

Contributed by Manuel Moliner

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Type Material: $(\text{SDA}^+)_{2.5}(\text{SiO}_2)_{20.6}(\text{GeO}_2)_{11.4}(\text{F}^-)_2 : w \text{ H}_2\text{O}$
(SDA = 1-benzyl-1,4-diazabicyclo [2.2.2]octane)^a

Method: A. Corma, M.T. Navarro, F. Rey, J. Rius, S. Valencia [1]

Batch Composition: 0.66 SiO₂ : 0.33 GeO₂ : 0.5 SDAOH : 0.5 HF : 8 H₂O

Source Materials

deionized water (DI)

1,4-diazabicyclo[2.2.2]octane (DABCO) (Aldrich, 98%)

benzyl chloride (Aldrich, 99%)

ethyl acetate (Scharlab, 98%)

amberlite[®] IRN78, hydroxide form (Supelco)

germanium oxide (Aldrich, 99.99 %)

tetraethylorthosilicate (Aldrich, 99.99%)

hydrofluoric acid (Aldrich, 48% in water)

Batch Preparation (for 2.7 g dry product)

- (1) [10.23 g benzyl chloride + 13.7 g 1,4-diazabicyclo[2.2.2]octane + 150 g ethyl acetate], stir in a flask for 24 h, at room temperature.^a
- (2) recover solid (1) by filtration and wash with 250 g diethyl ether.
- (3) dry solid (2) in vacuum at room temperature for 5 h.
- (4) [solid (3)^b + 70 g Amberlite[®] + 75 g water], stir for 24h at room temperature, remove Amberlite[®] by filtration.
- (5) [31.25 g solution (4)^c + 1.74 g germanium oxide], stir until dissolved.
- (6) [solution (5) + 6.94 g tetraethylorthosilicate], stir 10 minutes.
- (7) [solution (6) + 1 g HF], stir until 23 g are evaporated (water + ethanol) for adjusting the gel composition

Crystallization

Vessel: Teflon-lined stainless steel autoclave

Temperature: 150° C

Time: 3 days

Agitation: no

Product Recovery

- (1) Dilute reaction mixture with water
- (2) Filter and wash with water
- (3) Dry at 100°C
- (4) Yield: 2.7 g

Product Characterization

XRD: BEC;

Elemental analysis: 2 SiO₂: GeO₂^d

Crystal size and habit: rod like crystals with size below 1 μm

Reference

[1] A. Corma, M.T. Navarro, F. Rey, J. Rius, S. Valencia, *Angew. Chem. Ind. Ed.* 40 (2001) 2277

[2] G. Sastre, J.A. Vidal-Moya, T. Blasco, J. Rius, J.L. Jordá, M.T. Navarro, F. Rey, A. Corma, *Angew. Chem. Ind. Ed.* 41 (2002) 4722

Notes

- a. SDA made in house
- b. 90 % yield of SDA
- c. [0.8M] SDAOH, titrated with HCl 0.1 N
- d. as-synthesized; organic content not specified