

**BEA**

**[Ti,Al] Beta**

**Si(95), Ti(3), Al(2)**

**Contributed by** Dilson Cardoso

**Verified by** M. Cambor and W. S. Ahn

**Type Material** (TEA)  $1.5[\text{Ti}_{2.0}\text{Al}_{1.5}\text{Si}_{60.5}\text{O}_{128}]$  (TEA = tetraethylammonium)

**Method** S. Jahn, D. Cardoso [1,3], M. A. Cambor, A. Corma, J. Pérez-Pariente [2]

**Batch Composition** 0.033 TiO<sub>2</sub> : SiO<sub>2</sub> : 0.0026 Al<sub>2</sub>O<sub>3</sub> : 0.269 (TEA)<sub>2</sub>O : 15.5 H<sub>2</sub>O

**Source Materials**

tetraethylammonium hydroxide (Aldrich, 35% TEA-OH aqueous solution)  
tetraethylorthotitanate (Aldrich, 99%)  
silica (Degussa Aerosil 380, 99+%)  
aluminum nitrate (Al(NO<sub>3</sub>)<sub>3</sub> 9 H<sub>2</sub>O) deionized water

**Batch Preparation** (for ~1 g product)

- (1) [13.24 g tetraethylammonium hydroxide solution + 0.44 g tetraethylortho-titanate], mix in a glove box; stir for 10 minutes <sup>a</sup>
- (2) [(1) + 3.52 g silica], stir for 15 minutes
- (3) [0.11 g aluminum nitrate + 7.69 g water], stir until dissolved
- (4) [(3) + (2)], stir until homogenized (10 minutes minimum) <sup>b</sup>

**Crystallization**

Vessel: 50 mL stainless steel autoclave with Teflon liner  
Time: 60-96 hours  
Temperature: 140°C  
Agitation: optional  
Final pH: ~12.5

**Product Recovery**

- (1) Quench autoclave in cold water
- (2) Centrifuge (7000 rpm) and wash until pH is about 9
- (3) Dry overnight at 80°C
- (4) Yield: 3-4 g/100 g batch (about 90% based on Al, 40% on Ti and 25% on Si)

**Product Characterization**

XRD: zeolite beta (no other phases)  
Elemental analyses:  $\text{Ti}_{2.0}\text{Al}_{1.5}\text{Si}_{60.5}\text{O}_{128}$  (for material with 96 hours crystallization. As synthesized material contains TEA<sup>+</sup> cations)  
Crystal size and habit: round-shaped particles with average size 0.28 μm (for 96 hours crystallization) and a narrow size distribution

**References**

- [1] S. L. Jahn, P. A. P. Nascente, D. Cardoso, Zeolites 19 (1997) 416
- [2] M. A. Cambor, A. Corma, J. Pérez-Pariente, Zeolites 13 (1993) 82

- [3] S. L. Jahn, D. Cardoso, in Proceedings of 12th International Zeolite Conference, Vol. III, M. M. J. Treacy, B. K. Marcus, M. F. Bisher, J. B. Higgins (eds.), Materials Research Soc., Warrendale, PA, USA (1998), pp 1885-1892

**Notes**

- a. Tetraethylorthotitanate hydrolyses very rapidly under normal atmospheric conditions forming extra framework  $\text{TiO}_2$ . Using this Ti-source, the gel must be prepared in a glove box.
- b. If this equipment is not available, the gel can be prepared under normal atmosphere using a solution of tetraethylortho-titanate in isopropyl alcohol (TEOTi/iPrOH molar ratio of 1/10). This results in a small loss in solid yield.
- c. It is difficult to synthesize the beta structure in total absence of aluminum. This recipe employs the minimum content of this element (Si/Al ~400).
- d. The solid particles formed during synthesis are very small, and it is very difficult to filter them. Unfortunately, the appropriate method for purification is by centrifugation.