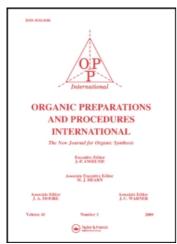
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# IMPROVED SYNTHESIS AND PURIFICATION OF *p*-METHOXYBENZAL-*p*-(*n*-BUTYL)ANILINE (MBBA)

N. V. V. Raghavan<sup>a</sup>; L. A. Paddock<sup>a</sup>
<sup>a</sup> AMI Display Systems, Sunnyvale, CA

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

Submitted by N. V. V. Raghavan\* and L. A. Paddock
(11/13/75)

AMI Display Systems
848 Stewart Drive, Sunnyvale, CA 94086

Carefully prepared and purified <u>p</u>-methoxybenzal-<u>p</u>- $(\underline{n}\text{-butyl})\text{ aniline (MBBA),}^1 \text{ which is liquid crystalline at room}$   $\underline{p}\text{-CH}_3\text{-CC}_6\text{-H}_4\text{-CHO} + \underline{p}\text{-CC}_4\text{-H}_9\text{-CC}_6\text{-H}_4\text{-NH}_2 \longrightarrow \underline{p}\text{-CH}_3\text{-CC}_6\text{-H}_4\text{-CH}\text{-N-CC}_6\text{-H}_4\text{-PCC}_4\text{-H}_9}$   $(\underline{MBBA})$ 

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 1. 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 5 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-butyl-aniline 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

 H. Kelker and B. Scheurle, Angew. Chem. Int. Ed., <u>8</u>, 884 (1969).