CHA SSZ-13 Si(93), Al(7)

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Type Material (aRN, bNa) [$Al_{2.4}Si_{33.6}O_{72}$] : wH_2O[a] (w = 1 to 7)
(RN = N,N,N, trimethyl-1-adamantammonium)

Method S. I. Zones, R. A. Van Nordstrand [1]

Batch Composition 10 Na_2O : 2.5 $A1_2O_3$: 100 SiO_2 : 4400 H_2O : 20 RN-OH

Source Materials
sodium hydroxide (1 N), (Baker, reagent grade)
N,N,N, trimethyl-1-adamantanimonium hydroxide (RN-OH)(O.72M)[b]
deionized water
aluminum hydroxide (Reheis F-2000 dried gel, 50% Al_2O_3)
fumed silica (Cab-Q-Sil, M5 grade, 97% SiO_2)

Batch Preparation (for 0.6 g product)
(1) [2.00 g 1N NaOH + 2.78 g 0.72 M RN·OH + 3.22 water], add sequentially to the Teflon cup of a Parr 23 mL autoclave [c]
(2) [(1) + 0.05 g aluminum hydroxide], mix until solution clears
(3) [(2) + 0.60 g fumed silica], mix until uniform

Crystallization
Vessel: Teflon-lined 23 mL autoclave (Parr model 4745)
Temperature: 160˚C [d]
Time: 4 days
Agitation: none

Product Recovery
(1) Cool to room temperature [e]
(2) Filter in a medium frit glass funnel
(3) Wash with about one liter of water
(4) Air dry at room temperature with vacuum pulling through frit
(5) Yield: 0.57 g, 74% based on TO_2

Product Characterization
XRD: CHA (only crystalline phase); competing phases when observed: analcime or quartz; impurities can occur at this temperature or higher.
Elemental Analyses: RN is approximately 15 wt% and SiO_2/Al_2O_3 = 28 by ICP
Crystal Size and Habit: cubes of 2-5 μm with some occasional intergrowth.

References

[1] S. I. Zones, R A. Van Nordstrand, Zeolites 8 (1988) 166
[2] S. I. Zones, US Patent 4 544 538 (1985)
[3] S. I. Zones, Trans. Faraday Soc. 87 (1991) 3709

Notes

a. Typical values: a 1.4 to 2.9, b = 0.7 to 4.3
b. The description of template preparation is given in [2]. The fastest synthesis of this product is from FAU sources [3].
c. The Teflon cup is washed between runs with HF (48%), water, KOH solution, and water again.
d. The reactor is placed into a Blue M convection-heating oven preset at 160°C.
e. PH of the treated batch should measure in the range of 12.20 to 12.64 using a calibrated probe.