MFI Silicalite-1 Si(100)

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Type Material $Si_{96}O_{192}F_4(TPA)_4$ (TPA = tetra-n-propylammonium)

Method J.-L Guth, H. Kessler, R. Wey [1]

Batch Composition 1 SiO_2 : 0.08 (TPA)Br: 0.04 NH_4F: 20 H_2O

Source Materials

distilled water
tetrapropylammonium bromide (Fluka, 98%)
ammonium fluoride (Fluka, 98%)
silica (Degussa Aerosil 130, 99+%)

Batch Preparation (for 12 g product)

(1) [72 g water + 4.26 g tetrapropylammonium bromide + 0.296 g ammonium fluoride], stir until dissolved
(2) [(1) + 12 g silica], mix with a spatula, and then stir until homogenized. Initial pH = 6

Crystallization

Vessel: PTFE-lined autoclave
Time: 15 days [a]
Temperature: 200°C
Agitation: none

Product Recovery

(1) Filter, wash with distilled water
(2) Dry at 80°C
(3) Yield: 12.7 g; near 100% based on silica

Product Characterization

XRD: characteristic strong reflections at d = 11.01, 3.829, 3.806 and 3.698 Å
Elemental Analyses: $Si_{96}O_{192}F_4(TPA)_4$
Crystal Size and Habit: prisms 95 x 80 μm

Reference

[1] J.-L Guth, H. Kessler, R Wey, in Stud. Surf. Sci. Catal., Vol. 28, Y. Murakami, A. Iijima, J. W. Ward (eds.), Kodansha-Elsevier, Tokyo, 1986, p. 121

Note

a. Increasing NH_4F leads to a decrease in crystallization time (2 days for NH_4F/SiO_2 = 1).