PAU ECR-18 Si(76), Al(24)

Contributed by David Vaughan

Verified by P. Piccione and B. Subotic

Type Material $(Na_{82}K_{66}TEA_{16})[Al_{164}Si_{508}O_{1344}] : wH_2O$ (TEA = tetraethylammomum)

Method D. E. W. Vaughan, K. G. Strohmaier [1,2]

Batch Composition $0.4\ K_2O : 0.6\ Na_2O : 1.4\ (TEA)_2O : Al_2O_3 : 9\ SiO_2 : 0.3\ Na_2SO_4 : 140\ H_2O$

Source Materials

deionized water
potassium hydroxide (J. T. Baker, >99% KOH. $0.5\ H_2O$)
sodium hydroxide (J. T. Baker, ~99% NaOH)
alumina (Alcoa C-31, >99% $Al_2O_3 \cdot 3\ H_2O$)

silica sol (Dupont HS-40, 40% SiO_2, 0.4% Na_2O)
tetraethylammonium hydroxide (SACHEM, 35% $N(C_2H_5)_4OH$)
aluminum sulfate (reagent grade,> 99% $Al_2(SO_4)_3 . 17H_2O$)

Batch Preparation (for 20 g dry product)

(1) [8.0 g water + 1.79 g potassium hydroxide + 2.8 g sodium hydroxide + 5.1 g alumina], reflux until a clear solution is obtained; cool to room temperature. Replace water lost by evaporation
(2) [10 g aluminum sulfate + 10 g water], mix and warm if necessary to make a solution
(3) [49.7 g silica sol + 43.3 g tetraethylammonium hydroxide + (1) + 5.29 g (2) + 19 g water], add sequentially with mixing in a beaker

Crystallization

Vessel: 125 mL Teflon jar (Nalgene)
Incubation: 3 days at room temperature
Time: 12 to 16 days[a]
Temperature: 100°C
Agitation: none

Product Characterization

XRD: excellent PAU
Elemental Analysis: $0.4\ K_2O : 0.5\ Na_2O : 0.1\ (TEA)_2O : Al_2O_3 : 6.2\ SiO_2 : H_2O$
Crystal size and Habit: spherical aggregates (1 to 10 μm) of submicron crystals (<0.1 to 0.2 μm)[b]

References

[1] D. E. W. Vaughan, K. G. Strohmaier, U. S. Patent 5 013 536 (1991)
[2] D. E. W. Vaughan, K. G. Strohmaier, Micropor. Mesopor. Mater. 28 (1999) 233

Notes

a. Both 12 and 16 day samples were excellent PAU.
b. ^{13}C-NMR shows two sites; ^{29}Si-NMR shows three broad peaks.