

CON

CIT-1

Si(96), B(4)

Contributed by Mark E. Davis

Verified by S. B. Hong, E.-P. Ng, J. Shin

Type Material: [H₂][B₂Si₅₄O₁₁₂]

Method: Raul F. Lobo and Mark E. Davis [1]

Batch Composition: 50SiO₂ : B₂O₃ : 10ROH : 5NaOH : 3000H₂O

Source Materials

sodium boratedecahydrate (Na₂B₄O₇·10H₂O)

sodium hydroxide (NaOH)

template ROH (N,N,N-trimethyl(-)-cis-myrtanyl ammonium)

distilled water (DD)

fumed silica (Cab-0-Si1 grade M-5 or Aerosil 200, Degussa)

Synthesis of template (*N,N,N*-trimethyl(-)-cis-myrtanyl ammonium hydroxide)

10 g of (-)-*cis*-myrtanylamine were dissolved in 40 mL of methanol. To this solution were added 10.47 g K₂CO₃ and 21.95 g methyl iodide. The mixture was stirred for several minutes and then refluxed at 40 °C for 24 h. The reaction mixture was filtered, and the solid filtrate was washed with an additional 20 mL of methanol. The combined methanol solutions were heated at 50 °C in a rotavapor. A white solid is formed in the flask, which is then extracted with 2 portions each of 40 mL of chloroform. Recrystallization was performed in a ca. 1:10 (volume ratio) chloroform–*n*-hexane mixture.

The exchange of the iodide salt into a hydroxide was performed as follows: 4.5 g iodide salt was dissolved in 13.8 g distilled water and passed through 11.6 g of Amberlite IRN-78 OH anion exchange resin. The collected solution was concentrated in a rotavapor at 70 °C.

Batch Preparation (for 23.85 g initial gel)

- (1) [6.60 g of 13.0 wt.% ROH + 15.89 g distilled water], stir
- (2) [0.0767 g sodium borate decahydrate (Na₂B₄O₇·10.0H₂O) + 0.08 g NaOH + (1)], stir until dissolved
- (3) [1.20 g fumed silica (Aerosil 200, Degussa) + (2)], stir until a homogeneous gel is obtained

Crystallization

Vessel: pure-silica glass tubes^a or Teflon-lined 23 mL autoclave

Temperature: 150 C^b

Time: 42 days

Agitation: no

Product Recovery

- (1) Dilute reaction mixture with water

(2) Centrifuge and wash with water

Product Characterization

XRD: CON; competing phase: SSZ-33

Crystal size and habit: CIT-I crystals (typically $3\ \mu\text{m} \times 3\ \mu\text{m} \times 6\ \mu\text{m}$)

Reference

[1] Lobo, R.F. and Davis, M.E. J. Am. Chem. Soc. 117 (1995) 3766

[2] Christopher W. Jones, Son-Jong Hwang, Tatsuya Okubo and Mark E. Davis. Chem. Mat. 13 (2001), 1041

[3] Mark E. Davis and F. Lobo. U.S. Pat. 5,512,267 (1996)

Notes

- a. 25 mm i.d., 100 mm long approximately 85% filled with the gel
- b. heated statically in convection oven