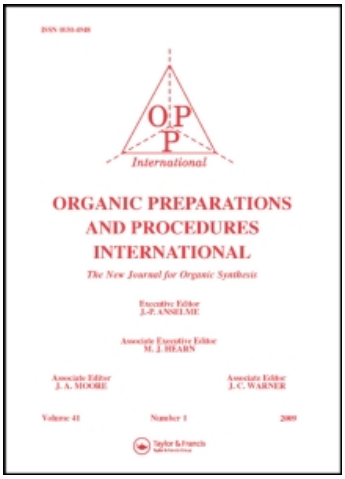

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IMPROVED SYNTHESIS AND PURIFICATION OF p-METHOXYBENZAL-p-(n-BUTYL)ANILINE (MBBA)

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Carefully prepared and purified p-methoxybenzal-p-(n-butyl)aniline (MBBA),[1] which is liquid crystalline at room

$$p\text{-}CH_3OC_6H_4CHO + p\text{-}C_4H_9C_6H_4NH_2 \longrightarrow \underset{\text{(MBBA)}}{p\text{-}CH_3OC_6H_4CH{=}N\text{-}C_6H_4\text{-}pC_4H_9}$$

temperature, exhibits a nematic range of 20° to 48°. Published details of synthesis and purification are lacking. We have therefore investigated the preparation and purification of this material and report our findings.

EXPERIMENTAL

In a 2 l. round-bottomed flask equipped with a Dean-Stark trap and a reflux condenser, were added 500 g (3.67 moles) of p-anisaldehyde (freshly distilled over anhydrous potassium carbonate, bp. 249°-250°), 600 g (4.0 moles) of p-(n-butyl)-aniline (freshly distilled, bp. 75°-77°/1 mm) and 500 ml of reagent grade heptane. The mixture was heated for 30 hrs. and most of the water of condensation was collected during the first 3 hrs. of the reaction. The remaining few ml. of water were collected over the rest of the 30-hour reaction

period. The moisture trap and the reflux condenser were replaced with a distillation head. The solution was stirred magnetically while heptane was removed under reduced pressure (water aspirator) below 70°. When most of the heptane had been removed, the residue was fractionally distilled under high vacuum (diffusion pump). A forerun of excess n-butylaniline was collected. The first fraction of MBBA distilled at 159-160°/0.5 mm, 200 g. This fraction had an isotropic temperature of 42.0°. The subsequent fractions distilled at 168°/0.5 mm, yield 720 g. Isotropic point 46.2°.

MBBA (300 g, isotropic point 46.2°) and dry hexane (1000 ml) were placed in a 2 l. flat-bottomed freeze-drying flask. The flask was cooled to -50° in an ethanol-liquid nitrogen bath. During crystallization, the solution was stirred under a blanket of dry nitrogen. The product was collected by filtration on a Büchner funnel and excess hexane was pressed from the crystals with a latex rubber dam. The product was dried under vacuum, yield 240 g, isotropic point 47.5°. The transition temperature was measured with a Du Pont model 990 thermal analyser.

REFERENCE

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