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I. Teucher^a, C. M. Paleos^a & M. M. Labes^{a b}

^a Department of Chemistry, Drexel University, Philadelphia, Pennsylvania, 19104

^b Department of Chemistry, Temple University, Philadelphia, Pa., 19122

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Properties of Structurally Stabilized Anil-Type Nematic Liquid Crystals

I. TEUCHER, C. M. PALEOS and M. M. LABES†

Department of Chemistry
Drexel University
Philadelphia, Pennsylvania 19104

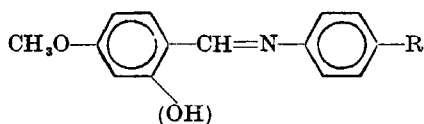
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One of the major problems associated with the use of substituted benzylideneaniline type liquid crystals is their relative instability to cleavage or reaction of the anil linkage. Many workers have noted the presence of ubiquitous impurities in these materials which influence the reproducibility of the phase transition temperatures and their electro-optic properties, and it would be of obvious advantage to enhance their stability in some way. A room temperature nematic liquid crystal, N-(*p*-methoxybenzylidene)-*p*-*n*-butylaniline (MBBA), has been recently synthesized,⁽¹⁾ and a large number of investigations of its physical properties have been undertaken.⁽²⁻⁴⁾ In particular, "dynamic scattering",⁽⁵⁾ which has application in information display devices, occurs in these materials, and the role of impurities appears to be critical, albeit not well understood.⁽⁶⁾

We have prepared a group of *o*-hydroxy substituted anils in which intramolecular hydrogen bonding stabilizes the anil linkage; these are listed in Table 1. It has been reported⁽⁷⁾ that intramolecular proton exchange between the oxygen and nitrogen in such structures is quite rapid, and preliminary indications are that the stability to cleavage of the anil linkage is considerably enhanced when the *o*-hydroxy group is present. For example, dissolving tartaric acid in MBBA leads to the rapid formation of *p*-*n*-butylanilinium tartrate at room temperature; the *o*-hydroxy analog forms the analogous salt at a much slower rate.

The preparation of the *o*-hydroxy analog of MBBA (OHMBBA) is illustrative of the general technique utilized for these compounds.

† Present address: Department of Chemistry, Temple University, Philadelphia, Pa. 19122.

TABLE 1 Melting Range of Several Anil-Type Nematic Liquid Crystals and Their *o*-Hydroxy Substituted Analogs.

Compound <i>R</i> equals	Nematic range °C	<i>o</i> -Hydroxy substituted nematic range °C
—CN	106–117	141–147
—phenyl	161–177	165–197
—CH=CH ₂	97–111	111–124
— <i>n</i> -C ₄ H ₉	21 → 41 heat 41 → 11 cool	44 → 64.5 heat 64.5 → <8 cool

(The vinyl compound, however, is prepared at room temperature in the presence of an inhibitor.) Equimolar quantities of *p*-methoxy-*o*-hydroxybenzaldehyde, prepared by the method of Zemplén *et al.*,⁽⁴⁾ and *p*-*n*-butylaniline (Aldrich Chem. Co.) were dissolved in absolute ethanol, mixed and heated to boiling for 30 minutes. Upon cooling to 0°, the crystalline solid forms. It can be easily recrystallized from ethanol or hexane. Anal. Found %C 76.36, %H 7.54, %N 5.21. Theor. %C 76.29, %H 7.46, %N 4.94. OHMBBA upon cooling from the isotropic phase, remains nematic to <8° in a capillary melting tube or in a thick film between microscope slides. In a thin film such as is used in dynamic scattering experiments, crystallization does occur, however, near 26°. OHMBBA was tested for its dynamic scattering properties by measuring the transmission,⁽⁵⁾ as determined by a thermopile reading, of a 24 μ layer sandwiched between two tin-oxide coated quartz plates. Fig. 1 shows the voltage-transmission relationship as well as the calculated contrast ratio which attains a value > 60. Although these results were determined at 37°, the material manifests dynamic scattering down to 26° at which point it crystallized. The liquid crystal film showed very homogeneous and reproducible scattering characteristics as contrasted with the behavior of MBBA which is much more difficult to purify and decomposes slowly. OHMBBA has a room temperature resistivity > 3 × 10¹⁰ Ωcm indicative of its purity.

The stability and high contrast ratios obtained with this com-

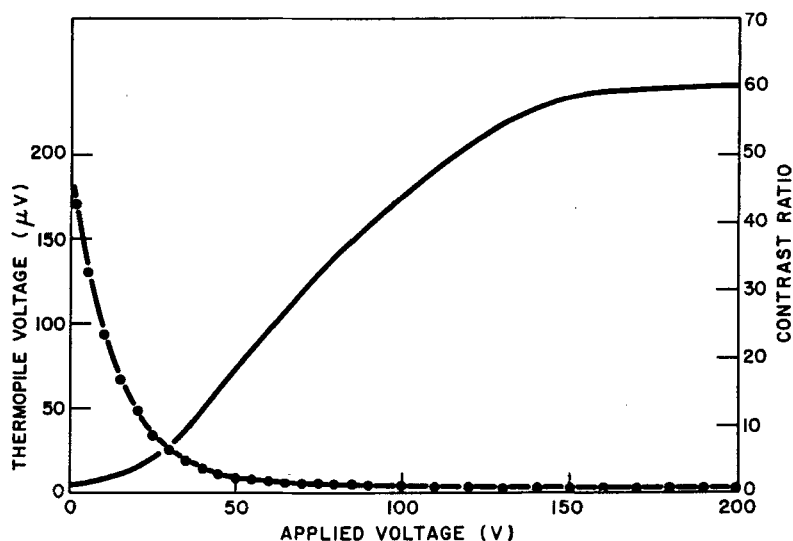


Figure 1. Contrast ratio and transmission (as determined by thermopile voltage) vs applied voltage for a 24μ film of N-(*p*-methoxy-*o*-hydroxybenzylidene)-*p*-*n*-butylaniline at 37° .

pound demonstrates the potential utility of such materials in display devices. By proper doping or admixture, it is possible to obtain room temperature performance.

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