

possible in a hot needle flame. The glass stem remaining is then bent into a hook, using the tiny gas flame. The wax is then melted off and the bulb cleaned with an appropriate solvent. A bituminous base wax, such as Picein, is recommended over a shellac wax, such as deKhotinsky, because it is readily removed with carbon disulfide or carbon tetrachloride.

Buckets prepared in this manner may be made in any desired size. The author has prepared a number which are more than 1 cm. in diameter and which weigh less than 0.1 g. The weight of such buckets is usually about 0.07 g. These buckets will withstand a baking-out temperature of 450°.

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ALPHA-NAPHTHOLSULFONIC ACIDS AND DERIVATIVES. II

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In a series of articles it has been shown that phenols can be converted in phenolpolysulfonyl chlorides¹ by the action of chlorosulfonic acid. It also was noticed that the reagent caused four distinct types of reaction: namely, sulfonation, sulfochlorination, chlorination and oxidation. In certain cases the hydroxyl group of the phenol was found to be esterified prior to sulfonation, while in other instances condensation products such as the sulfonylides were isolated. The predominance of any one of the above-mentioned reaction types will depend on the time, temperature, quantity of reagent and the nature of the phenols.

The most striking aspect of the reaction between unsubstituted phenols and chlorosulfonic acid is the fact that so far it has been impossible to obtain the respective monosulfonyl chlorides.

From these facts a reaction mechanism for the action of chlorosulfonic acid on phenols (and phenolsulfonic acids) was postulated comprising as the first step the primary formation of the phenolsulfonic acid ester, and as the second step its rearrangement to form a phenolsulfonic acid; this is followed by reesterification of the hydroxy group and a second rearrangement to form the phenoldisulfonic acid in presence of sufficient amounts of the reagent.² Only when the phenoldisulfonic acid is formed and further

¹ J. Pollak and E. Gebauer-Fuelnegg, *Monatsh.*, **46**, 383 (1925); **46**, 499 (1925); **47**, 109 (1926); **47**, 511 (1926); **49**, 187 (1928); O. Litvay, E. Riesz and L. Landau, *Ber.*, **62**, 1863 (1929); E. Katscher, *Monatsh.*, **56**, 381 (1930).

² E. Gebauer-Fuelnegg and A. Schlesinger, *Ber.*, **61**, 781 (1928).