

VFI

VPI-5 (TBA method)

Al(50), P(50)

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Verified by A. Karlsson and W. Schmidt

Type Material: [Al₁₈P₁₈O₇₂]: 42 H₂O

Method: H. He, J. Klinowski [1]

Batch Composition: 1.00 Al₂O₃: 1.00 P₂O₅: 50 H₂O: 1.12 TBA-OH_a
(TBA-OH = tetrabutylammonium hydroxide)

Source materials

distilled water

pseudoboehmite (Catapal B, 68.01 wt% Al₂O₃)

phosphoric acid (Aldrich, 88.30 wt% H₃PO₄)

tetrabutylammonium hydroxide (Fluka, 58.08 wt% TBA-OH)

Batch Preparation

- (1) [61.63 g water + 15.00 g pseudoboehmite], disperse alumina in water
- (2) [(1) + 22.20 g phosphoric acid], stir until homogeneous (for several minutes) and age for 2 hours without stirring
- (3) [(2) + 50.04 g tetrabutylammonium hydroxide], stir for 2 hours

Crystallization

Vessel: Teflon-lined autoclave

Time: 20 hours

Temperature: 150°C

Agitation: none

Product Recovery

- (1) Dilute the reaction mixture with distilled water
- (2) After the crystals precipitate, decant the upper layer of liquid and discard. Repeat the operation three times
- (3) Filter and wash the crystals with distilled water
- (4) Dry in an air oven below 50°C

Product Characterization

XRD: VFI ($a_0 = 18.9752$ Å, $C_0 = 8.1044$ Å, space group P6₃); competing phases: AlPO₄-H₂ and H₃ [2]

Elemental Analysis: 0.006 TBA+: Al₂O₃: P₂O₅

Crystal Size and Habit: needle-like and aggregated into bundles, - 10 μm. dia.

References

- [1] H. He, J. Klinowski, J. Phys. Chem. 98 (1994) 1192
- [2] F. d'Yvoire, Bull. Soc. Chim. 372 (1961) 1762

Note

- a. The amount of water quoted includes water in pseudoboehmite (100% - wt% Al_2O_3), phosphoric acid (100% - wt% P_2O_5), and tetrabutylammonium hydroxide (100% - wt% TBA-OH).