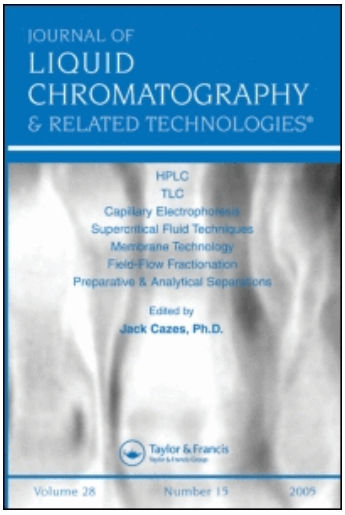

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The Separation of 2-, 3- and 4-Cyclopentylphenol by Thin-Layer Chromatography

T. M. Atkins[a]; D. G. Towles[a]; R. H. Bishara[a]
[a] Lilly Research Laboratories Division of Eli Lilly and Company Indianapolis, Indiana

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THE SEPARATION OF 2-, 3- AND 4-CYCLOPENTYLPHENOL BY THIN-LAYER CHROMATOGRAPHY

T.M. Atkins, D.G. Towles, and R.H. Bishara
Lilly Research Laboratories
Division of Eli Lilly and Company
Indianapolis, Indiana 46285

ABSTRACT

A TLC system is described for the resolution of the three positional isomers of cyclopentylphenol. Quantitation of the 3- and 4- isomers in 2-cyclopentylphenol at a sensitivity of 0.1% may be achieved.

INTRODUCTION

The widespread use of substituted cyclopentylphenols is well documented in the literature. Substituted cyclopentylphenols have been used as antioxidants for organic materials, e.g., polymers, rubbers, lubricants (1), as tough, hard, weather resistant coatings, e.g., on wood (2), as bactericides and fungicides (3,4), in the synthesis of a β-sympatholytic drug (5) and as diuretics which do not increase K^+ excretion as much as that of Na^+ (6). In addition, their phosphorus acid esters showed anti-corrosive and antioxidative properties as lubricating oil additives when tested at the 1% concentration (7). In the synthesis of the cyclopentylphenols, small

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quantities of isomeric impurities are also produced. In this laboratory, the isomeric purity of 2-cyclopentylphenol must be controlled to prevent the formation of undesired by-products. The chromatographic behavior of 4-cyclopentylphenol on paper (8), alumina-impregnated papers and thin layers of alumina (9) and by reverse-phase partition chromatography on home made thin layers of cellulose/ethyl oleate (10) has been investigated. No reference was found in the literature describing resolution of the three isomers of cyclopentylphenol. Using commercially available precoated silica gel plates, a variety of solvent systems was screened according to a previously described procedure (11). This paper describes a two-component TLC system that separates the 2-, 3- and 4-cyclopentylphenols.

EXPERIMENTAL

Silica gel 60, F254 TLC plates (EM Laboratories, Inc., Elmsford, N.Y.), 20 cm x 20 cm with 0.25 mm thick adsorbent layer and a conventional TLC chamber, 30 cm x 25 cm x 8 cm, of heavywall glass were used for all of the development work.

One-tenth, one-fifth and one percent solutions of the 3- and 4-isomers spiked in the 2-cyclopentylphenol standard at a concentration of 10 mg/ml were prepared. The 2-cyclopentylphenol sample to be tested was prepared at a concentration of 10 mg/ml. All solutions were prepared in methanol. Ten microliter aliquots were applied at 2.5 cm from the bottom of the plate. The developing solvent was a mixture of chloroform and ethyl acetate (95:5), v/v. The solvent front was allowed to travel 15 cm above the point of application. The developing time was approximately 90 minutes. The plate was allowed to dry in a ventilated hood and then placed in an iodine chamber (a conventional TLC chamber containing several grams of metallic iodine) for about 30 minutes. The spots were photographed under short-wavelength (254 nm) ultraviolet light.

RESULTS AND DISCUSSION

Viewing the developed, dried plate under short-wavelength ultraviolet light after exposure to iodine reveals the cyclopentylphenols as purple spots on a yellowish-green fluorescent background.

The R_f values were 0.65, 0.51, and 0.44 for 2-, 3-, and 4-cyclopentylphenol respectively. This is in agreement with the conventionally documented mobility for the 2-alkylphenol derivatives which are less strongly adsorbed due to the "ortho effect", than the 3- and 4-isomers (12) and hence move faster.

To quantitate any 3- or 4-isomers in the 2-cyclopentylphenol sample, the intensity of the extra spots with similar mobilities was compared to the lanes containing the spiked 0.1, 0.2 and 1% standards.

In summary, this simple method provides a relatively quick way to identify the isomers and to semi-quantitate the isomeric purity of 2-cyclopentylphenol.

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