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Characterization of synthetic Cs-Li cancrinite grown in a butanediol-water system: An NMR spectroscopic and Rietveld refinement study

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ABSTRACT

A Cs-Li-analogue of cancrinite has been synthesized from tetraethoxysilane and Al sec-butoxide in a mixture of 1,3-butanediol and water. Cs and Li were introduced as hydroxides. Synthesis was done at autogeneous pressure and T = 473 K for 100 h. The synthesis product was characterized by powder X-ray diffraction, IR-spectroscopy, thermogravimetry, ^{1}H, ^{6}Li, ^{7}Li, ^{133}Cs, ^{27}Al, ^{29}Si MAS, and {^{1}H} ^{13}C CPMAS NMR and Rietveld structure refinement. ^{29}Si MAS NMR and ^{27}Al MAS NMR spectroscopy show a framework with strong alternating order of the SiO_4 and AlO_4 tetrahedra and an Si/Al ratio of one. ^{6}Li MAS NMR suggests the existence of two different signals that cannot be resolved in the ^{7}Li MAS NMR spectrum. ^{133}Cs MAS NMR shows only a single resonance. The thermogravimetric analysis reveals a continuous weight loss in several steps, indicating the evaporation of water of hydration. IR spectroscopy shows evidence for hydroxyl anions and water included in the cancrinite framework. The Rietveld structure refinement is consistent with a completely ordered alumosilicate framework with an Si/Al ratio of one and the presence of both molecular water and hydroxyl groups in the hexagonal channel, a result different from the previously published structure. Cs is exclusively located in the ε-cages; Li ions are located in the channels and are coordinated by water molecules.