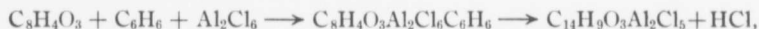


FRIEDEL AND CRAFTS' REACTION—THE PREPARATION OF ORTHOBENZOYL-BENZOIC ACID AND BENZOPHENONE.

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Orthobenzoyl-benzoic acid had been prepared by several investigators but was first prepared by means of the reaction between phthalic anhydride, benzene and aluminium chloride, by Friedel and Crafts.¹ Heller² showed that the aluminium chloride was not a catalytic agent and that a good yield depended on the phthalic anhydride and the aluminium chloride being used in the proportion of their formula weights (using Al_2Cl_6). In a later article Heller and Shülke³ suggested that the reaction might be represented as taking place in two stages:



the latter compound with hydrochloric acid giving orthobenzoyl-benzoic acid. Heller obtained a yield of 93%, or counting in the recovered phthalic acid 97%, and in two experiments obtained 0.87 mol and 0.93 mol, respectively, of hydrogen chloride for each mol of phthalic anhydride used. Heller also states that if one-half the usual amount of aluminium chloride is used the yield is reduced to less than one-half.

The following experiments on the preparation of orthobenzoyl-benzoic acid and benzophenone, using varying quantities of the reagents, were begun by the first named author and finished by the second.

The aluminium chloride used was Kahlbaum's "sublimed" and had been kept in sealed bottles in the laboratory for more than a year; preliminary experiments showed that this gave as good yields as were obtained when the aluminium chloride had been freshly sublimed. The phthalic anhydride and benzene were obtained from the same firm but it was found better to resublime the phthalic anhydride.

The apparatus consisted of a flask which was connected with a reflux condenser and had also an inlet for dry air; the condenser was connected with a U-tube containing calcium chloride and then with two Liebig bulbs containing potassium hydroxide solution. Dry air could thus be drawn through the apparatus, and the hydrogen chloride given off determined. Five grams of phthalic anhydride and 18–20 cc. benzene were placed in the flask and heated until the anhydride was dissolved, after cooling, 9 g. aluminium chloride were added gradually; this was allowed to stand

¹ *Ann. chim. phys.*, [6] **14**, 446 (1888).

² *Z. angew. Chem.*, **19**, 669 (1906).

³ *Ber.*, **41**, 3627 (1908).