Contributed by Poul Norby

Verified by J. MacDougall and D. Vaughan

Type Material Li4[Al4Si4O16]: 4 H2Oa

Method P. Norby, A. Norlund Christensen, I. G. Krogh Andersen [1]

Batch Composition NaAlSiO₄: 4.3 LiCl: 154 H₂O^b

Source Materials

distilled water lithium chloride (LiCl) (Merck, analytical grade) sodium Zeolite A (Na-LTA) (Union Carbide, NaAlSiO4.25H₂O)

Batch Preparation (for 6.9 g product)

- (1) [150 mLwater + l0g LiCl], stir until dissolved
- (2) [(1) + 10 g Na-LTA], mix until uniform slurry

Crystallization

Vessel: Teflon-lined autoclave^c Time: 72 hours Temperature: 250°C^d Agitation: none

Product Recovery

- (1) Cool to ambient temperature
- (2) Filter
- (3) Wash until chloride-free
- (4) Dry at 110°C
- (5) Yield: close to 100%

Product Characterization

XRD: ABW, no other crystalline phases or amorphous material detected ^{e,f} Elemental Analysis: Li_{1.02}Na_{0.004} AlSiO₄:1.1 H₂O ^{g,h} Crystal Size and Habit: small elongated flat needles

References

- [1] P. Norby, A. Nørlund Christensen, I. G. Krogh Andersen, Acta Chem. Scand. A40 (1986) 500
- [2] P. Norby, H. Fjellvåg, Zeolites 12 (1992) 898
- [3] R M. Barrer, E. A. D. White, J. Chem. Soc., London (1951) 1267
- [4] P. Norby, Zeolites 10 (1990) 193
- [5] E. Krogh Andersen, G. Plough Sørensen, Z. Kristallogr. 176 (1986) 67

Notes

- a. Of the aluminosilicate ABW type analogues, only the hydrated Li-form and the anhydrous Rb-, Cs-, and TI-forms may be directly synthesized. [2]
- b. Addition of small amounts of potassium fluoride are reported to have a marked mineralizing effect. [3]
- c. In a gold-lined steel autoclave, the reaction was performed at 285°C in 72 hours.
- d. Good results have been obtained by crystallization at 200°C for 93 hours.
- e. At temperatures above 350°C a -eucriptite is formed. Li₂SiO₃ may occur. [1]
- f. Upon dehydration of zeolite Li-A(BW) the framework topology is retained, but the 8-ring channel is considerably narrowed. The lithium cation coordinates across the channel, preventing rehydration of the material even when submerged in water. However, by hydrothermal treatment at temperatures above 110°C, rehydration to the original zeolite is possible.
- g. The Si/Al ratios of ABW type are close to 1. Though Li-A(BW) may be synthesized from oxide compositions with Si/Al ratios from 0.5 to 7, there is no evidence of variation in the Si/Al ratio of the product. [3]
- h. Reversible dehydration of Li-A(BW) is possible to a limited extent. [4] The anhydrous ABW-types do not show any sorption of water at ambient conditions.
- i. In one experiment (sodium bromosodalite and LiBr, hydrothermal 260°C, 72 h), single crystals large enough for single crystal diffraction were obtained (0.1 x 0.05 x 0.01 mm) [1,5]