AEI SAPO-18 (TEA method) P(50), Al(48), Si(02)

Contributed by Rune Wendelbo

Verified by E Dumitriu and C. Round

Type Material $[Al_{23.2}SiO_{0.8}P_{24}O_{96}]$[b]

Method: R Wendelbo, D. Akporiaye, A. Andersen, I. M. Dahl, H. B. Mostad [1]

Batch Composition Al_2O_3 : 0.98 P_2O_5 : 0.015 HCl : 0.075 SiO_2 : $(TEA)_2O$: 70 H_2O : 2.8 i-C_3H_7OH[c] (TEA = tetraethylammonium)

Source Materials

distilled water
aluminum isopropoxide (Jansen, 98+%)
phosphoric acid (85%)
hydrochloric acid (3 7%)
silica sol (DuPont Ludox LS-30, 30% SiO_2)
tetraethylammonium hydroxide (Aldrich, 40 % (TEA)OH)[d]

Batch Preparation: (for 13.8 g dry product)

(1) [108 g water + 81.6 g aluminum isopropoxide], mix in a one-liter poly-propylene bottle and shake for 1 minute
(2) [(1) + 45 g phosphoric acid], shake for 1 minute then cool under running tap water
(3) [(2) + 0.6 g hydrochloric acid], shake bottle[e]
(4) [(3) + 3.0 g silica sol], shake bottle. filter gel for 10 minutes (water suction). About 100 g filtrate is removed and discarded. Transfer one-third of filter cake gel to a 250 mL plastic bottle
(5) [One-third (4) + 49 g tetraethylammonium hydroxide solution], shake

Crystallization

Vessel: 200 mL stainless steel, Teflon-lined autoclave (Berghof)
Incubation: 12 hours at room temperature
Time: 120 hours
Temp: 215°C
Agitation: gentle [f]

Product Recovery

(1) Recover solid product by centrifugation.[g]
(2) Wash once with distilled water; recover product by centrifugation[g]
(3) Dry overnight at 100°C
(4) Calcine for 4 hours at 550°C in flowing, dry air
(5) Store under nitrogen. Yield near 100%

Product Characterization

XRD: fully crystalline AEI; competing phase AFI and CHA[h]
Elemental Analyses: 0.84% Si, 19.9% Al, 26.6% P [i]
Crystal size and habit: square platelets, 0.1-2.0 μm x 0.1 μm thick[j]

Reference

[1] R Wendelbo, D. Akporiaye, A. Andersen, I. M Dahl, H. B. Mostad, Appi. Catal. A: General 142 (1996) L197

Notes

a. Mg AIPO-18 and Zn APO-18 were synthesized like SAPO-18 by using Mg and Zn nitrates in place of colloidal silica in equivalent amounts on a molar basis. The Mg and Zn nitrates were dissolved in the phosphoric acid 20 m prior to mixing with the other reagents. Products showed 0.14 Mg and 0.10 Zn (nmol/g).

b. Based on Si content; excess P is unexplained.

c. It is assumed that water, HC1 and isopropanol are lost in equal proportions and no other components are lost in gel filtration.

d. This product is now traded as 35% (TEA)OH solution. I would use the same volume of the 35% solution, since the synthesis is not sensitive to a variation of the template concentration of this order. It is important that the (TEA)OH source have minimum K^+ and Na^+ concentrations.

e. Addition of HCl has previously been found to allow better control of Si substitution in SAPO-34 and has been used in this case for the same purpose.

f. Standing autoclaves in a heated block on a "shaking table" rotated at about 60 rpm.

g. Filtration leads to loss of fine material or goes very slowly depending on the filter.

h. At lower water content, AFI appears as a contaminant; at higher water content, CHA appears.

i. Analysis by XRF.

j. Micropore volume 0.25 mL/g (by N_2 sorption)