

Contributed by Valentin Valtchev

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Type Material (SiO₂)₁₃₆ q R. q » 10, (R = TMA⁺ and TrMA) ^a

Method I. Vergilov, V. Valtchev [1]

Batch Composition 15 Na₂O:16 TrMA:16 (TMA)Cl : 55 SiO₂ : 3387 H₂O: 10 H₂SO₄ ^{b,c}

Source Materials

bidistilled water

sodium silicate (Riedel de Haen, 63% SiO₂, 18% Na₂O, 18% H₂O)

sulfuric acid (98% H₂SO₄)

trimethylammonium (TrMA) chloride (Merck, 98% TrMA·HCl)

tetramethylammonium (TMA) chloride (Merck, > 98% (TMA)Cl)

Batch Preparation (for 30 g dry product)

- (1) [300 g water + 52.5 g sodium silicate], heat and stir until dissolved ^d
- (2) [150 g water + 10.7 g sulfuric acid]
- (3) [(1) + (2)], mix thoroughly
- (4) [15.29 g trimethylammonium chloride + 17.54 g tetramethylammonium chloride + 150 g water], mix thoroughly
- (5) [(3) + (4)], mix thoroughly; pH 9.2 to 9.5

Crystallization

Vessel: stainless steel or Teflon jar

Time: 12 to 14 days

Temperature: 200°C

Agitation: none

Product Recovery

- (1) After crystallization, pH 10.5
- (2) Filter to recover solids, and wash with hot water
- (3) Dry at ambient temperature (drying at 90-110°C acceptable)
- (4) Yield: 88% based on silica

Characterization

XRD: MTN only crystalline phase, ^e a₀ = 19.39 Å, single crystal structure refinement [2];

competing phases: NU-1 (when gel contains aluminum), quartz (when pH of starting gel >11.5)

Elemental Analysis: 0.0018 Na₂O. SiO₂ ^f

Crystal Size and Habit octahedral morphology, 100 to 200 nm dia. [1]

References

- [1] I. Vergiov, V. Valtchev, *Zeolites* 11 (1991) 387
- [2] J. Macicek, V. Valtchev, G. Kirov, in *Collected Abstracts, 14th Congress and General Assembly Int. Union of Crystallography*, H. C. Freeman (ed.), Lamb Printers, Perth, Australia, 1987, C-134

Notes

- a. Crystalline product is 7.4 wt% TMA⁺ and TrMA by thermal analysis.
- b. With TMA⁺ only, complex crystal twins are formed.
- c. H₂O includes water from sodium silicate, sulfuric acid and added water.
- d. Sodium silicate solution must be heated and stirred until converted to a clear solution.
- e. XRD patterns of as-synthesized product and product after 600°C calcination do not differ significantly.
- f. Analysis of ZSM-39 calcined for 1 hour at 950°C in air gave 94.7% SiO₂, 0.17% Na₂O, and a remainder, probably consisting of carbonaceous material and water.