary rays were measured within $\pm$ 0.001 with the help of thin prism with a refractive angle of about 1^o - 2^o. Refractive indices were measured for four different wavelengths for BBBA and PBBA but for APAPA only two wavelengths were used. The details of these measurements are given in chapter II sec. 2.3.2 and the same procedure was followed for obtaining mono-domain samples. I measured the density values at different temperatures for BBBA and PBBA with an accuracy of 1%, whereas the density values of APAPA at different temperatures were taken from the paper by Leenhouts et al[23]. From the refractive index data and density values I have calculated the principal polarizabilities (α_o , α_e) of these compounds using Vuks' formula and Neugebauer's relations which are given in chapter II.

4.3 Results and discussions

The values of experimental refractive indices (n_o , n_e) are tabulated in Tables 4.1-4.3 for the three compounds. The variation of n_o and n_e with temperature for

Table -4.1

Refractive indices (n_o, n_e) at different temperatures of BBBA.

Temp. (°C)	λ = 6907 Å		λ = 5780 Å		λ = 5461 Å		λ = 4358 Å	
	n_o	n_e	n_o	n_e	n_o	n_e	n_o	n_e
35.9	1.497	——	1.507	——	1.512	——	1.532	——
37.8	1.497	——	1.507	——	1.512	——	1.532	——
39.8	1.497	——	1.507	——	1.512	——	1.532	——
42.3	1.502	1.746	1.512	1.761	1.517	1.771	1.537	1.860
44.2	1.502	1.736	1.512	1.751	1.517	1.761	1.537	1.848
46	1.492	1.721	1.502	1.736	1.507	1.746	1.527	1.830
52.8	1.492	1.716	1.502	1.731	1.507	1.741	1.527	1.822
57.3	1.497	1.711	1.507	1.726	1.512	1.736	1.532	1.814
62	1.502	1.701	1.512	1.716	1.517	1.726	1.537	1.803
65.5	1.507	1.691	1.517	1.706	1.522	1.716	1.543	1.790
70	1.512	1.676	1.522	1.691	1.527	1.701	1.552	1.773
72.5	1.519	1.666	1.529	1.681	1.534	1.691	1.562	1.762
75(Iso.)	1.577		1.587		1.592		1.637	
77.5	1.572		1.582		1.587		1.632	
80	1.572		1.582		1.587		1.632	

Table -4.2

Refractive indices (n_o, n_e) at different temperatures of PBBA

Temp. (°C)	λ = 6907 Å		λ = 5780 Å		λ = 5461 Å		λ = 4358 Å	
	n_o	n_e	n_o	n_e	n_o	n_e	n_o	n_e
27.7	1.538	1.768	1.550	1.779	1.560	1.792	1.588	1.878
32	1.538	1-764	1-550	1.775	1.560	1.788	1.588	1.872
36	1.540	1.758	1.552	1.770	1.562	1.783	1.590	1.866
40	1.542	1.750	1.554	1.765	1.562	1-776	1.592	1.860
44	1.544	1.744	1.558	1.759	1.566	1.769	1.594	1.850
48	1.550	1.736	1.562	1.750	1.570	1.761	1.597	1.839
52	1.555	1.727	1.567	1.740	1.574	1.750	1.602	1.826
56	1.561	1.713	1.573	1.726	1.580	1.735	1.607	1.805
57	1.564	1.707	1.576	1.720	1.582	1.728	1.611	1.795
57.5(Iso)	1.590		1.602		1.612		1.660	
60	1.588		1.600		1.610		1.658	
62	1.586		1.598		1.608		1.656	

Table -4.3

Density (ρ), refractive indices (n_o, n_e), polarizabilities (α_o, α_e) and order parameters ($\langle P_2 \rangle$) of APAPA

Temp. (°C)	ρ (gm/c.c)	λ = 5780 Å					λ = 5461 Å				
		n_o	n_e	α_o	α_e	$\langle P_2 \rangle$	n_o	n_e	α_o	α_e	$\langle P_2 \rangle$
75	1.1360	1.510	1.848	25.80	48.68	.6288	1.525	1.868	26.41	49.60	.6283
80	1.1314	1.510	1.840	25.96	48.39	.6163	1.525	1.860	26.57	49.30	.6160
85	1.1265	1.510	1.830	26.15	47.98	.6000	1.525	1.850	26.76	48.90	.6002
90	1.1212	1.515	1.820	26.59	47.48	.5740	1.530	1.840	27.41	48.40	.5744
95	1.1155	1.520	1.810	27.05	46.99	.5493	1.535	1.830	27.67	47.92	.5488
100	1.1096	1.530	1.790	27.85	45.78	.4928	1.545	1.810	28.47	46.72	.4925
105	1.1033	1.545	1.760	29.00	43.86	.4084	1.560	1.780	29.63	44.81	.4115
110 (Iso.)	1.095	1.620					1.635				

α_o and α_e are in unit 10^{-24} cm^3. and calculated from Vuks' formula.

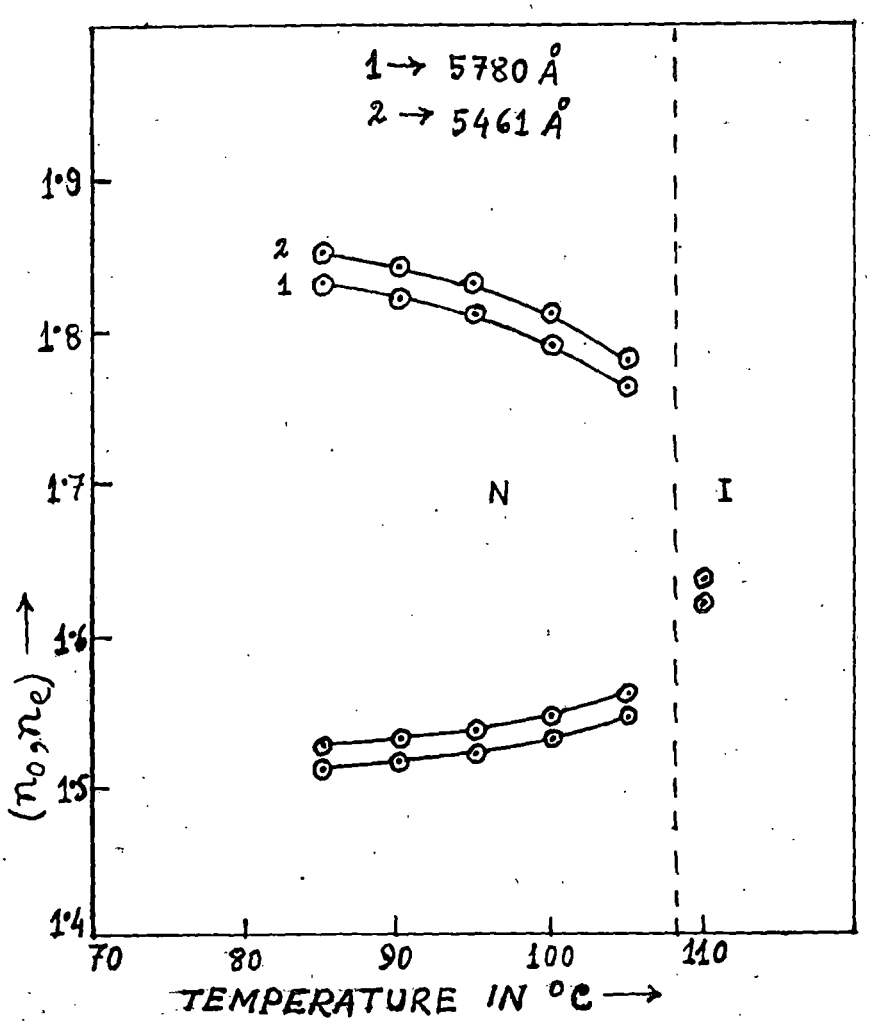


Fig.4.3 Variation of refractive indices (n_o,n_e) at different temperatures of the sample APAPA.

the three compounds are displayed in figures 4.1-4.3. I have calculated the optical anisotropy ($\Delta n = n_e - n_o$) at different wavelengths for BBBA and PBBA. The variations of Δn with wavelength are shown in figures 4.4(a) and 4.4(b). It is clear from these two figures that Δn for blue light is much greater than those for the other three colours. This type of variations of optical anisotropy with wavelength were also found by Blinov et al[24] for PAA and MBBA. Different phase changes in BBBA is seen clearly from the figure 4.1. I failed to take the refractive index data in smectic A phase (within narrow temperature range, 45°C and 45.5°C), because the temperature could be controlled only within ± 0.5°C. The figure 4.1 does not show any discontinuity at 59°C where the compound BBBA undergoes a phase change from cybotactic nematic (N_c) to ordinary nematic (N_o)[1, 16] phase.

The principal polarizabilities (α_o, α_e) calculated by using Vuks' isotropic model and density (ρ) values are given in the Tables 4.4-4.5 for BBBA and PBBA and for APAPA these values are included in Table 4.3. The results of the calculated polarizability values (α_o, α_e) using Neugebauer's relations (anisotropic model) are arranged in the Tables 4.6-4.8 for these three compounds.

Finally I have calculated the orientational order parameter values $\langle P_2 \rangle$ for the three compounds adopting the Haller's extrapolation procedure. The order parameters computed using isotropic local field model (Vuks' approach) and the anisotropic local field model (Neugebauer's approach)

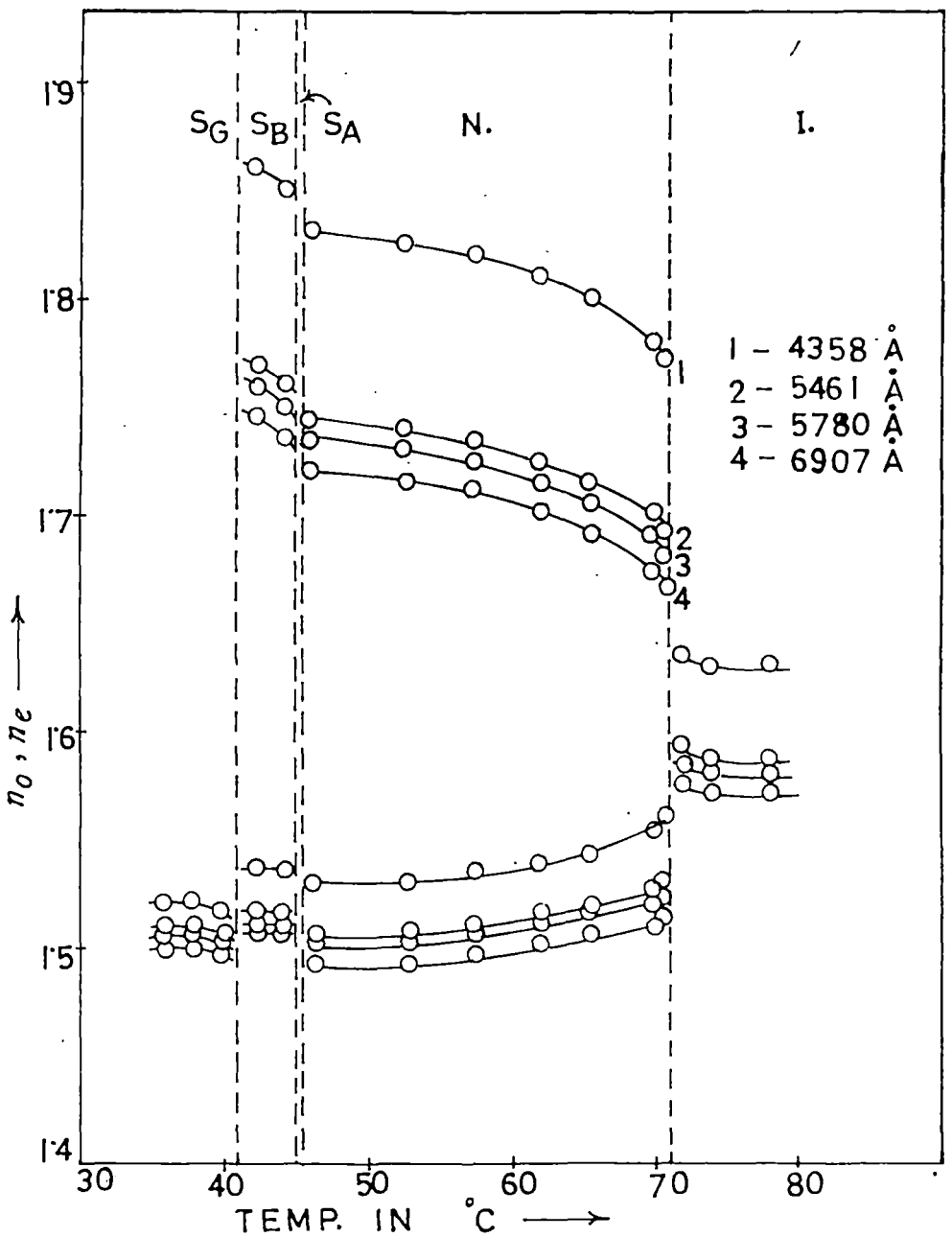


Fig.4.1 Values of refractive indices (n_o,n_e) at different temperatures of the sample BBBA.

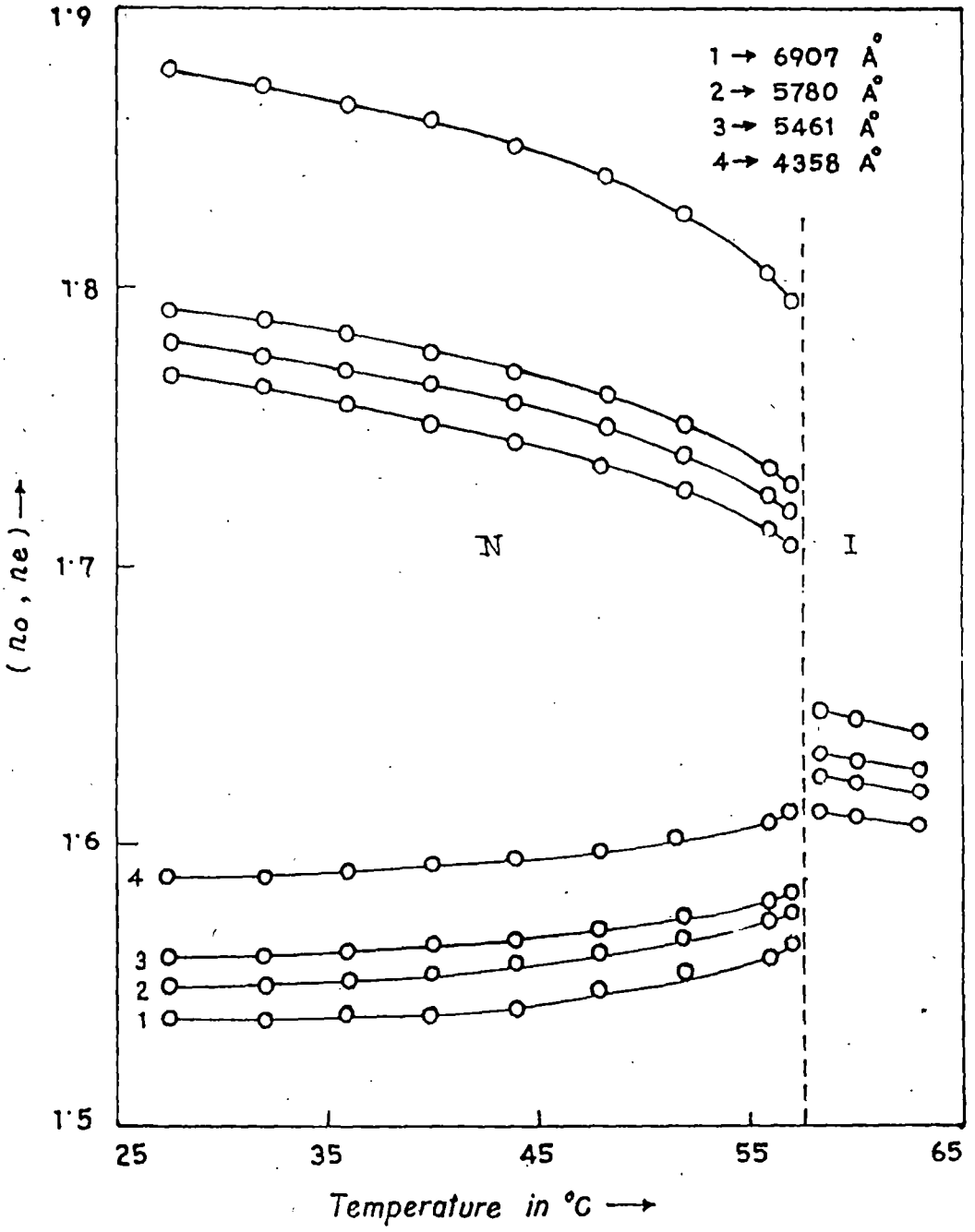


Fig.4.2 Variation of refractive indices (n_o,n_e) at different temperatures of the sample PBBA.

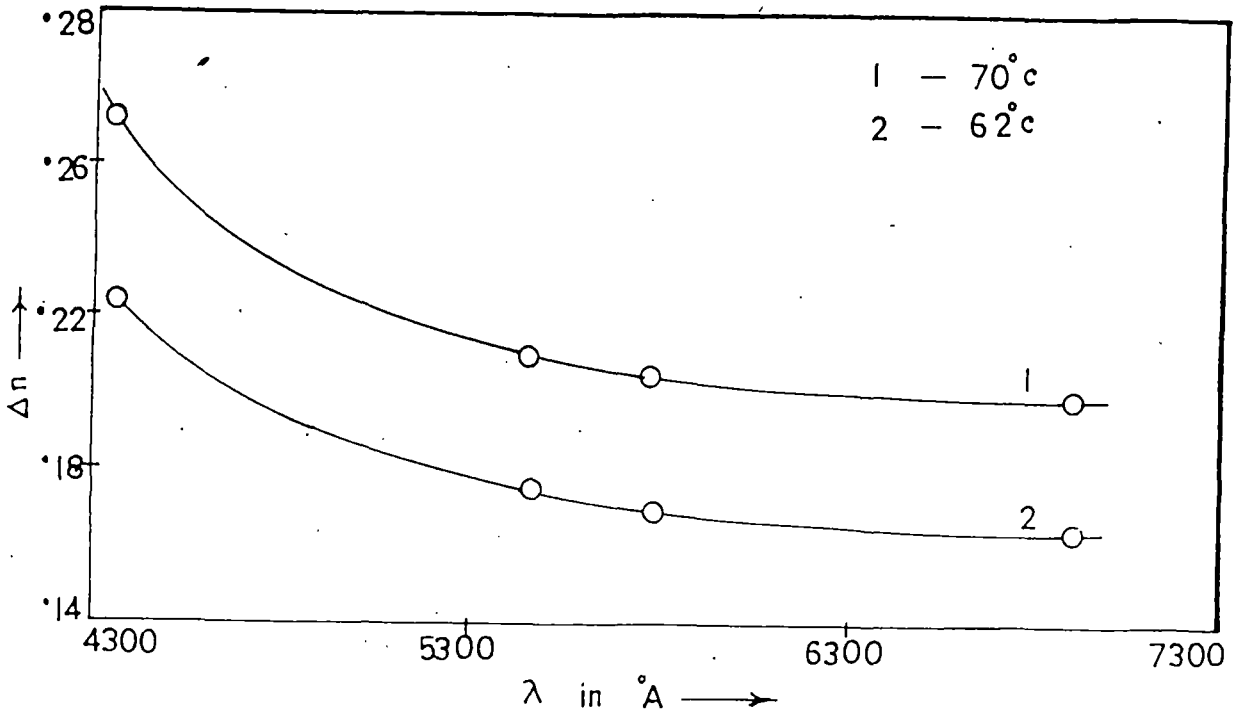


Fig.4.4a Variation of Δ n with wavelength of BBBA.

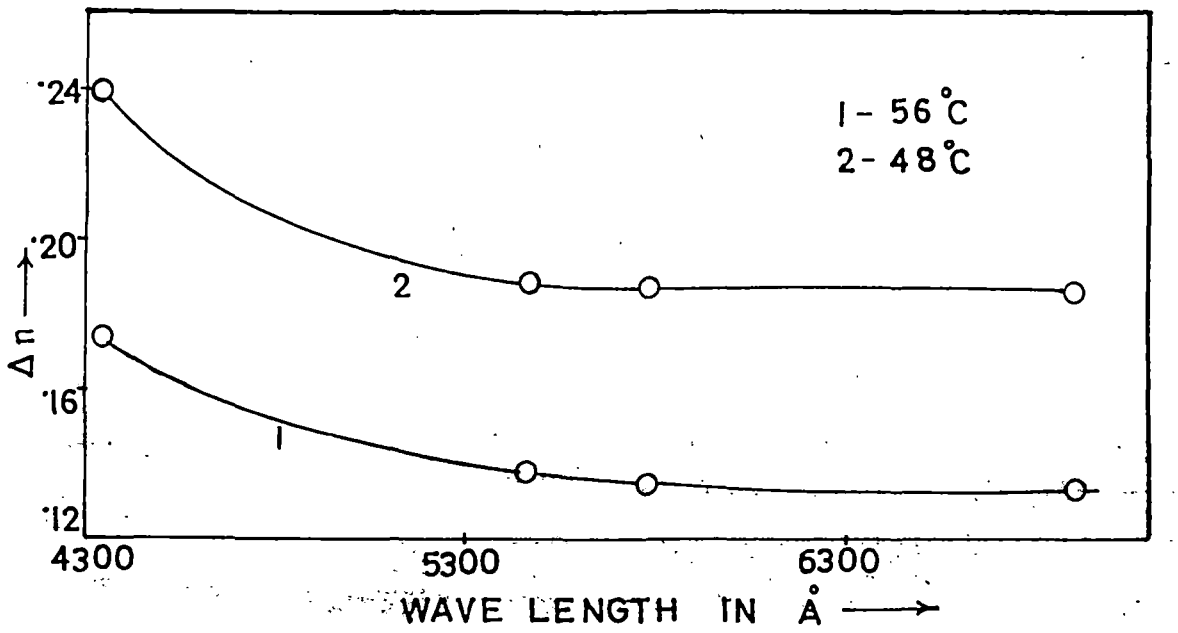


Fig.4.4b Variation of Δn with wavelength of PBBA.

Table-4.4

Density (ρ), polarizability (α) and orientational order parameter ($\langle P_2 \rangle$) of BBBA.

$\lambda \rightarrow$	6907 Å				5780 Å			5461 Å			4358 Å		
T°C	ρ in gm/c.c.	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$
42.3	1.0165	33.50	54.63	.6345	34.02	55.57	.6350	34.25	56.24	.6341	34.74	62.71	.6365
44.2	1.0160	33.60	53.37	.6085	34.12	54.81	.6095	34.36	55.48	.6092	34.86	61.30	.6129
46.0	1.0110	33.24	53.18	.5937	33.77	54.14	.6001	34.01	54.81	.6000	34.56	60.95	.6005
52.0	0.9960	33.78	53.53	.5943	34.32	54.55	.5959	34.56	55.23	.5961	35.15	61.23	.5933
57.3	0.9360	34.51	53.59	.5730	35.05	54.57	.5751	35.30	55.25	.5759	35.91	61.07	.5724
62.0	0.9750	35.33	53.25	.5332	35.88	54.24	.5411	36.13	54.94	.5427	36.76	60.72	.5453
65.5	0.9590	35.93	52.64	.5001	36.53	53.64	.5033	36.78	54.34	.5023	37.51	59.31	.5033
70.0	0.9600	36.31	51.77	.4492	37.36	52.78	.4540	37.62	53.43	.4576	38.64	58.30	.4536
72.5	0.9450	37.98	51.58	.4033	38.55	52.61	.4141	38.80	53.32	.4138	40.05	58.55	.4203
75.0	0.9295 (Iso.)												

α_o and α_e are in units $10^{-24} cm^3$ and calculated from Vuks' formula.

Table: 4.5

Density (ρ), polarizability (α) and orientational order parameter ($\langle P_2 \rangle$) of PBBA (Vuks' approach)

$\lambda \rightarrow$ Temp. (°C)	Density (ρ) (gm/cm^3)	6907 Å			5780 Å			5461 Å			4358 Å		
		α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$
27.7	1.165	29.69	46.22	.6191	30.24	46.68	.6157	30.68	47.32	.6163	31.46	52.25	.6161
32	1.163	29.78	46.06	.6096	30.34	46.52	.6062	30.77	47.16	.6069	31.58	51.97	.6046
36	1.158	30.04	45.79	.5891	30.59	46.32	.5891	31.03	46.96	.5900	31.84	51.72	.5893
40	1.155	30.30	45.43	.5668	30.84	46.10	.5716	31.29	46.61	.5674	32.10	51.45	.5737
44	1.151	30.55	45.07	.5437	31.20	45.77	.5458	31.55	46.26	.5447	32.39	50.92	.5494
48	1.149	31.06	44.60	.5067	31.61	45.27	.5119	31.95	45.83	.5140	32.76	50.33	.5209
52	1.140	31.59	44.17	.4710	32.15	44.78	.4733	32.45	45.30	.4759	33.33	49.67	.4844
56	1.120	32.61	43.90	.4230	33.17	44.53	.4253	33.49	44.98	.4259	34.44	49.04	.4329
57	1.100	33.12	43.83	.4012	33.69	44.46	.4036	33.96	44.88	.4047	35.00	48.75	.4076
57.5	1.068 (Iso.)												

α_o and α_e are in units 10^{-24}cm^3

Table :4.6

Polarizability (α) and order parameter ($\langle P_2 \rangle$) of BBBA (using Neugebauer's relations)

Temp. (°C)	λ = 6907 Å			λ = 5780 Å			λ = 5461 Å			λ = 4358 Å		
	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$
42.3	30.53	45.53	.6329	31.03	46.27	.6264	31.24	46.73	.6259	31.39	50.38	.6278
44.2	30.70	45.17	.6106	31.20	45.91	.6046	31.41	46.37	.6045	31.57	49.96	.6079
46	30.33	44.62	.6030	30.84	45.37	.5972	31.05	45.84	.5976	31.26	49.36	.5983
52.8	30.88	45.11	.6000	31.39	45.88	.5964	31.61	45.36	.5960	31.85	49.82	.5940
57.3	31.65	45.44	.5819	32.17	46.21	.5771	32.39	46.70	.5782	32.66	50.08	.5759
62	32.58	45.65	.5515	33.11	46.43	.5475	33.32	46.92	.5450	33.58	50.31	.5530
65.5	33.35	45.58	.5150	33.88	46.37	.5134	34.09	46.87	.5164	34.44	50.19	.5207
70	34.37	45.48	.4688	34.89	46.28	.4682	35.10	46.78	.4715	35.76	50.13	.4750
72.5	35.70	45.89	.4300	36.2	46.71	.4320	36.43	47.22	.4360	37.32	50.63	.4400

α_o and α_e are in $10^{-24} cm^3$ unit

Table : 4.7

Polarizability (α) and order parameter ($\langle P_2 \rangle$) of PBBA (using Neugebauer's relations)

Temp. (°C)	λ = 6907 Å			λ = 5780 Å			λ = 5461 Å			λ = 4358 Å		
	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$
27.7	27.33	39.09	.6143	27.92	39.52	.6146	28.35	40.12	.6181	28.89	43.10	.6128
32	27.46	39.05	.6054	28.03	39.55	.6067	28.46	40.08	.6102	29.02	43.01	.6034
36	27.77	39.05	.5893	28.34	39.53	.5919	28.77	40.12	.5960	29.34	43.03	.5904
40	28.08	38.91	.5657	28.61	39.55	.5761	28.95	40.00	.5803	29.61	42.98	.5766
44	28.39	38.38	.5479	29.04	39.54	.5530	29.40	39.96	.5546	29.96	42.85	.5559
48	28.93	38.76	.5135	29.48	39.37	.5208	29.82	39.82	.5252	30.35	42.61	.5288
52	29.62	38.83	.4811	30.18	39.41	.4861	30.47	39.82	.4910	31.07	42.59	.4960
56	30.77	39.13	.4367	31.34	39.73	.4418	31.65	40.12	.4448	32.26	42.74	.4520
57	31.64	39.68	.4200	32.22	40.29	.4250	32.49	40.64	.4280	33.27	43.24	.4300

α_o and α_e are in $10^{-24} cm^3$ unit.

Table :4.8

Polarizability (α) and order parameter ($\langle P_2 \rangle$) of APAPA (using Neugebauer's relations)

Temp. (°C)	λ = 5780 Å			λ = 5461 Å		
	α_o	α_e	$\langle P_2 \rangle$	α_o	α_e	$\langle P_2 \rangle$
75	23.10	38.56	.6247	23.71	39.32	.6275
80	23.28	38.50	.6150	23.90	39.26	.6175
85	23.49	38.37	.6017	24.10	39.14	.6047
90	23.98	38.32	.5795	24.60	39.09	.5825
95	24.49	38.27	.5569	25.11	39.04	.5600
100	25.44	38.01	.5080	26.06	38.79	.5117
105	26.35	37.49	.4300	27.46	38.28	.4350

α_o and α_e are $10^{-24} cm^3$ unit.

agree fairly well for all the three liquid crystals in their respective mesophases. Of course the $\langle P_2 \rangle$ values obtained from Neugebauer's approach are slightly higher than those obtained from the other approach for these compounds. These values of order parameter along with the order parameter values from X-ray data have been shown in the figure 4.5. The continuous curves correspond to the Maier-Saupe mean field theoretical values. From the figure 4.5 it is clear that the agreement, with Maier-Saupe theoretical predictions, of my experimental $\langle P_2 \rangle$ values from refractive index measurements is fairly good for these three compounds. My experimental $\langle P_2 \rangle$ values are also close to the values obtained by others[14, 23] using different methods. $\langle P_2 \rangle$ values from X-ray data are slightly higher than the theoretical values, specially for BBBA and APAPA. This type of observations are frequently encountered[25-27]. In the previous chapter I have discussed elaborately the possible causes for such discrepancy between the order parameter values obtained from X-ray data and from refractive index measurements and these hold good for the present case as well.

Most striking question arises here that whether the isotropic local field model (Vuks' approach) is more realistic than the anisotropic local field model (Neugebauer's approach) or reverse is true for describing molecular polarizabilities of such liquid crystalline materials possessing high degree of anisotropy. As it is clear from the figure 4.5

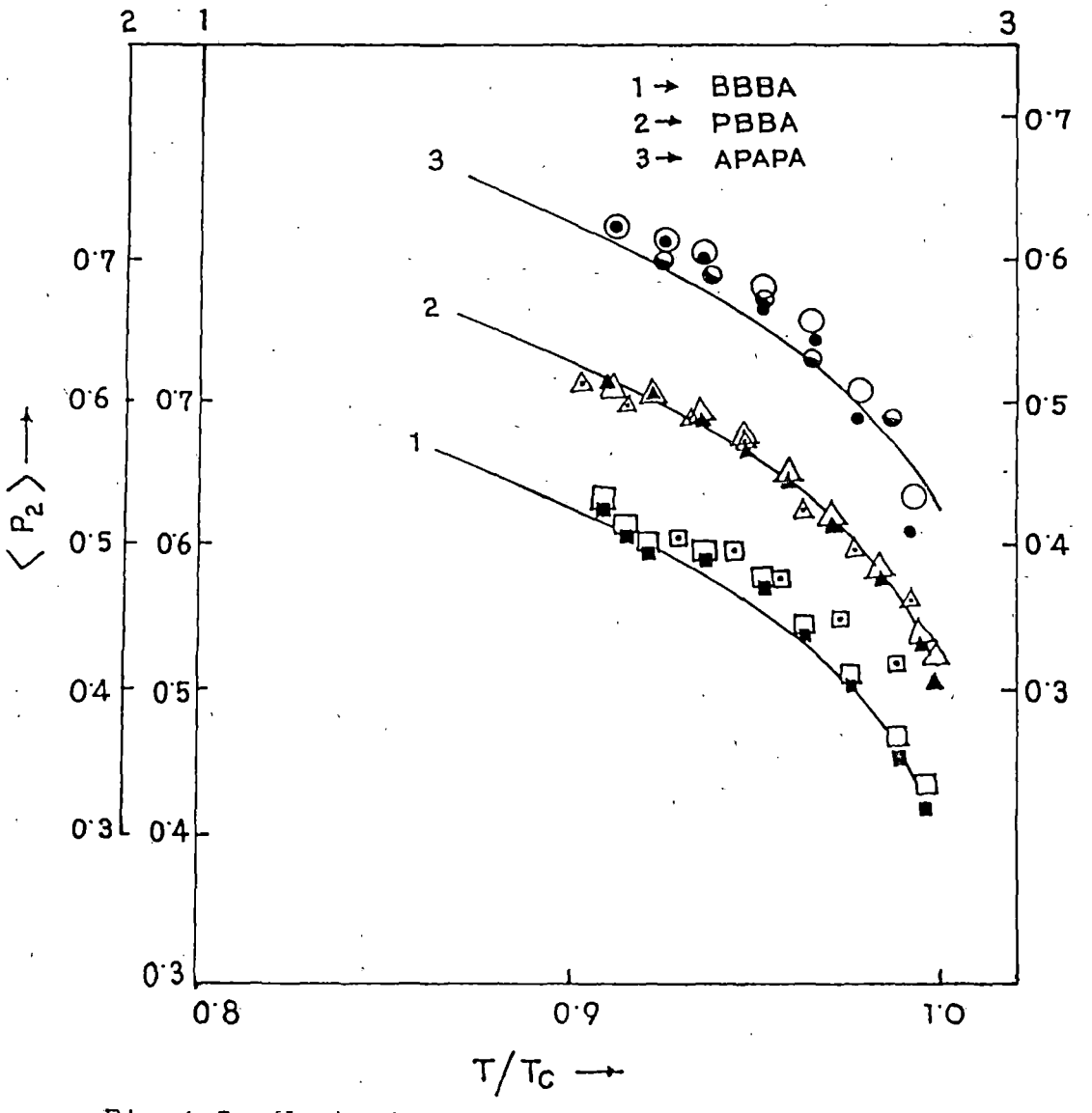


Fig.4.5 Variation of order parameters P_2 with reduced temperature. Solid lines correspond to Maier-Saupe values.

$\langle P_2 \rangle$ from Vuks' formula : ●, ▲, ■ ;

$\langle P_2 \rangle$ from Neugebauer's relations : ○, △, □ ;

$\langle P_2 \rangle$ from X-ray data : ⊖, ⊿, ⊡ .

and Tables 4.3-4.8 that although the values of α_o and α_e vary considerably in two approaches but order parameter values obtained from the two approaches maintained good agreement among themselves for the three compounds. As already mentioned in the previous chapter that this happens due to the fact that the variation of $(\alpha_e - \alpha_o)$ with temperature is same for the two approaches, which is crucial for determining order parameter values. Simple considerations will show that the Neugebauer's approach is more realistic than Vuks' approach for these highly anisotropic compounds, as in the first approach the anisotropy of the local field factors has been considered. This conclusion may be drawn by a comparison of the values of $(\alpha_{\parallel} - \alpha_{\perp})$ and the mean polarizability values (α) evaluated using both these theories with those calculated using the additive rule of bond polarizability together with the estimated values of bond polarizabilities. The bond polarizability calculations have been performed using the different bond polarizability values available in the literatures[28-31]. Table 4.9 contain the $(\alpha_{\parallel} - \alpha_{\perp})$ values obtained from Vuks' approach, Neugebauer's formula and bond polarizability method for $\lambda = 5780$ Å.

It is seen from the Table 4.9 that the bond polarizability anisotropy values $(\alpha_{\parallel} - \alpha_{\perp})$ ~~and the mean polarizability values (α)~~ are much closer to the Neugebauer's anisotropy values than those obtained from the Vuks' formula. The anisotropy from bond polarizability values are the lowest,

Table-4.9

Compounds	$\alpha \times 10^{24}$ cm^3			$(\alpha_{\parallel} - \alpha_{\perp}) \times 10^{24}$ cm^3		
	Vuks' approach / Neugebauer's approach (Isotropic liquid)		Calculated from bond polarizability	Vuks' approach	Neugebauer's approach	Calculated from bond polarizability
BBBA	45.69 ± 1.63		38.51	33.95	24.83	19.03
PBBA	38.96 ± 1.55		36.02	28.00	19.00	18.75
APAPA	36.86 ± 0.48		31.80	36.40	24.74	21.59

showing that the estimated values of bond polarizability anisotropy are probably under estimated and needs revision especially in a system having extensive conjugation like in the molecules studied in this chapter.

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