

2013 Reaction of cinnamic acid with thionyl chloride to cinnamoyl chloride



Classification

Reaction types and substance classes

reaction of the carbonyl group in carboxylic acids
 carboxylic acid, carboxylic acid chloride

Work methods

Working with moisture exclusion, stirring with magnetic stir bar, draining of gases, working with wash bottles, distilling under reduced pressure, heating with oil bath

Instruction (batch scale 100 mmol)

Equipment

100 mL three-neck flask, reflux condenser, bubble counter, 2 wash bottles, distillation apparatus, heatable magnetic stirrer, magnetic stir bar, hot-air dryer, vacuum pump, oil bath

Substances

<i>trans</i> -cinnamic acid (mp 135-136 °C)	14.8 g (100 mmol)
thionyl chloride (freshly distilled) (bp 76 °C)	17.8 g (10.9 mL, 150 mmol)
aqueous solution of sodium hydroxide (20%)	100 mL

Reaction

The reaction apparatus consists of a 100 mL three-neck flask with magnetic stir bar and reflux condenser. For draining of the evolving gases HCl and SO₂ the reflux condenser is connected consecutively to a bubble counter filled with paraffin oil, an empty safety wash bottle and a wash bottle filled with 100 mL of an aqueous solution of sodium hydroxide (20%).

The reaction flask is charged with 17.8 g (10.9 mL, 150 mmol) freshly distilled thionyl chloride. Whilst stirring, 14.8 g (100 mmol) *trans*-cinnamic acid is added in several portions by means of a powder funnel. The last added portions are firstly insoluble, accordingly stirring with the magnetic stirrer is temporarily not possible. The remaining flask openings are closed with stoppers and the reaction mixture is initially slowly heated whilst stirring (as far

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