

and hydrobromides. The pharmacological extended.

### Summary

and furoates of a number of amino

alcohols have been prepared. Both types of esters have a low order of topical anesthetic activity but the furoates are frequently somewhat superior.

CINCINNATI, OHIO

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[CONTRIBUTION FROM THE CHEMISTRY DEPARTMENT OF FURMAN UNIVERSITY]

## sym-Tritolylbenzene

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benzenes have been made by the condensation of methyl phenyl ketones in various

Hydrogen chloride and sulfuric acid are used most often as the condensing agents. In condensations with the former, however, the reaction is slow; Claus<sup>1,d</sup> allowed six to eight days for condensations, and in a trial run with *p*-tolyl ketone saturated with dry hydrogen chloride we obtained only a 30% yield of *sym*-tritolylbenzene in five weeks. Condensations with sulfuric acid have been accomplished by low yields; Bernhauer<sup>1,g</sup> reported a 10% yield of *sym*-tritolylbenzene from the action of sulfuric acid and potassium pyrosulfate on *p*-tolyl ketone.

In order to the preparation of a series of *sym*-tritolylbenzenes, a systematic investigation was made of the effect of changes in time, temperature, and concentration on the yield of *sym*-tritolylbenzene from the action of sulfuric acid and potassium pyrosulfate or potassium acid sulfate on *p*-tolyl ketone. Condensations were made at various pressures ranging from room temperature to 100°C. The acid concentrations were varied from 10% to concentrated sulfuric acid per 10.0 cc. of ketone to 5.0 cc. of acid. The yields from the two potassium salts ranged from 10% to 70%.

At the lower concentrations of acid

*Ber.*, **7**, 1123 (1874); **26**, 1444 (1893); (b) Vorländer, *Ber.*, **28**, 2836-2844 (1929); (c) LeFèvre, *J. Chem. Soc.*, **1929**, 1405; (d) Claus, *J. prakt. Chem.*, [2] **41**, 405 (1890); (e) Odell, *J. Am. Chem. Soc.*, **55**, 102, II, 3101 (1931); (f) Odell, *This Journal*, **62**, 102, II, 3101 (1931); (g) Bernhauer, *J. prakt. Chem.*, **145**, 301-308 (1936).

and the lower temperatures much unchanged ketone was recovered, while at the higher concentrations the yield of triarylbenzene was reduced by the formation of a dark resinous mass. After more than fifty experiments the following conditions were found to give optimum yields in a reasonable time.

Exactly 10.0-g. samples of methyl *p*-tolyl ketone were placed in large Pyrex test-tubes (29 × 200 mm.) attached to reflux condensers and protected by calcium chloride tubes. Concentrated sulfuric acid, 0.2 cc. to 0.3 cc., and potassium pyrosulfate, 2.0 g., or potassium acid sulfate, anhydrous, 2.0 g., were added. The test-tubes were suspended in an oil-bath heated to 190° for six hours. The tubes were then removed, chilled, and 25 cc. of water added; the mixture was warmed and stirred with a heavy glass rod until the potassium salt dissolved. The *sym*-tritolylbenzene was separated and crystallized from hot acetic acid; yield 67-70% *sym*-tritolylbenzene, m. p. 170-171°.

Recognition is made of the assistance of Dr. E. Emmet Reid on this investigation.

### Summary

*sym*-Tritolylbenzene has been prepared in 67-70% yields by the condensation of methyl *p*-tolyl ketone with sulfuric acid and potassium pyrosulfate or potassium acid sulfate, anhydrous.

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