

Synthesis of cobalt(III) acetylacetonate

Summary of the analysed protocols

$2\text{CoCO}_3 + 6(\text{Hacac}) + \text{H}_2\text{O}_2 \rightarrow 2[\text{Co}(\text{acac})_3] + 2\text{CO}_2 + 4\text{H}_2\text{O} \quad (1)$
Protocol A¹
<p>Reaction (R₁): equation (1), 208% exc. acetylacetonate, 738% exc. hydrogen peroxide 10%, T < 100 °C Isolation (I₁): cooling (ice-salt bath) → filtration (suction) → drying (oven at 110 °C) Purification (Pu₁): recrystallization – dissolution (hot toluene and petroleum ether) → decantation → heating → cooling (ice bath) → filtration → washing (petroleum ether) → drying (air)</p>
Protocol B²
<p>Reaction (R₁): ≡ Pr A (scale decreased to half) Isolation (I₁): ≡ Pr A Purification (Pu₂): recrystallization – dissolution (toluene and petroleum ether or heptane) → heating → filtration → cooling (ice-salt bath) → filtration (suction) → drying (air)</p>
Protocol C³
<p>Reaction (R₁): ≡ Pr B Isolation (I₁): ≡ Pr A Purification: not prescribed</p>
Protocol D⁴
<p>Reaction (R₁): ≡ Pr A Isolation (I₂): cooling (ice-salt bath) → filtration (suction) → washing (cold ethanol) → drying (oven at 110 °C) Purification (Pu₃): recrystallization – dissolution (boiling toluene and heptane) → cooling (ice bath) → filtration → drying (air, oven at 110 °C)</p>
Protocol E⁵
<p>Reaction (R₁): ≡ Pr A Isolation (I₃): ≡ Pr D (but is done drying in the air, instead of using the oven) Purification (Pu₄): ≡ Pr D (but is done drying in the air, instead of using the oven)</p>
Protocol F⁶
<p>Reaction (R₁): ≡ Pr A (scale enlarged to double) Isolation (I₁): ≡ Pr A Purification (Pu₅): recrystallization – dissolution (boiling benzene and petroleum ether or heptane) → cooling (ice-salt bath) → filtration → drying (air)</p>

^a → – Sequential

References

- (1) Faculdade de Ciências e Tecnologia da Universidade de Coimbra, <https://woc.uc.pt/quimica/getFile.do?tipo=2&id=1438> (accessed February 2011).
- (2) University of Malaya, <http://www.kimia.un.edu.my/images/kimia/lab%20manual/level%202/Lab%20Manual%20Yr%202%20Inorganic.pdf> (accessed May 2011).
- (3) Glidewell, C.; “Metal Acetylacetonate Complexes: Preparation and Characterization” in Woollins, J., Ed.; *Inorganic Experiments*, 2nd ed., Wiley-VCH, Weinheim, 2003; Exp. 3.16.
- (4) Shalhoub, G.M. Co(acac)₃: Synthesis, Reactions and Spectra. *J. Chem. Educ.* **1980**, 57 (7), 525-526.
- (5) Radboud University of Nijmegen, www.orgchem.science.ru.nl/molmat/mm-web/srm4.doc (accessed April 2011).
- (6) Moeller, T. *et al. Inorganic Syntheses – Volume V*. McGraw-Hill Book Company, Inc: New York, 1957, pp. 188-189.