MFI [Fe]-ZSM-5 Si(96), Fe(4)

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Verified by Y. Kiyozumi and P. Fejes

Type Material $Na_{0.3}(TPA)_{3.7}[Fe_4Si_{92}O_{192}]$ wH_2O (TPA = tetra-n-propylammonium)

Method A. Brückner, R. Lück, W. Wieker, B. Fahlke [1]

Batch Composition 30 Na_2O : Fe_2O_3 : 30 SiO_2 : 1040 H_2O : 5 (TPA)Br : 25 H_2SO_4

Source Materials

distilled water
sulfuric acid (reagent grade, 98% H_2SO_4)
iron(III) sulfate (reagent grade, $Fe_2(S0_4)_3$. 9 H_2O) sodium
metasiicate (reagent grade, Na_2SiO_3. 9 H_2O) [a]
tetrapropylammonium bromide (TPABr) (Fluka, CH-9470 Buchs)

Batch Preparation (for ~20 g volatile-free product)

(1) [100 g water + 22 g sulfuric acid + 5.62 g iron(III) sulfate], stir until dissolved
(2) [163.4 g water + 85.26 g sodium metasiicate], stir until dissolved
(3) [(1) + (2)], add silicate slowly to iron(III) sulfate solution with good mixing
(4) [(3) + 13.31 g TPABr], mix vigorously until uniform (~ 400 rpm)

Crystallization

Vessel: stainless steel autoclaves
Temperature: 170°C
Time: 72 hours
Agitation: autoclaves are rotated axially

Product Recovery

(1) After cooling, filter and wash with water several times
(2) Dry over P_40_{10}
(3) Calcine at 550°C for 4 hours to remove template

Product Characterization

XRD: ZSM-5 (only phase)
Elemental Analysis: 0.07 Na_2O : Fe_2O_3 : 48 SiO_2 : wH_2O
Crystal Size and Habit: 1-6 μm, snowball-like

Reference

[1] A. Brückner, R. Lück, W. Wieker, B. Fahlke, Zeolites 12 (1992) 380

Note

a. Al(III), a common impurity in silica sources, displaces Fe(III) from T-atom positions in MFI. [1]