

Contributed by Moussa Zaarour and Svetlana Mintova

Verified by G. Rioland, T.J. Daou, J. Patarin, H. Awala, M. Tsapatsis, Z. Qin

Type Material: [Si₉₆O₁₉₂]

Method: A.E. Persson, B.J. Schoeman, J. Sterte, J.-E. Otterstedt [1]
S. Mintova, N.H. Olson, J. Senker, T. Bein [2]^a

Batch Composition: 9 TPAOH : 25 SiO₂ : 480 H₂O : 100 EtOH

Source Materials

tetraethylorthosilicate TEOS (99.99 %, Aldrich)
tetrapropylammonium hydroxide (TPAOH, 1.0 M aqueous solution, Aldrich)
double distilled water

Batch Preparation

[20 g TEOS + 35 g TPAOH + 5.26 g H₂O] hydrolyze for 24 h on a gyratory shaker (180 rpm)^b

Crystallization

Vessel: polypropylene (PP) bottle
Temperature: 90 °C
Time: 30 h

Product Recovery

- (1) Centrifugation (20000 rpm, 1h) and redispersion in water till pH=8
- (2) Drying at 70 °C for 15 hours or freeze-drying
- (3) Calcination: 600 °C for 6h
- (4) Solid product: 2.9 g

Product Characterization

XRD: MFI; DLS: particle size of 80-90 nm; SEM: zeolite nanocrystals with spheroidal shape

References

- [1] A.E. Persson, B.J. Schoeman, J. Sterte and J.-E. Otterstedt, Zeolites 14 (1994) 557
- [2] S. Mintova, N.H. Olson, J. Senker, T. Bein, Angew. Chem. 41 (2002) 2558

Notes

- a. The procedure reported in [2] is slightly different from [1], but the final product is the same (nanosized TPA-silicalite-1).
- b. Clear precursor solution is obtained.