

Synthesis of chromium(III) acetylacetonate

Summary of the analysed protocols

$\text{CO}(\text{NH}_2)_2 + \text{H}_2\text{O} \rightarrow 2\text{NH}_3 + \text{CO}_2$ (1) $\text{CrCl}_3 + 3(\text{Hacac}) + 3\text{NH}_3 \rightarrow [\text{Cr}(\text{acac})_3] + 3\text{NH}_4\text{Cl}$ (2)
Protocol A¹
Reaction (R₁): equations (1) and (2), 100% exc. acetylacetonate, 10 g of urea, T ~ 80-90 °C Isolation (I₁): cooling → filtration → drying (air) Purification (Pu₁): recrystallization – dissolution (hot cyclohexane) → cooling → filtration → drying (air)
Protocol B²
Reaction (R₂): equations (1) and (2), 89% exc. acetylacetonate, 1.5 g of urea, T ~ 100 °C Isolation (I₁): ≡ Pr A Purification (Pu₂): recrystallization – dissolution (cyclohexane) → heating (steam bath) → cooling → filtration (suction) → drying (suction, air)
Protocol C³
Reaction (R₃): equations (1) and (2), 127% exc. acetylacetonate, 6 g of urea, T < 100 °C Isolation (I₁): ≡ Pr A Purification: not prescribed
Protocol D^{4,5}
Reaction (R₄): equations (1) and (2), 165% exc. acetylacetonate, 500 mg of urea, T < 100 °C Isolation (I₂): cooling → filtration (suction) → washing (distilled water) → drying (air) Purification: not prescribed
Protocol E⁶
Reaction (R₄): ≡ Pr D (scale enlarged to double) Isolation (I₃): ≡ Pr D (but different amount of water for washings) Purification: not prescribed
Protocol F⁷
Reaction (R₅): equations (1) and (2), 100% exc. acetylacetonate, 20 g of urea, T ~ 100 °C Isolation (I₄): filtration (suction) → drying (air) Purification (Pu₃): recrystallization – dissolution (hot benzene and petroleum ether) → cooling (ice-salt bath) → filtration → drying (air)
Protocol G⁸
Reaction (R₆): equations (1) and (2), 92% exc. acetylacetonate, 5 g of urea, T < 100 °C Isolation (I₅): filtration → washing (water, ethanol, isopropyl alcohol) → drying (oven at 110 °C) Purification (Pu₄): recrystallization – dissolution (hot toluene and hexane) → cooling (ice bath) → filtration (suction) → washing (cold hexane) → drying (air)

^α → – 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) Radboud University of Nijmegen, www.orgchem.science.ru.nl/molmat/mm-web/srm4.doc (accessed April 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) Szafran, Z.; Pike, R.M.; Singh, M.M. *Microscale Inorganic Chemistry – A Comprehensive Laboratory Experience*. Wiley: New York, 1991, pp. 224-229.
- (5) Colby College, <http://www.colby.edu/chemistry/Grants/CH141L4CrFall2002.pdf> (accessed May 2011).

- (6) University of Massachusetts Amherst,
<http://people.chem.umass.edu/pkhalifah/chem242/242-S2007-EP/2007-5-CrCF-EP.pdf> (accessed May 2011).
- (7) Moeller, T. *et al.* *Inorganic Syntheses – Volume V*. McGraw-Hill Book Company, Inc: New York, 1957, pp. 130-131.
- (8) Chemicando,
http://www.chimicando.it/esperimenti/chimica_inorganica/E6-Sintesi_del_tris-acetilacetato_di_CrIII.pdf
(accessed May 2011).