

Contributed by Lynne Bridget McCusker

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Type Material: $[\text{Si}_{64}\text{O}_{128}] \cdot 2(\text{Cp}^*)_2\text{CoF}_{0.75}\text{OH}_{0.25}$
(Cp* = pentamethylcyclopentadienyl)

Method: T. Wessels, C. Baerlocher, L.B. McCusker, and E.J. Croyghton [1]

Batch Composition: 100 SiO₂ : 40 [(Cp*)₂Co]F : 3125 H₂O

Source Materials

distilled water
silica cabosil M5 (Aldrich)
[(Cp*)₂Co]OH aqueous solution (8.95 wt%)
HF aqueous solution (38 wt % J.T. Baker)

Batch Preparation (for 2.05 g product)

- (1) [27.8 g of Cabosil M5 + 68.4 g of water], stir until homogeneous to prepare a silica slurry
- (2) [6.60 g of sample (1) + 49.15 g of 8.95 wt % aqueous [(Cp*)₂Co]OH solution], stir with a magnetic stirrer for 1.5 h
- (3) [0.67 g of aqueous 38 wt % HF (J. T. Baker) in about 70 g of water + (2)^a], vigorous stirring for 20 min
- (4) concentrate (3) by removing excess water under reduced pressure

Crystallization

Vessel: Teflon-lined autoclave
Temperature: 170° C
Time: 25 days
Agitation:

Product Recovery

- (1) Filter and wash extensively with water
- (2) Yield: 2.05 g

Product Characterization

XRD: pure phase
Elemental analysis: 32Si:1.03Co: 19.13C:0.75F
Crystal size and habit: needlelike crystallites

Reference

- [1] T. Wessels, C. Baerlocher, L.B. McCusker, and E.J. Croyghton, J. Am. Chem. Soc., 121, (1999) 6242
- [2] Stacey I. Zones and Cong-Yan Chen. U.S. Pat. 6,103,215 (2000)

Notes

- a. HF solution was slowly added to (2)