

Contributed by Yoshihiro Kubota

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Type Material: Si_{101.6}Al_{11.4}O₂₂₄

(SDA = TEBOP²⁺(I⁻)₂ = *N,N'*-diethylbicyclo[2.2.2]oct-7-ene-2,3:5,6-dipyrrolidine diiodide)

Method: D. L. Dorset, S. C. Weston, S. S. Dhingra [1]

Batch Composition: 1.0 SiO₂ : 0.1 Al(OH)₃^a : 0.375 KOH : 0.1 TEBOP²⁺(I⁻)₂ : 30 H₂O

Source Materials

colloidal silica (Ludox HS 40)

aluminum hydroxide (50 wt.%)

deionized water (DI)

8 mol/L potassium hydroxide aqueous solution (titrated as 5.93 mmol/g)

TEBOP₂⁺(I⁻)₂ synthesized from bicyclo[2.2.2]oct-7-ene-2,3:5,6- tetracarboxylic anhydride (Aldrich)

Batch Preparation (for ca. 6 g as-synthesized dry product)

- (1) [15.0 g Ludox HS-40 + 40.0 g DI water + 0.78 g aluminum hydroxide], stir for 10 min in a Teflon beaker
- (2) [(1) + 6.32 g potassium hydroxide aqueous solution 5.93 mmol/g]; stir for 30 min
- (3) [(2) + 5.58 g TEBOP₂⁺(I⁻)₂]; stir for 4 h at room temperature

Crystallization

Vessel: Teflon-lined stainless steel autoclave

Temperature: 160 °C

Time: 16 days

Agitation: 0 rpm

Product Recovery

- (1) Dilute reaction mixture with water
- (2) Filter and wash with water
- (3) Dry at ambient temperature or at 80 °C
- (4) Yield: 6.0 g
- (5) Calcination conditions to remove SDA: 600°C for 10 h in air

Product Characterization

XRD: MSE

Crystal size: smaller than 100 nm

Reference

- [1] D. L. Dorset, S. C. Weston, S. S. Dhingra, J. Phys. Chem. B 110 (2006) 2045
- [2] S. Inagaki, Y. Tsuboi, Y. Nishita, T. Syahylah, T. Wakihara, Y. Kubota, Chem. Eur. J. 19 (2013) 7780

Notes

- a. Al(OH)₃ source is important. The brand should be Pfalz and Bauer or Aldrich.