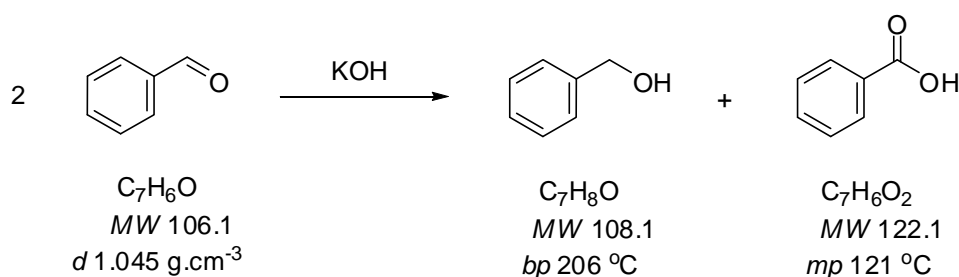


Cannizzaro reaction (Benzaldehyde reduction-oxidation)



Add into a wide neck 250 ml Erlenmeyer flask potassium hydroxide solution (a cooled solution of 14.5 g KOH and 14 ml water)¹ and benzaldehyde² (16 g; 0,15 mol; 15 ml). After the addition shake the sealed flask (do not use ground neck flasks) intensively until a strong emulsion is forming (20-25 min). Let this emulsion stay in the closed flask at room temperature until the next lab practice³.

At the next lab add sufficient amount of water to dissolve the forming potassium benzoate,⁴ then pour the forming slurry to a separatory funnel⁵ and extract it with dichloromethane (3×15 ml). Store the aqueous phase and wash the unified dichloromethane layers with saturated sodium metabisulfite solution⁶ (2×10 ml), 10% sodium bicarbonate solution (10 ml) and water (10 ml). After drying⁷ remove the dichloromethane in vacuum rotary evaporator and purify the residual oil by distillation in vacuum. Usual yield is 6.5 g (86 %) of benzyl alcohol. Pour the extracted (and stored) aqueous layer into ice-cooled acid (50 g of ice, 40 ml of water and 40 ml of conc. hydrochloric acid). Collect the precipitating benzoic acid by filtration, wash it with a few amount of cold water and recrystallize it from water (ca. 15 ml/g crude product). The recrystallized product is white crystalline solid (6.6 g; 78 %; m.p 121 °C).

Notes

- (1) Add the solid KOH in small portions to stirred and cooled water. The potassium hydroxide solution can be substituted by a solution of sodium hydroxide (16 g) in water (13 ml).
- (2) The benzaldehyde should be free from benzoic acid.
- (3) The minimum required reaction time at room temperature is 24 h.
- (4) Usually, 50-55 ml of water is sufficient to dissolve the solid. However, the solution process is not immediate. To dissolve the solid effectively, the bigger clumps should be crashed by appropriate devices.
- (5) After the transfer, washing the flask with a few amount of water and dichloromethane can reduce the loss of products.
- (6) Washing with sodium metabisulfite solution removes the residual benzaldehyde.
- (7) Drying should be made over anhydrous potassium carbonate or sodium sulfate. Use of CaCl₂ should be avoided due to its complex forming ability with alcohols.