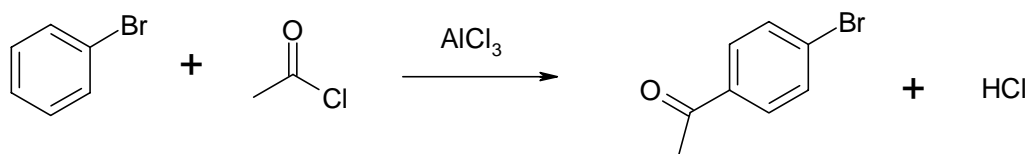


Friedel-Crafts acylation: **p-Bromoacetophenone***Macroscale*

Chemicals: bromobenzene acetyl chloride:
 AlCl₃ dry

Procedure:

A 250 ml round-bottom flask is fitted with a KPG-stirrer, an addition funnel and a reflux condenser. During the reaction HCl-gas is evolved which is absorbed in a sodium hydroxide solution in a beaker. For that purpose the top of the condenser is connected via a flexible tube with a funnel which dips circa 5 mm into the sodium hydroxide solution.

Place 20.0 g (150 mmole) dry aluminium trichlorid into the flask and add cautiously with stirring 19.6 g (125 mmole) bromobenzene. Warm up the mixture to 50 °C and add at this temperature 8.3 g (130 mmol) acetyl chloride dropwise. After complete addition stirring is continued at 50 °C for 5 hours.

Isolation and purification:

The cooled mixture is cautiously poured on 100 g of ice, the flask is rinsed with 20 ml MTBE which is added to the ice. When the mixture contains solid aluminium hydroxide, add conc. hydrochloric acid until the aluminium hydroxide dissolves. In a separatory funnel the organic layer is separated and the aqueous layer is extracted twice with 20 ml MTBE. The combined organic extracts are washed with water, then with 2 % sodium hydroxide solution and again with water. After drying the organic layer over potassium carbonate the solvent MTBE is removed at a rotary evaporator und the product distilled at reduced pressure (15 - 20 mbar). When the product becomes solid during the distillation, stop cooling with water, if necessary warm up with a heat gun.

Literature yield: 70 %

Literature boiling point: 130 °C at 20 mbar

melting point: 50 °C