9 Conclusions

This thesis presents the design and construction of two SC NMR probes which represent the "state-of-the-art" in the field of SC NMR probe technologies. In combination with a computer interface for automation of the rotation of the crystal, the three-axis goniometer probe has a very precise angular adjustment of the crystal while the two-axis goniometer probe is designed for sensitivity enhancement of the NMR response from small crystals. The sensitivity-performance is demonstrated employing the smallest crystals used so far in conventional SC NMR studies.

The high precision for the angle setting with the new SC NMR probes allows determination of the solid-state NMR parameters with a high degree of precision. Presently this method has, in particular, been used for determination of the parameters describing the combined effect of the quadrupole coupling and CSA interactions. A comparison of the performance of SC and powder NMR methods for determination of these parameters clearly favours SC NMR with respect to the precision of the parameters and the amount of information which may be extracted from the spectra. The improved precision of the SC NMR method provides the possibility for determination of parameters for even *very* weak interactions as exemplified by the ^{87}Rb SC NMR study of Rb_2SO_4 (site 1) where the CSA is in the order of 3 ppm.

Due to its high-resolution properties and insensitivity to intensity distortions caused by imperfect pulse excitation, SC NMR is a powerful technique in studies of large anisotropic interactions. For example, we have investigated a crystal of β-Ga_2O_3 by ^{71}Ga and ^{69}Ga SC NMR and determined quadrupole coupling constants as large as 18 MHz. In such cases the powder methods struggle with distortions on

the line shape of the very large powder patterns.

In studies of low-γ nuclei the above mentioned properties of SC NMR may be of advantage because the problems normally encountered in solid-state NMR studies of low-γ nuclei (low sensitivity and acoustic ringing of the probe) are less important for the analysis of SC NMR spectra. The performance of SC NMR of low-γ nuclei is exemplified by a natural abundance ^{67}Zn SC NMR study of $Zn(CH_3COO)_2{\cdot}2H_2O$.

In addition to the numerous SC NMR studies presented it is also demonstrated that the parameters for the combined effect of quadrupole coupling and CSA may be obtained from MAS and static-powder NMR methods. The MAS method is probably the more precise of the two methods because of the improved spectral features of MAS spectra. However, its applications are limited to materials with small and intermediate quadrupole couplings as the width of the second-order quadrupolar line shape should not be larger than the applied spinning frequency. Contrary, the static-powder method is not limited by the magnitude of the interactions but the reliability of the parameters determined from the normally quite featureless spectra may be questionable.