VFI   VPI-5 (DPA method)   Si(95), Al(05)

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Type Material: $[Al_{19}P_{17}O_{72}]$: 0.4 DPA: 42 H_2O (DPA = di-n-propylamine)

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

Batch Composition: 1.00 Al_2O_3: 1.00 P_2O_5: 40 H_2O: 1.00 DPA[a]

Source materials
> distilled water
> pseudoboehmite (Catapal B, 68.01 wt% Al_2O_3)
> phosphoric acid (Aldrich, 88.30 wt% H_3PO_4)
> di-n-propylamine (DPA) (Aldrich, > 98% pure)

Batch Preparation (for -18 g dry product)
(1)   [64.60 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) + 10.11 g di-n-propylamine], stir for 2 hours[b]

Crystallization
> Vessel: Teflon-lined autoclave
> Time: 4 hours
> Temperature: 142°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 phase: $AlPO_4$-11[c]
> Elemental Analysis: 0.04 DPA: Al_2O_3: 0.9 P_2O_5
> Crystal Size and Habit: Crystals are spherical and aggregated, - 100 μm dia.

Reference
[1]   H. He, J. Klinowski, J. Phys. Chem. 98 (1994) 1192
Notes
a.   The amount of water quoted includes water in pseudoboehmite (100% - wt% Al_2O_3), phosphoric acid (100% - wt% P_2C_5).

b. After adding DPA, the gel is very viscous. Homogeneous stirring is therefore essential.

c. $AlPO_4$-11 is found when stirring during Batch Preparation (3) is not vigorous enough.