TON ZSM-22 Si(95), Al(05)

Contributed by Gunter Kühl

Verified by M. Derewinski and Y. Oumi
Type Material $K_{0.1}Al_{0.6}Si_{23.4}O_{48}(DAO)_{0.86}$[a] (DAO = 1 ,8-diamino-octane)

Method E. W. Valyocsik [1]

Batch Composition 8.9 K_2O : Al_2O_3 : 90 SiO_2 : 3 K_2SO_4: 27.3 DAO : 3588 H_2O

Source Materials

deionized water
aluminum sulfate (99+% $Al_2(SO_4)_3$ á18 H2O)
potassium hydroxide (87.9% KOH)
1, 8-diamino-octane (99+%)
silica sol (Dupont AS-30, ammonia stabilized, 30% SiO_2)

Batch Preparation (for 7.5 g dry product)

(1) [18.2 g water + 1.76 g aluminum sulfate], stir until dissolved
(2) [18.2 g water + 4.0 g potassium hydroxide], stir until dissolved
(3) [72.8 g water + 10.4 g 1,8-diamino-octane], stir until dissolved
(4) [26.95 g water + 47.65 g silica sol], mix well
(5) [(1) + (2)], mix well
(6) [(3) + (5)], add (3) to (5); blend
(7) [(4) + (6)], add (4) to (6), stir for 30 minutes[b]

Crystallization

Vessel: stirred autoclave with stainless steel liner
Incubation: 24 hours at room temperature[c]
Temperature: 160°C
Time: 2-3 days
Agitation: vigorous stirring[d]

Product Recovery

(1) Dilute reaction mixture with water
(2) Filter and wash with water
(3) Dry at ambient temperature or at 110¡C
(4) Yield: 7.5 g (near 100% on Al_2O_3)

Product Characterization

XRD: TON; competing phase: MEL (trace)[d]
Elemental Analysis: 0.2 K_2O : Al_2O_3 : 39 SiO_2 : 1.44 DAO
Crystal size and habit: needles [e]

Reference

[1] E. W. Valyocsik, US Patent 4 902 406

Notes

a. Missing cations assumed to be protonated DAO.
b. Reaction mixture becomes cloudy but does not gel.
c. It is not certain that aging (or seeding) is beneficial.
d. Ref. [1] recommends a stirring rate of 400 rpm; static preparations or slow stirring produce MEL or mixtures of MEL + TON.
e. The size of the crystallites decreases with increasing stirring rate.