# Abstract

The thesis describes the development of instrumentation for single-crystal (SC) NMR and application of SC NMR in studies of quadrupolar nuclei. The instrumentation includes two SC NMR probes of quite different design, an interface between the host-computer of the spectrometer and the SC probes, and a software package ASICS (Analysis of Single-Crystal Spectra). The applications cover studies of the combined effect of the quadrupole coupling and chemical shielding anisotropy (CSA) interactions and crystal structure studies by SC NMR.

Two SC NMR probes with new goniometer designs have been developed. One probe employs a quite traditional design in which the crystal is mounted in the goniometer in three orthogonal orientations, however, with improvements in the accuracy for the stepwise rotation and in the ease of mounting the crystal on the three axes. The other SC NMR probe is designed particularly for improving the sensitivity of small crystals and requires only two different mountings of the crystal. The enhanced sensitivity is achieved by moving the goniometer outside the radio-frequency coil which improves the filling factor of the crystal within the coil and thereby the sensitivity. The improved sensitivity performance of this probe is illustrated by $^{31}$P and $^{87}$Rb SC NMR spectra of small crystals ($< 0.1$ mm$^3$) of $(NH_4)_2HPO_4$ and $RbZn_2(HPO_4)PO_4$, respectively.

The software package, ASICS, is generally applicable for interpretation of SC NMR spectra and as such includes both of the above mentioned SC probe designs. The spectra may be analyzed in terms of the quadrupole coupling, CSA, and dipolar interactions, either a single interaction or their combined effect.

The SC NMR hardware and software have been employed in studies of quadru-

polar nuclei in inorganic salts, in particular for determination of the parameters describing the combined effect of quadrupole coupling and CSA. For evaluation of the quality and reliability of SC NMR and powder methods (magic-angle spinning (MAS), multiple-quantum MAS, and static) to determine these parameters, a series of rubidium salts ($RbClO_4$, $RbVO_3$, $Rb_2SO_4$, $Rb_2CrO_4$, and $RbNO_3$) have been studied by the different $^{87}$Rb solid-state NMR methods. This comparison allows to conclude that SC NMR is a very powerful method for retrieval of these parameters. Furthermore, $^{27}$Al and $^{71}$Ga SC NMR investigations of $\alpha$-$Al_2O_3$ and $Y_3Ga_5O_{12}$ have allowed the first clearcut determinations of $^{27}$Al and $^{71}$Ga CSA's.

In contrast to the powder methods, SC NMR provides the orientation of the solid-state NMR tensors with respect to the crystal frame. This information has been used in $^{27}$Al SC NMR studies of the garnet $Y_3Al_5O_{12}$. From these SC NMR results it is possible to obtain considerable amount of information on the garnet crystal structure. Similarly, from $^{71}$Ga and $^{69}$Ga SC NMR studies of $\beta$-$Ga_2O_3$ twins the relevant twin law for the sample crystal has been determined.