2D-^{23}Na NUTATION NMR ON SODALITES

E. de Boer and R. Janssen

Department of Molecular Spectroscopy
Faculty of Science
University of Nijmegen
Toernooiveld, 6525 ED NIJMEGEN
The Netherlands, tel.: 080-613429

1. Introduction

Two dimensional nutation NMR is an efficient technique for obtaining detailed information on quadrupolar interactions in solid materials [1]. In such an experiment the system is irradiated during the evolution period (t_1) with a near-resonant rf magnetic field, after which, during the detection period (t_2) the free-induction decay is measured. During the evolution period, the spins precess around the rf field with nutation frequencies dependent on the quadrupole interaction. When the quadrupole interaction is small compared to the Zeeman interaction with the rf field the nutation frequency is equal to ω_{rf}. When the quadrupole interaction is large compared to the interaction with rf field, the nutation frequency amounts to $(I+1/2)\omega_{rf}$. Thus spectra along the F_1-direction (t_1 period) furnish information about the quadrupole interaction.

2. Results and discussion

We have carried out 2D-^{23}Na nutation NMR on dehydrated and hydrated Cl-, Br- and I-sodalite [2]. The latter have compositions $Na_8[AlO_2)_6(SiO_2)_6]Cl_2 2.1H_2O$, $Na_8[AlO_2)_6(SiO_2)_6]Br_2 1.9H_2O$ and $Na_8[AlO_2)_6(SiO_2)_6]I_2 2.0H_2O$.

The 2D-^{23}Na nutation spectra of the dehydrated samples are characterized by a strong peak at $2\omega_{rf}$ ($I_{Na}=3/2$) and a small peak at ω_{rf}. From comparison with simulated spectra the quadrupole coupling constants, qcc, could be derived.

Interestingly the 2D-^{23}Na nutation spectrum of hydrated Cl-sodalite shows at room temperature a very broad peak at ω_{rf} with a width at half-height in the F_1-direction of about 60 kHz. At higher temperatures this peak narrowed, at lower temperatures two peaks emerged at ω_{rf} and $2\omega_{rf}$ of about equal intensity. The broad peak at room temperature is interpreted as being due to rapid fluctuations of the electric field gradients (10^5 s^{-1}) by movement of the water molecules. Via rotary echo nutation experiments two different Na sites were discovered, one with a qcc≤0.1 MHz (occurrence of 80%) and another one with qcc=0.4 MHz. The first is attributed to Na ions in sodalite cages containing one or two water molecules and the second to Na ions in sodalite cages containing no water molecules. The mobility of the water molecules is not reflected in the 2D-^{27}Al nutation NMR spectra.

[1] A. Samoson and E. Lippmaa, Chem. Phys. Lett. 100 (1983) 205
[2] R. Janssen, R.E.H. Breuer and E. de Boer, Zeolites '89, in press