

Contributed by Suk Bong Hong

Verified by J. Rimer, M. Hartmann, T. Okubo, S. H. Cha

Type Material: $\text{Na}_{4.0}(\text{SDA})_{3.3}[\text{Al}_{8.9}\text{Si}_{63.1}\text{O}_{144}] \cdot w \text{H}_2\text{O}$ ($w \sim 8$)
(SDA = 1,4-bis[*N*-methylpyrrolidinium]butane (1,4-MPB))

Method: S. B. Hong, E. G. Lear, P. A. Wright, W. Zhou, P. A. Cox, C. -H. Shin, J. -H. Park, I. -S. Nam [1]

Batch Composition: 4.5 (1,4-MPB) : 15.0 Na_2O : 1.0 Al_2O_3 : 30 SiO_2 : 1200 H_2O

Source Materials

deionized water
1,4-MPB dibromide^a
sodium hydroxide (Aldrich, 50% NaOH solution)
aluminium nitrate nonahydrate (Junsei, 98%, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$)
silicon dioxide (Degussa Aerosil 200 or Cabot Cab-O-Sil M5)

Batch Preparation (for 2 g dry product)

- (1) [66.57 g water + 1.15 g aluminium nitrate nonahydrate + 8.00 g sodium hydroxide (50% solution)], stir until dissolved
- (2) [(1) + 6.00 g silica] mix thoroughly and stir for 30 minutes
- (3) [(2) + 5.46 g 1,4-MPB dibromide], stir for 24 hours^b

Crystallization

Vessel: Teflon-lined stainless steel autoclave
Temperature: 160 °C
Time: 7 days
Agitation: 100 rpm

Product Recovery

- (1) Dilute reaction mixture with water
- (2) Filter and wash with water
- (3) Dry at ambient temperature or at 90 °C
- (4) Yield: 1.97 g

Product Characterization

XRD: STI; competing phase: ANA (when $\text{NaOH}/\text{SiO}_2 > 1.13$)
Elemental analysis: Si/Al = 6.5 ~ 7.5 [1]
Crystal size and habit: agglomerates and intergrown rectangular plates of about $0.5 \times 1.0 \times 0.1 \mu\text{m}^3$

Reference

- [1] S. B. Hong, E. G. Lear, P. A. Wright, W. Zhou, P. A. Cox, C. -H. Shin, J. -H. Park, I. -S. Nam, *J. Am. Chem. Soc.* 126 (2004) 5817

Notes

- a. The description of template preparation is given in [1]
- b. pH of the final gel is 11.8.