AIPO₄-16

AI(50), P(50)

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Type Material (AI₁₀P₁₀O₁₀)Q_{2.0}F_{1..6}: 3.0 H₂O ^a (Q= quinucidine)

Method C. Schott-Darie, J. Patarin, P. Y. Le Goff, H. Kessler and E. Benazzi [1]

Batch Composition 1 P_2O_5 : 1 Al_2O_3 : 1 Q : 1 HF : 60 H_2O

Source Materials

AST

distilled water phosphoric acid (Fluka, 85% H₃PO₄) aluminum isopropoxide (Aldrich, 98%) quinuclidine C₇H₁₃N (Fluka, 97%) hydrofluoric acid (Prolabo, 40%)

Batch Preparationb (for 1.5 g of as-synthesized product)

- (1) [4.00 g water + 2.31 g phosphoric acid + 4.17 g aluminum isopropoxide], stir until homogenized
- (2) [6.15 g water + 1.14 g quinucidine], stir until dissolved
- (3) [(1) + (2) + 0.50 g hydrofluoric acid], stir for 2 minutes. Gel pH= 7 to 7.5

Crystallization

Vessel: Teflon-lined stainless steel autoclave (50 cm³) Temperature: 150°C Tine: 24 hours Agitation: none Final pH: approximately 8

Product Recovery

- (1) Dilute the reaction mixture with distilled water
- (2) Filter or centrifuge
- (3) Wash until the pH of the filtrate is 5.5 to 6
- (4) Dry at 60-70°C overnight
- (5) Yield: 50% based on aluminum (as-synthesized product containing quinudidine and some water) ^d

Product Characterization

XRD: AST (only crystalline phase), Space group 14, $a_0 = 9.3423(1)$ Å $c_0 = 13.4760(2)$ Å ^e Elemental Analysis (wt%): Al₂O₃ = 32.8, P₂O₅ = 44.8, F = 1.9, (C₇H₁₃)N = 15.4, H₂O = 3.4 Crystal Size and Habit: tetrahedra, 0.5 to 3 µm

References

- [1] C. Schott-Darie, J. Patarin, P. Y. Le Goff, H. Kessler, E. Benazzi, Micropor. Mater. 3 (1994) 123
- [2] J. M. Bennett, R. M. Kirchner, Zeolites 11(1991) 502

Notes

- a. The Q/F molar ratio is lower than 1. Part of the quinucidine is either not protonated or OH⁻ groups are present in order to get a neutral material.
- b. The starting mixture is prepared in a polyethylene vessel.
- c. The reaction is exothermic.
- d. Alter calcination (removal of the organic and fluoride species) the cubic form [2] of AIPO₄-16 is obtained.
- e. According to reference [1].