

Chapter 1

Introduction

Nuclear magnetic resonance (NMR) spectroscopy is one of the most important techniques used in chemistry today. The use of liquid state ^1H spectra to identify organic compounds, along with infra-red spectroscopy, mass spectrometry and x-ray diffraction, has essentially replaced the chemical techniques used in previous decades. In addition, the use of NMR to examine large biologically active peptides, proteins, nucleic acids and sugars has greatly enhanced the speed and accuracy of structure determination, as well as giving site specific dynamical information. Finally, the use of magnetic resonance to do medical imaging has significantly improved the diagnostic capability of the medical community. Magnetic resonance imaging has provided a tool to do non-invasive examination of patients without the use of harmful high-energy radiation (i.e. x-rays) or radioactive chemical isotopes (i.e. positron emission tomography). However, all of these techniques primarily deal with molecules in solution. The application of magnetic resonance to solid state samples has been limited mostly to chemical physics and physical chemistry laboratories at large research institutes. The last ten years have seen an explosion in the use of solid state NMR. Primarily this has come about due to advances both in multiple-pulse techniques and in sample reorientation experiments. The single most important solid state NMR technique to be developed is cross-polarized magic-angle spinning (CPMAS). This experiment allows routine rapid collection of proton decoupled carbon-13 spectra with nearly liquid-like resolution. The last five years have seen the further advances of multi-dimensional solid state experiments which have continued to enhance information content in solid state NMR spectra.

Throughout this thesis, I will discuss experiments I carried out in the laboratory of Prof. A. Pines at the University of California, Berkeley. In particular, I will describe the advances we made with the technique of dynamic-angle spinning NMR. This technique

was designed to obtain high-resolution liquid-like spectra of solid samples containing quadrupolar nuclei. In this work, I will show the application of this technique primarily to ^{87}Rb and ^{17}O containing compounds. This is by no means a complete or systematic study of all such compounds, but merely a demonstration of the possibilities for the ultimate use of dynamic-angle spinning experiments once these experiments become as routine as CPMAS.