MFI [B] ZSM-5 Si(98), B(2)

Contributed by Rob de Ruiter

Verified by Z. Gabelica, U. Deforth and A. Cichocki

Type Material $Na_{0.4}(TPA)_{0.4}[B_2Si_{94}O_{192}] : wH_2O$ (TPA = tetra-n-propylammonium)

Method R de Ruiter, J. C. Jansen, H. van Bekkum [1]

Batch Composition 2.1 Na_2O : B_2O_3 : 2.4 SiO_2 : 4 TPABr: 1050 H_2O

Source Materials

distilled water
silica (Aerosil 200-Degussa)
sodium hydroxide (J. T. Baker, reagent grade)
tetrapropylammonium bromide (TPABr)(CFZ Zaltbommel)
boric acid (Merck p.a.)

Batch Preparation (0.12 to 0.14 g product/35 mL autoclave)

(1) [280 g water + 2.66 g silica + 3.1 g sodium hydroxide], shake overnight at room temperature
(2) [(1) + 19.7 g tetrapropylammonium bromide], stir until dissolved
(3) [90 g water + 2.92 g boric acid], stir until dissolved
(4) [20 mL of (2) + 5 ml. of (3)], mix thoroughly; initial pH ~11

Crystallization

Vessel: Teflon-lined stainless steel autoclave, 35 mL
Time: 5 days
Temperature: 180°C
Agitation: none

Product Recovery

(1) Filter and wash
(2) Yield: 60-70% on SiO_2

Product Characterization

XRD: Pure MFI, no extraneous phases
Elemental Analysis: 93.5% SiO_2, 1.25% B_20_3, 2% TPABr, 0.25% Na_2O
Crystal Size and Habit: prismatic to lath morphology depending on boron content, the crystal thickness (in b-direction) decreases with boron content of framework

Reference

[1] R. de Ruiter, J. C. Jansen, H. van Bekkum, in Synthesis of Microporous Materials, Vol. 1, M. L Occelli, H. F, Robson (eds.), Van. Nostrand Reinhold, New York, 1992, p 167