AEI AlPO4-18 Al(50), P(50)

Contributed by Rune Wendelbo

Verified by S. Kaliaguine, E. Dumitnu and C. Round

Type Material [$Al_{24}P_{24}O_{96}$]

Method S. T. Wilson, E. M.Flanigen [1, 2]

Batch Composition $Al_2O_3 : P_2O_5 : (TEA)_2O : 60\ H_2O : 6\ i\text{-}C_3H_7OH$
(TEA tetraethylanimonium)

Source Materials
distilled water
phosphoric acid (85%)
aluminum isopropoxide (Jansen, 98+ %)
tetraethylammonium hydroxide (Aldrich, 40% (TEAOH)[a]

Batch Preparation (for 15.8 g dry product)
(1) [36.3 g water + 15.0 g phosphoric acid + 27.2 g aluminum isopropoxide], add diluted phosphoric acid to the aluminum isopropoxide in a 250 mL polypropylene bottle and shake vigorously for one minute
(2) [(1) + 49 g (TEAOH solution], shake to produce a uniform gel. Transfer to the autoclave

Crystallization
Vessel: 200 mL Teflon-lined stainless steel autoclave (Berghof)
Incubation: 6 hours at room temperature
Time: 69 hours [b]
Temperature: 215˚C [b]
Agitation: gentle [c]

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

Product Characterization
XRD: fully crystalline AEI; competing phase: AFI [e]
Elemental Analysis: 24.26% Al, 20.59% P, 0.20% Si [f]
Crystal size and habit: square platelets 0.2-8 µm x 0.1 µm thick [g]

References
[1] S. T. Wilson, E. M. Flanigen, US Patent 4310440 (1982)
[2] R Wendelbo, D. Akporiaye, A. Andersen, I. M. Dahl, H. B. Mostad, Appl. Catal. A General 142 (1996) L197

Notes

a. This product is now traded as 35% TEAOH 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.

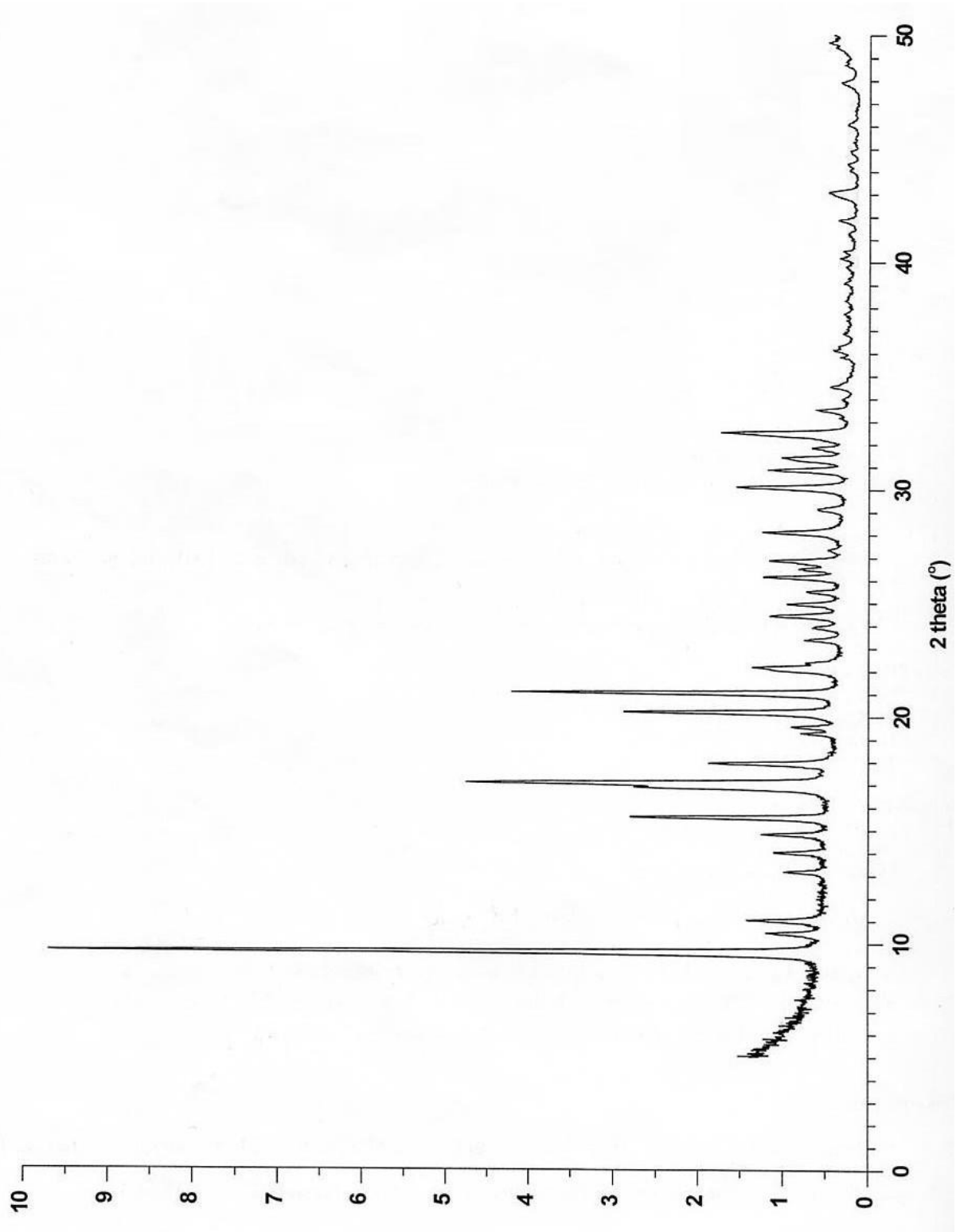
b. The synthesis temperature can probably be reduced to 200˚C and the time reduced substantially, but this has not been tested.

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

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

e. AFI appears as a contaminant.

f. A Cameca microprobe was used averaging 5 points each 50 x 50 μm. The analysis was done on "as synthesized material".

g. Micropore volume 0.28 mL/g (by N_2 adsorption).

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