MTT ZSM-23 Si(97), Al(3)

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Type Material $Na[AlSi_{23}O_{48}]$ wH_2O (w » 4)

Method S. Ernst, R Kumar, J. Weitkamp [1]

Batch Composition $20.6Na_2O : Al_2O_3 : 100SiO_2 : 4610H_2O : 46.2Pyrr : 18.8\ H_2SO_4$

Source Materials
demineralized water
sodium hydroxide, reagent grade (Riedel-de Haen)
pyrogenic silica (Cab-o-Sil M5) [a]
pyrrolidine (Pyrr), (Fluka)
aluminum sulfate ($Al_2(SO_4)_3$. $18H_2O$, Fluka)
sulfuric acid (96 wt% H_2SO_4, Fluka)

Batch Preparation (for 14 g product) [b]
(1) [195.8 g water + 4.40 g sodium hydroxide], mix until dissolved
(2) [(1) + 16.5 g silica], add silica to solution (1) over the course of ~5 minutes under continuous stirring
(3) [24.4 g water + 8.81 g pyrrolidine + 1.79 g aluminum sulfate], mix until dissolved
(4) [(2) + (3)], add solution (3) to solution (2) with good mixing
(5) [(4) + 4.33 g sulfuric acid], add acid dropwise with stirring; final gel pH = 12.6

Crystallization
Vessel: stainless steel autoclave (300 mL) [c] Temperature: 180¡C Time: 50 hours Agitation: autoclaves are rotated ~30/minutes

Product Recovery
(1) Cool and filter
(2) Wash extensively with demineralized water
(3) Dry at 100¼C for 16 hours
(4) Yield: approx. 14 g

Product Characterization
XRD: MTT (only crystalline phase), competing phases: MFI (contaminated autoclave) cristobalite (occasional overheating, or pH too high)
Elemental Analysis: SiO_2/Al_2O_3 = 78 (AFS/ICP) [d]
Crystal Size and Habit: bundles of needles [e]

Reference
[1] S. Ernst, R Kumar, J. Weitkamp, in Zeolite Synthesis, Am. Chem. Soc. Symposium Series 398, M. Occelli, H. Robson (eds.), 1989, pp 560-573
Notes

a. Cab-O-Sil M5 must be used; other silica sources, such as silica sol or sodium silicate, lead to different products.
b. The synthesis has been successfully scaled up to a 5 liter autoclave (stirring rate: 120/minutes, yield: 250 g).
c. The crystallization vessels have to be cleaned very thoroughly in order to avoid seeding effects from residual crystallites of ZSM-5, for example.
d. With pyrrolidine as template, the molar ratio can be varied between 70 and 150.
e. Platelet-like ~1 um can be synthesized using C_7diquat $[(CH_3)_3N\text{-}(CH_2)_7\text{-}N(CH_3)_3]Br_2$ as template. [1]