

Experimental Approaches to the Solid-State NMR Spectroscopic Observation of Metal Nuclides in Proteins

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Abstract

We will put forward a general strategy for the observation of a rare spin in a matrix that contains protons and a metal center of interest. The basic idea is to utilize magnetization transfer experiments, e.g. a cross-polarization experiment, whereby we transfer magnetization from protons to a metal center. Because of our interest in metalloproteins we will focus the discussion on biological applications. However, the methods are more general. This same strategy could be applied to problems in catalysis, materials issues, and environmental methods for species characterization. The metal centers of interest to the present discussion are Mg^{2+} , Ca^{2+} , Zn^{2+} , and Cu^{1+} . These nuclides are quadrupolar in nature and that in of itself puts several constraints on the NMR basic experiment. However, again, the methods are not limited to quadrupolar nuclides. The talk will address several issues:

- problems with the basic sensitivity of the NMR experiment (CP, DNP, etc);
- utilization of cryogenic temperatures as a means to *partially* offset the sensitivity issues;
- what have I gotten us into --- probes and relaxation times;
- additional means of improving sensitivity, e.g. MAS *vs* spin echo methods;
- broad lineshapes and methods to recovering tensor information.

Each of these points will be illustrated with examples from our ongoing efforts.