

Synthesis of benzalacetophenone

Summary of the analysed protocols

$\text{C}_6\text{H}_5\text{CHO} + \text{C}_6\text{H}_5\text{COCH}_3 \rightarrow \text{C}_6\text{H}_5\text{CH}=\text{CHCOC}_6\text{H}_5 + \text{H}_2\text{O}$	(1)
Protocol A¹	
Reaction (R₁): equation (1), 24% exc. benzaldehyde, methanol, 40% sodium hydroxide solution and hydrochloric acid (auxiliary substances), T ~ 0 °C	
Isolation (I₁): filtration (suction) → washing (water → cold ethanol) → drying (suction → oven at T < 50 °C)	
Purification (Pu₁): not prescribed	
Protocol B²	
Reaction (R₂): equation (1), stoichiometric proportions of acetophenone and benzaldehyde, 10% sodium hydroxide solution and ethanol (auxiliary substances), T ~ 0 °C	
Isolation (I₂): filtration (suction) → washing (water) → drying (air)	
Purification (Pu₁): recrystallization (ethanol)	
Protocol C³	
Reaction (R₃): equation (1), stoichiometric proportions of acetophenone and benzaldehyde, sodium hydroxide, water and ethanol (auxiliary substances), T ~ 0 °C	
Isolation (I₃): filtration (suction) → washing (cold water → ethanol) → drying (air)	
Purification (Pu₁): ≡ Pr B	
Protocol D^{4,5}	
Reaction (R₃): ≡ Pr C (scale enlarged 100 times)	
Isolation (I₄): filtration (suction) → washing (water → cold ethanol) → drying (air)	
Purification (Pu₂): recrystallization (hot ethanol)	
Protocol E⁶	
Reaction (R₃): ≡ Pr C (scale enlarged 10 times)	
Isolation (I₅): filtration (suction) → washing (cold water → cold ethanol) → drying (air)	
Purification (Pu₂): ≡ Pr D	
Protocol F⁷	
Reaction (R₄): equation (1), stoichiometric proportions of acetophenone and benzaldehyde, sodium hydroxide, water and ethanol (auxiliary substances), T ~ 0 °C	
Isolation (I₆): filtration (suction) → drying (suction)	
Purification (Pu₃): recrystallization – dissolution (ethanol) → filtration (suction)	
Protocol G⁸	
Reaction (R₅): equation (1), stoichiometric proportions of acetophenone and benzaldehyde, sodium hydroxide and water (auxiliary substances), room temperature	
Isolation (I₇): filtration (suction) → washing (water) → drying (air)	
Purification (Pu₁): ≡ Pr B	

^a → – Sequential

References

- (1) Universidade de Aveiro, <http://www.ua.pt/ensino/PageDisc.aspx?id=2528> (accessed April 2011).
- (2) Universidade Federal de Santa Maria, <http://w3.ufsm.br/lab2228/docs/Tecnicas-aulas-experimentais-pdf.pdf> (accessed December 2012).
- (3) Adam Mickiewicz University, <http://www.staff.amu.edu.pl/~psorg/serp.pdf> (accessed April 2013).
- (4) Blatt, A.H.; Gilman, H. *et al. Organic Syntheses, collective volume I - 2nd edition*. John Wiley & Sons, Inc: New York, 1958, pp. 78-80.
- (5) Fourneau, M.E. *et al. Synthèses Organiques*. Masson et C^{ie}, Éditeurs: Paris, 1935, pp. 134-135.

- (6) Vogel, A.I. *A Text-Book of Practical Organic Chemistry*. Longmans, Green and Co, Ltd: London, 1948, pp. 681-682.
- (7) Durst, H.D.; Gokel, G.W. *Experimental Organic Chemistry - 2nd edition*. McGraw-Hill Book Company: New York, 1987, pp. 428-429.
- (8) Palleros, D.R. Solvent-Free Synthesis of Chalcones. *J. Chem. Educ.* **2004**, *81*, 1345-1347.