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Publisher: Taylor & Francis

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Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gmcl16>

pVT-Data and Viscosity-Pressure Behavior of MBBA and EBBA

E. Kuss^a

^a Institut für Erdölforschung, 3 Hannover (BRD), Am Kleinen Felde, 30

Version of record first published: 28 Mar 2007.

To cite this article: E. Kuss (1978): pVT-Data and Viscosity-Pressure Behavior of MBBA and EBBA, *Molecular Crystals and Liquid Crystals*, 47:1-2, 71-83

To link to this article: <http://dx.doi.org/10.1080/00268947808083733>

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***pVT*-Data and Viscosity-Pressure Behavior of MBBA and EBBA**

E. KUSS

Institut für Erdölforschung, 3 Hannover (BRD), Am Kleinen Felde 30

(Received October 5, 1977; in final form April 15, 1978)

INTRODUCTION

In a preceding paper the melting- and pour-points of several homologous series of liquid crystals were measured up to 3 kbar.¹ Thereby it was found, that e.g. in the series of benzylidene-butylanilines the pourpoint temperature T_c and the slope (dp_c/dT_c) of the transition curve isotropic/nematic alternate with increasing chainlength, whereas the melting temperature increases continuously.

On the other hand alternating melting temperatures occasionally were found in series of isotropic substances (e.g. for alkoxybenzoic acids) and were attributed to alternating crystal structures of the solid phase.²⁻⁴

Alternating transition temperatures likewise found between other mesophases of liquid crystals (e.g. cholesteric/isotropic and cholesteric/smectic A)⁵⁻⁸ and the above mentioned behavior of the benzylidene-butylanilines indicate, that alternating properties are not necessarily related to alternating crystal structures of the solid phase.

To get further information about the physical properties of the benzylidene-butylanilines next the *pVT*-data and the viscosity-pressure behavior of MBBA and EBBA were investigated.

The lack of *pVT*-data is mentioned occasionally in the literature⁹ and indeed there are known to us only a few publications with experimental *pVT* data of cholesteric liquid crystals,¹⁰⁻¹² and one of MBBA in the low pressure region.¹³

The *viscosity* is very sensitive to structural influences. As example, it may be mentioned, that the di-*n*-alkyl-esters of cyclohexylmalonic acid show an

alternating viscosity in dependence of chainlength even at temperatures fifty degrees above the melting temperature.¹⁴

Concerning *liquid crystals* the viscosity behavior is much more complicated, since there are several possibilities for the orientation of the anisotropic large molecule within the flow field. For numerical acquisition six viscosity coefficients were introduced by Erickson¹⁵ and Leslie.¹⁶

Furthermore viscosity measurements may throw some light on:

- a) Pretransitions, observed occasionally at 1 bar on the low temperature side of the transition.¹⁷⁻¹⁹
- b) Tricritical points, where the first order phase transition changes to one of higher order.^{18, 20-23}
- c) The helical structure of cholesteric phases, since the pitch of the helix in solutions was found to be strongly dependent on pressure.²⁴

I THE pVT -DATA OF MBBA AND EBBA

MBBA [*N-(p-Methoxy-Benzylidene)p-n-Butyl-Aniline*] first prepared by Kelker and Scheurle²⁵ is influenced in its properties strongly by very small amounts of impurities. By new methods of purification^{26, 27} the pourpoint has raised nearly about 6.5 degrees centigrade.

The product used by us for pVT - and viscosity-measurements had a pourpoint of 45.7°C. Within the high pressure chamber the measuring substance is enclosed in a piezometer of glass and confined by mercury. The level of mercury in a calibrated ascending pipe is determined by moving down a tungsten tip until electric contact is achieved. For this a self-sealing stuffing box and a mechanical fine feed inside the pressure chamber is used. Details of the device have been published in earlier papers.^{28, 29}

The measurements were performed at 6 temperatures by steps of 100 bar up to 2 kbar resp. up to the point of solidification. The reproducibility in density was $\pm 0.0003 \text{ g cm}^{-3}$. The values are collected in Table I.

The lines between the values separate the isotropic phase (above the line) from the nematic one (below the line). Numerous points measured in the neighbourhood of the transition point are not listed here. Instead of this the transition pressure p_{Tr} , the density of the isotropic phase ρ (isotr.) and the nematic one ρ (nem.) at the transition isotropic/nematic and the pressure of solidification are given in the lower part of Table I.

pVT -data and compressibilities are related to intermolecular forces. They are a measure for the mean distance between the molecules and therefore the different physical properties preferably are regarded as function of density

TABLE I

The Density of MBBA in an isotropic and the nematic Phase in Dependence of Pressure

ρ [g·cm⁻³]

bar	40°C	50°C	60°C	70°C	80°C	90°C
1	<u>1.0332</u>	1.0228	1.0146	1.0064	0.9983	0.9901
100	1.0387	<u>1.0284</u>	1.0200	1.0117	1.0038	0.9957
200	1.0441	1.0359	1.0251	1.0171	1.0092	1.0014
300	1.0493	1.0414	1.0306	1.0224	1.0145	1.0068
400	1.0540	1.0464	<u>1.0357</u>	1.0277	1.0197	1.0120
500	1.0588	1.0515	1.0428	1.0328	1.0248	1.0171
600	1.0636	1.0562	1.0478	<u>1.0376</u>	1.0298	1.0221
700	solid	1.0607	1.0525	1.0442	1.0345	1.0270
800		1.0652	1.0569	1.0490	1.0391	1.0316
900		1.0693	1.0612	1.0535	<u>1.0435</u>	1.0361
1000		1.0730	1.0655	1.0577	<u>1.0496</u>	1.0405
1100		solid	1.0694	1.0617	1.0539	1.0448
1200			1.0734	1.0656	1.0580	<u>1.0489</u>
1300			1.0774	1.0694	1.0619	1.0548
1400			solid	1.0732	1.0658	1.0589
1500				1.0771	1.0698	1.0628
1600				1.0809	1.0735	1.0665
1700				1.0847	1.0772	1.0702
1800				solid	1.0807	1.0739
1900					1.0841	1.0775
2000					1.0875	1.0811
p_{Tr} (bar)	---	130	400	682	970	1270
(is.)	---	1.0299	1.0358	1.0415	1.0465	1.0516
(nem.)	---	1.0320	1.0379	1.0435	1.0483	1.0536
p_c (bar)	<u>645</u>	<u>1018</u>	<u>1398</u>	<u>1793</u>	<u>2208</u>	<u>2667</u>

instead of pressure. For this an appropriate equation of state is needed.

The equation of Tait:
$$\frac{V_0 - V}{V_0} = C \log \left(\frac{B + p}{B + p_0} \right)$$

which till now has proved very well for pure liquids, was tested for MBBA. The isotropic and nematic phase were regarded separately because of the jump in density.

The values of B and C given in Table II were obtained by computer evaluation from the experimental results. With only a small loss in exactness C can be regarded as independent of temperature. With B and C of Table II the measured densities are reproduced in both mesophases to $\pm 4 \cdot 10^{-4}$ g cm⁻³ exact. Furthermore the initial compressibility κ_0 and this of the two mesophases in the transition region are tabulated in Table II.

TABLE II

The Compressibility and the Coefficients of the Tait-Equation in both phases of MBBA

°C	$\kappa \cdot 10^6 [\text{bar}^{-1}]$			C for all temper.	$B [\text{bar}^{-1}]$	
	1 bar	Transition point			isotr.	nematic
	isotr.	isotr.	nemat.			
60	54.55	45.88	43.96	<i>isotrop.</i>	2116.2	2087.4
70	55.94	41.77	40.13	0.2658	2063.7	2034.5
80	57.35	38.92	36.65	<i>nematic</i>	2012.5	1977.9
90	59.10	36.60	34.38	0.2619	1947.0	1908.7

The following results are received:

1) The compressibility κ_0 at 1 bar and 60°C ($54.55 \cdot 10^{-6}$) is small in comparison with that of *n*-decane ($134.5 \cdot 10^{-6}$) or benzene ($123.1 \cdot 10^{-6}$) at the same temperature.

2) The compressibility κ at the transition point is nearly the same in both phases.

3) B decreases with increasing temperature and is always somewhat smaller in the nematic phase than in the isotropic liquid.

For comparison only the publication of A. C. Zawisza and J. Stecki¹³ can be adduced, but unfortunately these measurements are performed only up to 70 bar at 44 till 61°C and the volumes are given only in arbitrary units. The slope dp/dT of the transition curve given with 25.3 at/°C by Zawisza and Stecki and our value 28.0 at/°C are approximately in agreement. The densities at the transition point related to the same temperature are:

	<i>isotropic</i>	<i>nematic</i>
Zawisza and Stecki	1.0252	1.0263
our values	1.0255	1.0276

This means, we find a greater jump in density.

EBBA [N-(*p*-Ethoxy-Benzylidene)*p*-*n*-Butyl-Aniline] with a pourpoint of 79.8°C was investigated in the same manner. The pVT -data are compiled in Table III.

The following results are received:

a) Despite its greater chainlength EBBA has a smaller density than MBBA.

b) The change in density at the transition isotropic/nematic is noticeably greater for EBBA (about $27 \cdot 10^{-4} \text{ g cm}^{-3}$) than for MBBA (about $21 \cdot 10^{-4} \text{ g cm}^{-3}$).

TABLE III

The Density of EBBA in the isotropic and the nematic Phase as function of Pressure

 $\rho [\text{g} \cdot \text{cm}^{-3}]$

bar	50°C	60°C	70°C	80°C	90°C	100°C	110°C	120°C	130°C	
1	<u>1.0152</u>	<u>1.0071</u>	<u>0.9990</u>	<u>0.9881</u>	0.9803	0.9726	0.9649	0.9572	0.9490	
100	1.0207	1.0128	1.0050	0.9972	0.9865	0.9790	0.9715	0.9639	0.9558	
200	1.0260	1.0184	1.0108	1.0034	<u>0.9927</u>	0.9853	0.9780	0.9706	0.9624	
300	1.0311	1.0238	1.0165	1.0094	1.0015	0.9914	0.9843	0.9767	0.9689	
400	1.0361	1.0289	1.0219	1.0153	1.0077	0.9975	0.9905	0.9830	0.9750	
500	./.	1.0338	1.0271	1.0208	1.0136	<u>1.0034</u>	0.9964	0.9891	0.9807	
600	solid	1.0385	1.0321	1.0260	1.0192	1.0119	1.0024	0.9946	0.9867	
700		1.0431	1.0368	1.0309	1.0245	1.0176	1.0079	1.0005	0.9924	
800	solid	1.0475	1.0414	1.0356	1.0296	1.0231	<u>1.0134</u>	1.0060	0.9980	
900		solid	1.0460	1.0402	1.0344	1.0281	1.0210	1.0113	1.0034	
1000			1.0505	1.0446	1.0390	1.0331	1.0261	1.0164	1.0085	
1100			1.0549	1.0488	1.0433	1.0378	1.0311	<u>1.0211</u>	1.0134	
1200			./.	1.0529	1.0475	1.0422	1.0360	1.0285	1.0183	
1300			./.	1.0569	1.0516	1.0464	1.0403	1.0331	1.0228	
1400			./.	1.0608	1.0556	1.0506	1.0446	1.0376	<u>1.0274</u>	
1500			./.	1.0645	1.0595	1.0541	1.0484	1.0418	1.0345	
1600					1.0633	1.0580	1.0521	1.0456	1.0385	
1700					1.0670	1.0616	1.0559	1.0496	1.0425	
1800					1.0705	1.0651	1.0595	1.0533	1.0462	
1900					1.0740	1.0685	1.0630	1.0570	1.0500	
2000					./.	1.0719	1.0664	1.0606	1.0537	
p_{Tr} (bar)	./.	./.	./.	10	278	554	850	1150	1470	
p_c (bar)	470	820	1180	1560	1950	2370				

c) The density jump seems not to be diminished to higher pressures and temperatures.

d) The compressibility of EBBA (κ_0 at 60°C $56.68 \cdot 10^{-6} \text{ bar}^{-1}$) is a bit greater than of MBBA. Furthermore the isotropic phase is anew a little more compressible than the nematic one.

II THE VISCOSITY-PRESSURE BEHAVIOR OF MBBA AND EBBA

To cover a sufficient broad range in the isotropic and the nematic phase measurements at high pressures and simultaneous high temperatures are needed. For this a high pressure viscometer of the falling ball type for maximal 4 kbar at temperatures up to 300°C was constructed. For measuring small viscosities in the isotropic phase at high temperatures the pressure chamber is

arranged horizontally and is tilted only about small angles against the horizontal direction. At great viscosities (e.g. in smectic phases of other liquid crystals) the chamber can be swivelled about great angles. To avoid flexible high-pressure conducting tubings between the tiltable pressure chamber and the pressure generator the compressor with motor, gear, gauges and valves are installed on the same basic bottom and are tilted with.

Inside the pressure chamber the measuring substance is arranged in a precision glass tube of 6.500 ± 0.003 mm interior diameter. A glass piston grinded in the tube within a tolerance of 0.005 mm transfers the pressure from the compressor oil to the test substance inside the glass tube.

Using an inductive registration method often the insulation of the registration coils was damaged at high temperature. Therefore a capacitive registration method was developed. Because of the small capacity of the electrodes (below 1 pF) they were employed as coupling capacitors between a 20 kHz oscillator and a two stage amplifier. The passage of the ball through one of the capacitors actuates an electronic clock with six decades. Long measuring times of several hours could be measured within a reproducibility of 1 percent. Details and wiring diagrams are published in another paper.³⁰

In Figure 1 five isotherms of *MBBA*, measured with the instrument between 50 and 90°C, are reproduced.

The results may be summarized as follows:

a) The transition point isotropic/nematic can be recognized by a sharp peak of the viscosity. Whereas the density is altered only by two permille the viscosity at the transition point is diminished by about 25 percent.

b) Perhaps it is astonishing, that the nematic phase has *the higher density but a smaller viscosity* than the isotropic phase. The long-chain molecules within the nematic phase are orientated nearly parallel to each other and are approximately aligned in the direction of streaming. By this orientation the molecules can move more easily past each other in the dense nematic phase than in the disordered isotropic state.

c) The jump of viscosity scarcely changes along the transition curve. On the isotropic side, however, a sharp peak appears whereas on the nematic side a curvature of the isotherms is found, which becomes broader with increasing pressure. This behavior is probably caused by pretransitions within the nematic phase in the neighbourhood of the transition point.

The logarithmic viscosity plot of *EBBA* against pressure in Figure 2 represents nearly straight-lined isotherms. Thus in a first approximation the isotherms of the nematic phase as well as those of the isotropic liquid can be described by the statement:

$$\eta_p = \eta_0 \cdot \exp(\alpha \cdot p)$$

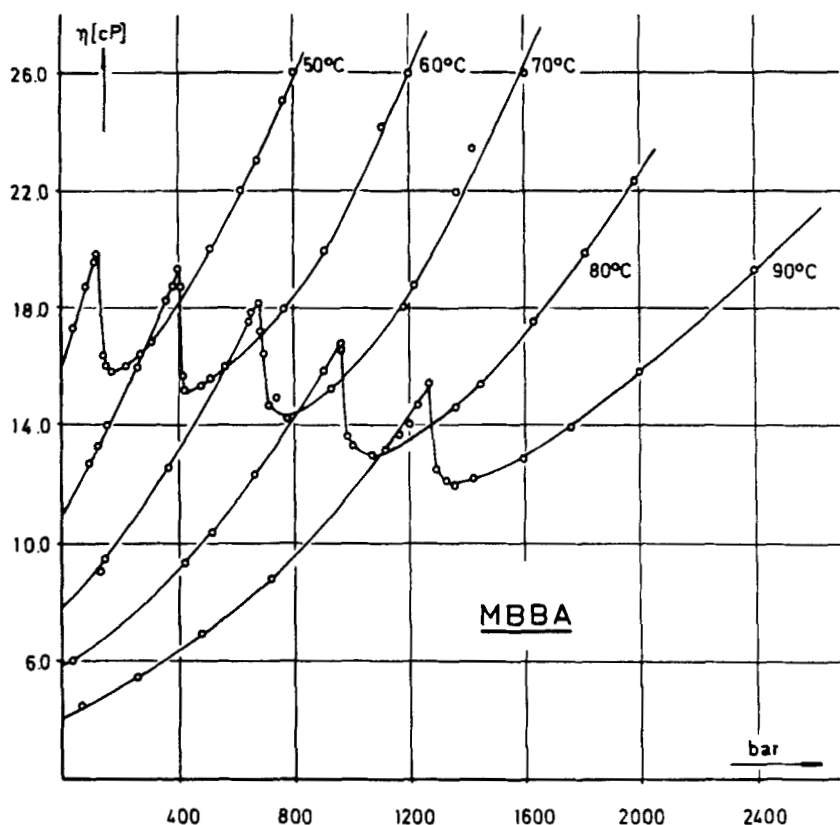


FIGURE 1 The Viscosity-Pressure Isotherms of MBBA.

Here also we find a sharp fall on passing from the isotropic to the nematic phase. The transition curve given by this viscosity jump in Figure 2 differs remarkably from a state diagram published by Keyes, Weston and Daniels,²¹ but it stands in sufficient agreement with our earlier optical measurements of the pourpoint¹ and with data of W. Spratte and G. M. Schneider³¹ obtained by DTA.

The numerical values of the viscosity in the two mesophases at high pressure are listed in Table IV for MBBA and Table V for EBBA.

For MBBA at 50°C and atmospheric pressure the value differs by 1.2% from that of White, Cladis and Torza³² probably because of somewhat different purity of the substance.

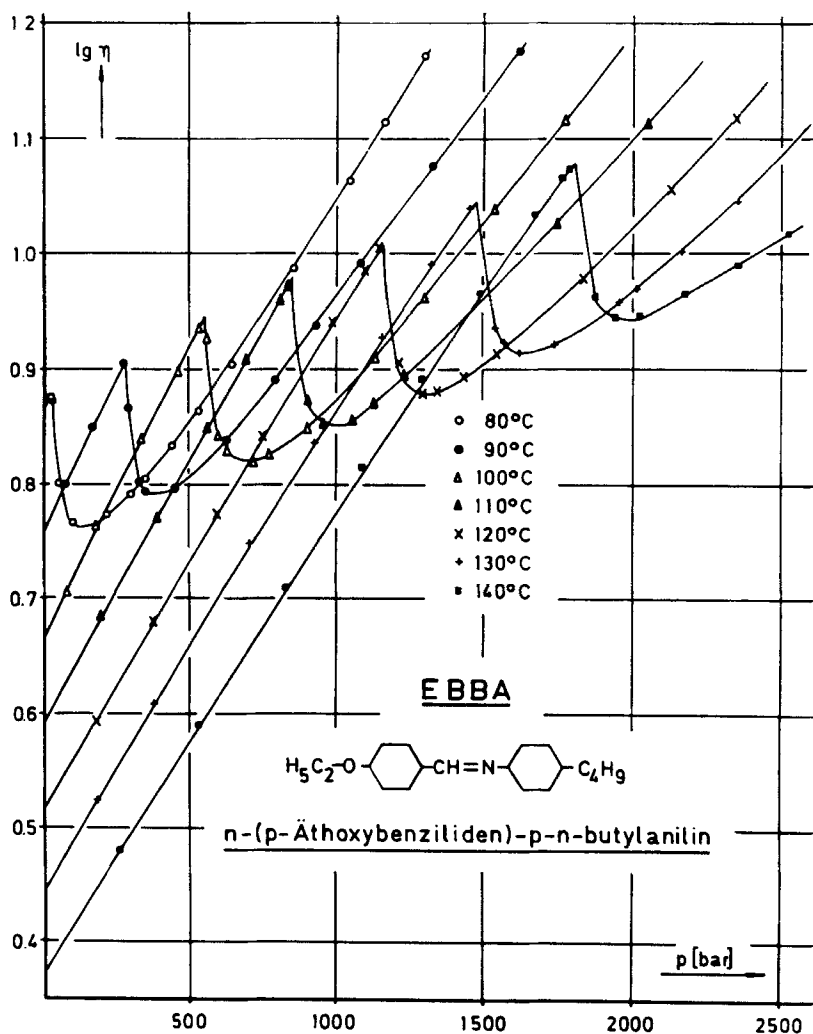


FIGURE 2 The Viscosity-Pressure Behavior of EBBA.

III DISCUSSION

As was mentioned above, for a complete description of viscosity in liquid crystals six viscosity coefficients, called the Leslie viscosities, would be required. This is caused by the different possibilities for the orientation of the anisotropic molecule against direction of flow velocity.

However, A. E. White, P. E. Cladis and S. Torza³² have shown, that in a

TABLE IV

The Pressure Dependence of Viscosity for MBBA in the isotropic and the nematic Phase (η in Centipoise)

bar	50°C	60°C	70°C	80°C	90°C
1	15.82	10.97	7.79	5.73	4.03
100	<u>19.30</u>	12.73	8.84	6.42	4.53
200	15.88	14.81	10.12	7.21	5.10
300	16.72	16.72	11.49	8.08	5.67
400	18.00	<u>19.23</u>	13.06	9.06	6.33
500	19.80	15.42	14.78	10.17	7.03
600	21.68	16.22	<u>16.75</u>	11.42	7.82
700	23.61	17.08	16.38	12.73	8.66
800	25.94	18.37	14.30	14.18	9.58
900	./.	19.81	15.02	<u>15.71</u>	10.55
1000	./.	21.70	15.90	13.32	11.62
1100		23.88	16.99	13.08	12.78
1200		25.97	18.22	13.53	<u>14.07</u>
1300		./.	19.60	14.17	12.38
1400		./.	21.15	14.95	12.03
1500		./.	22.88	15.98	12.39
1600			25.02	17.11	12.92
1700			./.	18.39	13.47
1800				19.70	14.10
1900				21.07	14.78
2000				22.47	15.59

Transition Temperature $T_{n-i} = 45.7^\circ\text{C}$: at 1 bar

$$\frac{dT_{n-i}}{dp} = 35.1^\circ/\text{kbar}$$

falling ball viscometer under conditions like those used by us the measured apparent viscosity is nearly the Miesowicz viscosity η_B . The molecule is approximately arranged parallel to the velocity field. The value of viscosity does not depend upon the shear rate. This was proved by us in separate experiments.

A considerable difference appears in the viscosity–pressure behavior of MBBA and EBBA. Along the transition curve the extreme values of viscosity for EBBA and other nematic substances measured by us increase with increasing pressure and temperature, whereas for MBBA a decrease of the maximum and minimum values is found. This clearly can be recognized from Figure 3, where the *isobars* of the two substances are represented as a function of temperature. The nematic region is situated on the left and the isotropic on the right side of the peaks.

TABLE V

The Pressure Dependence of Viscosity. EBBA in the isotropic and the nematic Phase. (η in Centipoise)

bar	80°C	90°C	100°C	110°C	120°C	130°C	140°C
1	<u>7.38</u>	5.775	4.667	3.930	3.32	2.790	2.383
100	5.85	6.42	5.25	4.37	3.63	3.06	2.59
200	5.93	<u>7.35</u>	5.94	4.86	4.01	3.39	2.85
300	6.21	7.05	6.68	5.40	4.44	3.76	3.13
400	6.65	6.21	7.46	5.99	4.90	4.16	3.43
500	7.15	6.435	<u>8.36</u>	6.66	5.44	4.58	3.78
600	7.75	6.80	6.95	7.39	6.01	5.03	4.18
700	8.48	7.34	6.63	8.21	6.69	5.56	4.58
800	9.28	7.89	6.80	<u>9.13</u>	7.32	6.09	5.04
900	10.12	8.51	7.11	<u>7.51</u>	8.09	6.71	5.51
1000	11.12	9.23	7.54	7.11	8.88	7.34	6.05
1100	12.25	10.00	8.03	7.34	<u>9.78</u>	8.04	6.65
1200	13.50	10.84	8.61	7.75	8.39	8.81	7.29
1300	14.90	11.72	9.24	8.19	7.59	9.65	7.91
1400	./.	12.71	9.94	8.64	7.74	<u>10.52</u>	8.57
1500	./.	13.66	10.70	9.15	8.07	10.00	9.35
1600		14.89	11.55	9.76	8.43	8.23	10.23
1700		./.	12.43	10.41	8.90	8.27	11.11
1800		./.	13.37	11.08	9.39	8.57	<u>11.75</u>
1900			14.46	11.83	9.99	8.90	8.91
2000			15.56	12.68	10.54	9.30	8.82
2100			./.	13.52	11.30	9.82	9.06
2200			./.	14.47	12.02	10.30	9.36
2300				./.	12.81	10.88	9.74
2400				./.	13.71	11.48	10.00
2500					./.	12.09	10.35
p_{Tr} (bar)	15	282	550	850	1152	1468	1800
η_{i-n} (cP)	7.58	8.12	8.81	9.56	10.29	11.10	11.94

Along the transition curve the density and the temperature increase simultaneously. Separating the influences of density and temperature it is found, that the change in density is relatively small along the transition curve for MBBA, whilst the influence of temperature is relatively great in comparison with EBBA. Thus the increase of viscosity with density along the transition curve of MBBA is overcompensated by the influence of the simultaneously increasing temperature.

Additionally the cohesive pressure

$$p_i = T \left(\frac{\partial p}{\partial T} \right)_v - p_a$$

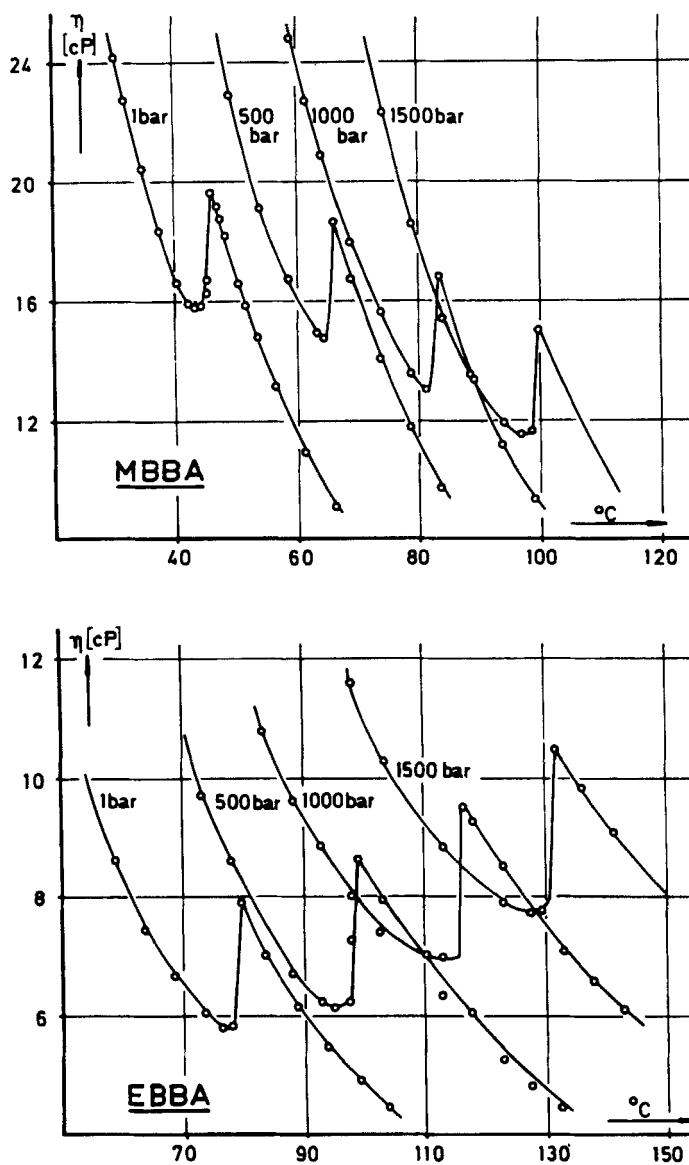


FIGURE 3 The Viscosity-Isobars of MBBA and EBBA as a function of Pressure.

and the activation energy of the viscous flow

$$E_p = R \frac{d(\ln \eta)}{d(1/T)}$$

may be regarded.

The internal pressure p_i increases with rising temperature and pressure, occupying values between 3500 and 5000 bar. The difference between the two mesophases is only small.

The activation energy E_p increases with rising temperature and pressure, too. But for MBBA in the isotropic phase it has much greater values (8.5 till 10 kcal/Mol) than for EBBA (5.3 till 6.7 kcal/Mol). The sudden removal of the activation energy at the transition isotropic/nematic thus is greater for MBBA than for EBBA. In the nematic phase it follows a renewed increase of the activation energy with rising pressure.

SUMMARY

The density of MBBA and EBBA was measured up to 2 kbar with a reproducibility of $\pm 0.0003 \text{ g cm}^{-3}$. In both mesophases the isotherms can be represented by the equation of TAIT.

At the transition point isotropic/nematic the density increases only by some permille whereas the viscosity decreases by about 25 percent, because of the alignment of the molecules nearly parallel to the velocity field. The shape of the viscosity isotherms indicates pretransitions in the nematic phase.

Along the transition line the viscosity increases for EBBA but decreases for MBBA, where the influence of increasing density is overcompensated by the influence of increasing temperature.

Acknowledgement

The financial support of this work by the Fonds der Chemischen Industrie Frankfurt/M. is thankfully appreciated.

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