

**CHA**

**SAPO-34**

**Al(47), P(32), Si(21)**

**Contributed by** A. M. Prakash

**Verified by** J. López Nieto and N. Tusar

**Type Material** mR[Al<sub>17</sub>P<sub>12</sub>Si<sub>7</sub>O<sub>12</sub>] (R = morpholine)

**Method** A. M. Prakash, S. Unnikrishnan [1]

**Batch Composition** Al<sub>2</sub>O<sub>3</sub> : 1.06 P<sub>2</sub>O<sub>5</sub> : 1.08 SiO<sub>2</sub> : 2.09 R : 66 H<sub>2</sub>O <sup>a,b</sup>

**Source Materials**

distilled water

phosphoric acid (Merck, 85%)

pseudoboehmite (Vista Catapal-B, 70% Al<sub>2</sub>O<sub>3</sub>)

fumed silica (Degussa Aerosil-200 99+% SiO<sub>2</sub>)

morpholine (Aldrich, 99% C<sub>4</sub>H<sub>9</sub>O)

**Batch Preparation** (for 20 g product) <sup>c</sup>

- (1) [18.0 g water + 15.37 g phosphoric acid], mix
- (2) [(1) + 9.20 g pseudoboehmite], add slowly (2 hours) with stirring
- (3) [(2) + 10 g water], stir thoroughly for 7 hours
- (4) [4.09 g fumed silica + 11.62 g morpholine + 15 g water], mix thoroughly
- (5) [(3) + (4)], add (4) dropwise to (3) while stirring
- (6) [(5) + 24 g water], stir thoroughly for 7 hours; pH of gel = 6.4 to 7.5

**Crystallization**

Vessel: 150 mL Teflon-lined autoclave

Incubation: 24 hours at 38 °C without agitation <sup>d</sup>

Temperature: 200 °C

Time: 24 hours

Agitation: none

**Product Recovery**

- (1) Decant the mother liquor
- (2) Dilute with distilled water and filter
- (3) Wash 3 to 4 times with distilled water
- (4) Dry at 100 °C for 6 hours
- (5) Yield: 98% based on alumina

**Product Characterization**

XRD: SAPO-34 (CHA) [1], a<sub>0</sub> = 13.78 Å, c<sub>0</sub> = 14.85 Å; competing phase: AlPO<sub>4</sub> (cristobalite) when silica and/or template concentration is low.

Elemental Analysis: 1.0 Al<sub>2</sub>O<sub>3</sub>: 0.68 P<sub>2</sub>O<sub>5</sub>: 0.87 SiO<sub>2</sub>: 0.59 R: 1.07H<sub>2</sub>O

Crystal size and habit: 5 to 20 μm crystals with cubic-like rhombohedral morphology. [1]

## Reference

- [1] A. M. Prakash, S. Unnikrishnan, J. Chem. Soc. Faraday Trans. 90 (1994) 2291

## Notes

- a. H<sub>2</sub>O includes water from phosphoric acid, pseudoboehmite and added water.
- b. Concentration of SiO<sub>2</sub> and organic template can vary over a range without affecting the phase purity. At low concentration of silica (SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ≤ 0.3) or template (R/Al<sub>2</sub>O<sub>3</sub> ≤ 1.5), however, dense phase AlPO<sub>4</sub>-cristobalite co-crystallizes with SAPO-34.
- c. Use Teflon or stainless steel equipment throughout.
- d. Although SAPO-34 crystallizes without aging, the crystallinity of the resulting product can be improved by aging.