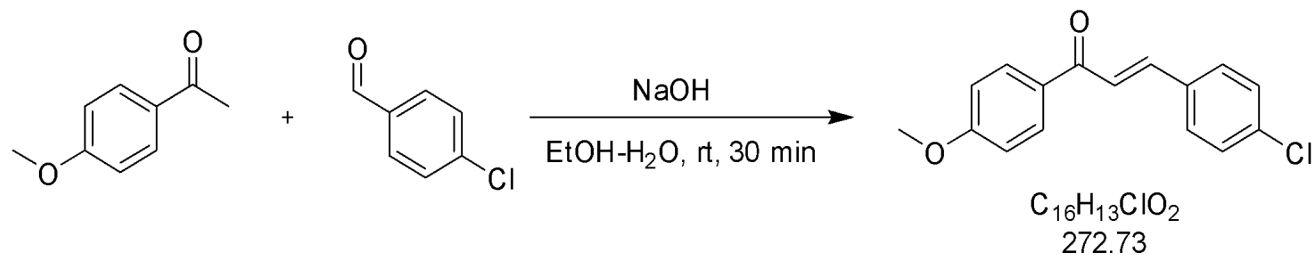


Preparation of 4-chloro-4'-methoxychalcone from 4'-methoxyacetophenone and 4-chlorobenzaldehyde

Scheme



Hazards

Sodium hydroxide solution is caustic. Recrystallisation involves heating flammable solvent.

Reagent quantities

Quantity	4'-Methoxy-acetophenone	4-Chloro-benzaldehyde	NaOH	EtOH
Source	Aldrich	Acros	Lab bottles	Solvent bottles
fw / g mol ⁻¹	150.17	140.57	-	-
Mass / g	1.50	1.55	-	-
mmol	10.0	11.0	-	-
Equivalents	1.0	1.1	-	-
Concentration / mol dm ⁻³	-	-	2	-
Density / g mL ⁻¹	-	-	-	-
Volume / mL	-	-	20	20
Taken	1.4978 g	1.56 g	Via 25 mL measuring cylinder	Via 25 mL measuring cylinder

Plan

- 4'-Methoxy-acetophenone, ethanol and 4-chloro-benzaldehyde to add into 100 mL round bottomed flask.
- Add NaOH solution and stir at rt for 30 minutes.
- Quench reaction by pouring into ice-water (50 g + 50 mL).
- Collect precipitate via vacuum filtration (sinter).
- Recrystallise from ethanol.
- 100% yield = 2.73 g. mp 125-126 °C (J. Med. Chem., 2009, 52, 2818).

Procedure

2026-10-08

- 1000: Weighed 4'-methoxy-acetophenone (pale-yellow solid) and transferred into 100 mL round bottomed flask.
- 1005: Clamped flask over stirrer hotplate, ethanol added and began stirring the resulting suspension.

- 1010: Added the 4-chlorobenzaldehyde (colourless solid) in a single portion. No significant change in the appearance of the suspension.
- 1015: Sodium hydroxide solution added in a single portion. Solution began to turn a milky colour.
- 1030: Reaction is a colourless opaque suspension with significant colourless precipitate forming.
- 1045: Flask removed and contents poured into a 250 mL beaker containing ice (50 g) and water (50 mL). Contents of flask rinsed into beaker with further portions of water.
- 1100: Most of the ice had melted and filtered mixture through a sinter and Buchner flask (250 mL) setup. Washing beaker contents with additional water.
- 1110: Buchner flask emptied of water and powder allowed to dry over vacuum. Solid broken up on sinter with spatula. Slightly off-white powder.
- 1115: Weighed 100 mL conical flask (136.7840 g) ready for crude compound.
- 1120: Transferred crude compound into conical flask (139.7045 g).
- 1300: Begin recrystallisation from hot ethanol.
- 1310: Took approximately 25 mL to dissolve. Allowing to cool slowly on hotplate. Solution is slightly yellow in colour, all material dissolved.
- 1330: Has cooled to room temperature. Large colourless flakes have formed. Placed into ice-bath to cool further along with 100 mL conical flask approx. 50% filled with ethanol.
- 1335: Vacuum filtration (sinter) set up.
- 1340: Collected via filtration. Washing with portions of ice-cold ethanol. Product are shiny colourless flakes.
- 1345: Mother liquor emptied from Buchner flask and crystals allowed to dry over vacuum.
- 1355: Watch glass tared (74.3265 g).
- 1400: Product collected (76.5028 g).
- 1430: Melting point range recorded. Ramped from 110 °C. 128-130 °C.
- 1500: IR spectrum recorded (ctlirpc1_20261008_151108).

Results

2026-10-09

Yield

- Crude: $139.7045 - 136.7840 = 2.92 \text{ g}$
 $2.92 \text{ g} / 2.73 \text{ g} = 107\%$. Probably quite wet with water.
- Purified: $76.5028 - 74.3265 = 2.18 \text{ g}$
 $2.18 / 2.73 \text{ g} = 80\%$ yield.

IR

- IR shows carbonyl stretch at 1654 cm^{-1} .