

## AFI

## SSZ-24

## Si(100)

**Contributed by** S. I. Zones and L T. Yuen

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**Type Material** (SiO<sub>2</sub>)<sub>24</sub>. aRN<sup>+</sup> a = 0.96 ± 0.24 (RN<sup>+</sup> = trimethyl-1-adamantammonium)

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

**Batch Composition** 5 K<sub>2</sub>O : 15 RN<sup>+</sup>:100 SiO<sub>2</sub> : 4400 H<sub>2</sub>O

### Source Materials

deionized water

RN<sup>+</sup> (see above) 0.72 Molar [2]

potassium hydroxide (Baker reagent, 87.8% KOH)

fumed silica (Cab-O-Sil M5, 97% SiO<sub>2</sub>, 3% H<sub>2</sub>O)

### Batch Preparation (for 3.5 g product)

- (1) [38.32 g water + 13.90 g (0.72M RN<sup>+</sup>) + 0.44 g potassium hydroxide], mix until dissolved
- (2) [(1) + 4.00 g fumed silica], mix in the Teflon liner of a 125 mL Parr stainless steel reactor <sup>a</sup>

### Crystallization

Vessel: Parr 125 mL reactor (Teflon-lined) heated in a Blue M oven

Temperature: 150 °C

Time: 7 days <sup>b,c</sup>

Agitation: none

### Product Recovery

- (1) Upon cooling to room temperature, the product should be settled to the bottom of the reactor liner; pH with a calibrated probe should be 11.50-11.70
- (2) Filter to recover solids (medium grade glass-frit funnel)
- (3) Wash product with approximately 100 mL 0.01N KOH solution <sup>d</sup>
- (4) Wash with approximately 1 liter water
- (5) Air dry overnight while pulling a vacuum through the frit
- (6) Yield: 3.52 g; 79% yield based on SiO<sub>2</sub>

### Product Characterization

XRD: AFI only crystalline phase

Elemental Analysis: RN<sup>+</sup> is approximately 10 wt% and alkali cation is usually less than 0.5 wt%. The remaining material is SiO<sub>2</sub> [3]

Crystal Size and Habit: Typically, hexagonal rods composed of identical hexagonal plates on top of each other. The rods are usually about 10 μm long

### References

- [1] R. A. Van Nordstrand, D. S. Santilli, S. I. Zones in Molecular Sieve Science, ACS Symp. Ser. 368. W. H. Flank, T. E Whyte, (eds.), Am. Chem. Soc., Washington, D. C., 1988, pp. 236-245
- [2] S. I. Zones, US Patent 4665 110 (1987)
- [3] I. Petrovic, A. Navrotsky, M. E.. Davis, S. I. Zones, Chem. Mater. 5 (1993) 1805
- [4] R. A. Van Nordstrand, D. S. Santilli, S. I. Zones, in Synthesis of Microporous Materials, Vol. 1, M. L Occelli, H. E. Robson (eds.), Van Nostrand Reinhold, New York, 1992. pp. 373-383

**Notes**

- a. High speed stirring of this preparation leads to SSZ-23 formation [1].
- b. The reaction can be accelerated by seeding after some initial material has been made.
- c. The synthesis of the borosilicate [4] requires only one day; the crystals are smaller.
- d. The alkaline wash helps to prevent unreacted silica from coming out of solution during washing and causing pore.