LEV [B]-Levyne Si(89), B(11)

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Type Material: $(Na,H)_5[B_5Si_{49}O_{108}]$: 8 Q : 5 H_2O[a] (Q = Quinuclidine)

Method: R. Millini, A. Carati, G. Bellussi [1]

Batch Composition: 3.0 Q : B_2O_3 :3.0 SiO_2 60 H_2O

Source Materials:

distilled water
quinuclidine (1-azabicyclo[2.2.2.] octane) (Aldrich, 97%)
silica sol (Dupont HS-40, 40% SiO_2, 0.3% Na_2O)

Batch Preparation

(1) [10 g water + 14.7 g quinuclidine], mix until dissolved
(2) [61 g water + 10.8 g boric acid + 14.7 g quinuclidine], mix until dissolved
(3) [39.5 g silica sol + (1)], mix until a uniform gel is obtained
(4) [(3) + (2)], add solution (2) to gel slowly with vigorous mixing. Final slurry pH – 11

Crystallization

Vessel: Filter to recover solids
Time: 5 days
Temperature: 170˚C
Agitation: autoclave is rotated 20 rpm

Product Recovery

(1) Filter to recover solids
(2) Wash with distilled water
(3) Dry at 120˚C
(4) Yield: 90% on SiO_2

Product Characterization

XRD: LEV (only crystalline phase), a = 12.944 Å, c = 21.914 Å
Elemental Analysis: 17 SiO_2 : B_2O_3 : 2.7 Q (dry basis)[b]
Crystal Size and Habib: Compact spherical aggregates (4 to 8 μm) of small crystals

Reference

[1] R. Millini, A. Carati, G. Bellussi, Zeolites 12 (1992) 265

Notes

a. Protonated quinuclidine (HQ^+) or Na^+ (from silica sol) act as counterions for structural boron.
b. By thermal analysis, organic material adsorbed in extra framework pores is eliminated at 268˚C. After treatment at 550˚C for a few hours, a limited structure collapse is observed with formation of amorphous material and trigonal BO_3 units. [1]