

Sodium cation dynamics in nitrate cancrinite: A low and high temperature ^{23}Na and ^1H MAS NMR study and high temperature Rietveld structure refinement

MICHAEL FECHTELKORD,* FRANK STIEF, AND JOSEF-CHRISTIAN BUHL

Institut für Mineralogie, Universität Hannover, Welfengarten 1, 30167 Hannover, Germany

ABSTRACT

Static and ^{23}Na MAS and ^1H MAS NMR experiments at temperatures down to 100 K as well as high temperature static and ^{23}Na MAS NMR experiments up to 873 K were carried out to study the dynamics of the sodium cation in nitrate cancrinite. In addition, X-ray powder data were recorded at 296 K and 673 K to obtain information about long-range order changes in the structure. Low temperature ^{23}Na NMR experiments show almost no change in signal, whereas the ^1H MAS NMR spectra show the freezing of cage water motion. The two different crystallographic sodium sites cannot be distinguished in the ^{23}Na MAS NMR spectra at low and room temperature. In contrast, two ^{23}Na NMR signals exist at high temperatures above 673 K, a quadrupolar pattern and a Lorentzian signal. Rietveld structure refinements of powders show no change in the local environment of sodium. The strong quadrupolar coupling results from the loss of cage water and the near planar O atom coordination of Na in the ϵ -cage, while the interaction of Na^+ in the channel remains similar. Thus the change in quadrupolar interaction is mainly caused by the absence of electrostatic shielding of cage water after dehydration.