

AEL

AlPO₄-11

Al(50), P(50)

Contributed by Kristin Vinje

Verified by J. Campelo and J. Kornatowski

Type Material [Al₂₀P₂₀O₂₀] : wH₂O

Method R Szostak, B. Duncan, R. Aiello, A. Nastro, K. Vinje, K. P. Lillerud [1]

Batch Composition 1.0 Al₂O₃ : 1.25 P₂O₅ : 2.37 DPA : 1.80 HF : 156 H₂O (DPA = di-n-propylamine)

Source Materials

deionized water

aluminum hydroxide (Aldrich 23918-6, 50-57.5% Al₂O₃)^a

phosphoric acid (Fisher, 85% H₃PO₄)

di-n-propylamine (DPA)(Kodak, 100%)

hydrofluoric acid (Fisher, 48% HF)

Batch Preparation (for 4 g dry product)

- (1) [20.0 g water + 7.8 g aluminum hydroxide] stir well
- (2) [(1) + 14.4 g phosphoric acid]; add acid dropwise and stir until effervescence is completed
- (3) [(2) + 100 g water]; dilute and stir
- (4) [(3) + 12.0 g DPA]; add amine dropwise and stir
- (5) [(4) + 10.0 g water 3.75 g hydrofluoric acid]: stir for two hours^b

Crystallization

Vessel: Teflon-lined steel autoclave

Temperature: 145°C

Time: 18 hours

Agitation: none

Product Recovery

- (1) Cool autoclave rapidly.
- (2) Filter immediately to recover solids and wash.
- (3) Dry overnight at room temperature.
- (4) Yield: ~70%^c

Product Characterization

XRD: AEL only crystalline phase: no other crystalline phases or amorphous material detected

Elemental Analysis: (atomic %): 15.5 Al, 13.9 P, Al/P = 1.1^d

Crystal Habit: Bow-tie crystals constructed of long needles (or needles ~ 1 mm long)^c

Reference

- [1] R. Szostak, B. Duncan, R Aiello, A. Nastro, K. Vinje, K. P. Lillerud, in *Synthesis of Microporous Materials*, M. Occelli, H. Robson, (eds.), Van Nostrand Reinhold, New York (1992), pp 240-7

Notes

- a. Consistently good results have been obtained using the Aldrich Al-source, but difficulties were encountered using other aluminum sources.
- b. Should produce a clear solution; initial pH = 5.5, final pH = 6.0
- c. After 18 hours the yield increases to 50-75% at the expense of the crystal size with smaller 5 to 10 μm crystals resulting.
- d. Determined on a CAMECA SX 100 Microbeam.