

RHO

High Silica Rho

Si(80), Al(20)

Contributed by Alain Matijasic and Joël Patarin

Verified by S. Schwarz, T. Cholley, G. Gbery and K. Balkus

Type Material $\text{Na}_{6.8}\text{Cs}_{3.0}[\text{Al}_{98}\text{Si}_{38.2}\text{O}_{96}] \cdot (18\text{-C-6}) \cdot w\text{H}_2\text{O}$ ($w \approx 29$)

Method T. Chatelain, J. Patarin, E. Fousson, M. Soulard, J. L Guth, P. Schulz [1]

Batch Composition $1.8\text{Na}_2\text{O} : 0.3\text{Cs}_2\text{O} : \text{Al}_2\text{O}_3 : 10\text{SiO}_2 : 0.5(18\text{-C-6}) : 100\text{H}_2\text{O}$

Source Materials

distilled water

18-C-6 (Lancaster, > 98% cycl. $(\text{C}_2\text{H}_4\text{O})_6$)

cesium hydroxide (Aldrich, 50% CsOH in water)

sodium hydroxide (SDS, > 98% NaOH)

sodium aluminate (Carlo Erba, 56% Al_2O_3 , 37% Na_2O , 7% H_2O)

silica sol (Dupont Ludox AS-40, 40% SiO_2)

Batch Preparation (for ~6 g product)^a

- (1) [7.84 g water + 1.35 g 18-C-6 + 1.80 g cesium hydroxide solution + 0.59 g sodium hydroxide], stir until dissolved ^b
- (2) [(1) + 1.82 g sodium aluminate], stir until homogenized
- (3) [(2) + 15.00 g silica sol], stir until homogenized (formation of a gel) Continue stirring with magnetic stirrer for 24 h., then transfer to a PTFE-lined stainless-steel autoclave. Gel pH = 14

Crystallization

Vessel: 120 mL PTFE-lined stainless steel autoclave

Time: 192 hours

Temperature: 110°C in a preheated oven

Agitation: none. Final pH approximately 12

Product Recovery

- (1) Dilute the reaction mixture with distilled water
- (2) Filter and wash until pH 10
- (3) Dry at 60°C overnight
- (4) Yield: 6g as-synthesized RHO-type sample (product containing about one molecule 1 8-C-6 as organic template per unit cell)^b

Product Characterization

XRD: Strong RHO pattern showing cubic symmetry. ($a_0=15.031(1)\text{\AA}$);^c no visible impurities

Elemental Analyses: Si/Al is close to 3.9 ^b

Crystal size and habit: The crystals display a sphere-like shape with an average size of 1 μm

Reference

- [1] T. Chatelain, J. Patarin, E. Fousson, M. Soulard, J. L Guth, P. Schulz, *Micropor. Mat.* 4 (1995) 231

Notes

- a. This recipe has been successfully scaled up by a factor of six.
- b. The starting mixture is prepared in a polyethylene vessel.
- c. According to Ref. [1].