SOD NaBr-Sodalite Si(50), Al(50)

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Verified by B. Schoeman and S. Kowalak

Type Material $Na_6Al_6Si_6O_{24}$: 2 NaBr [a,b]

Method A. Stem, G. Ozin, G. Stucky [1, 2][c]

Batch Composition $Al(OH)_3$ SiO_2: 12.5 NaOH : 7.5 NaBr: 144 H_2O

Source Materials

deionized water
sodium hydroxide (Mallinckrodt, 9 8.7%)
sodium bromide (Mallinckroft, 99.0%)
silica sol (DuPont Ludox HS-40, 40% SiO_2) aluminum hydroxide (Fisher, 99.8%)

Batch Preparation (for 34 g dry product)

(1) [300 mL water + 60.0 g sodium hydroxide + 154.3 g sodium bromide], stir until dissolved
(2) [(1) + 30.0 g silica soil, stir rapidly, heat to 95°C
(3) [200 mL water + 40.0 g sodium hydroxide + 15.6 g aluminum hydroxide], stir, heat at 95°C until dissolved
(4) [Add hot (3) with hot (2)], shake gel vigorously for 5 minutes

Crystallization

Vessel: 1000 mL capped Teflon bottle
Time: 24 hours
Temperature: 95°C
Agitation: none

Product Recovery

(1) Cool to ambient temperature
(2) Filterd
(3) Wash with deionized water until filtrate is bromide-free and pH ~7
(4) Dry at 110°C
(5) Yield; close to 100% on silica and alumina

Product Characterization

XRD: SOD, no other crystalline or amorphous material detected
Elemental Analysis: $Na_7.sBr_{18}(AlSiO4)_6$ [e]
Crystal Size and Habit: 50-500 nm[f] dodecahedra, some malformed dodecahedra and penetration twins

References

[1] A. Stem, G. A. Ozin, G. Stucky, J. Am. Chem Soc. 114 (1992) 5171

[2] A. Stem, G. Ozin, in Advances in the Synthesis and Reactivity of Solids, Vol. 2, JAI Press, Greenwich, CT, 1994, p. 93
[3] D. J. Schipper, C. Z. van Doom, P. T. Bolwijn, J. Am. Ceram. Soc. 55 (1972) 256
[4] R. R. Neurgaonkar, F. A. Hummel, Mater. Res. Bull. 11(1976) 61
[5] I. F. Chang, J. Electrochem. Soc. 121 (1974) 815
[6] S. C. Zilio, V. S. Bagnato, J. Phys. Chem. 88 (1984) 1373

Notes

a. Other anions that can be introduced by various methods include OH^-, Cl^-, Br^-, I^-, CN, SCN, ClO_3^-, $C1O_4$, BrO_3, NO_2^-, N_3^-, $B(OH)_4$, $Al(OH)_4^-$, HCO_2, $CH_3CO_2^-$, $C_2O_4^{2-}$, CO_3^{2-}, S_2^-, SO_3^{2-}, SO_4^{2-}, MnO_4^-, SeO_4^{2-}, MoO_4^2, TeO_4^{2-}, WO_4^2-, PO_4^{3-}, [e].

b. Synthesis of NaOH-SOD: $Na_6Al_6Si_6O_{24}$. 2 NaOH. 8 H_2O: batch $Al(OH)_3$ SiO_2: 5 NaOH : 41 H_2O. [1] Extensive washing, especially with hot water, results in extraction of hydroxide and sodium ions.

c. Other methods for sodalite synthesis include sintering [3, 4] and structure conversion. [5]

d. Alternately: centrifuge for 20 minutes, decant mother liquor, add fresh water and disperse the solid phase by shaking, followed by centrifuging. Repeat six times.

e. Low cation and anion content is due to formation of some OH^- containing cages (or anion-free cages in washing). These can be removed by heating the sodalite with NaBr. [6]

f. The larger crystallites are obtained in more dilute solutions.