Development of a Field Cycling NMR System for PQR Detection in Biopolymers*

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Z. Naturforsch. **53a**, 269–272 (1998); received October 31, 1997

Our goal is to extend the sensitivity of field cycling pure quadrupole resonance (PQR) methods to be of use in biological systems. The nuclei of interest are ^{25}Mg, ^{67}Zn, ^{43}Ca, ^{11}B and ^{17}O. The experiment is based on a field cycling double resonance technique, in which the quadrupole resonance of a rare nucleus is found through its effect on the magnetic order of the abundant nucleus to which the rare nucleus is coupled through dipole-dipole interaction. A field-cycling NMR spectrometer has been developed, based on our existing 500 MHz high resolution spectrometer. The sample can be shuttled pneumatically from the high field of a commercial 500 MHz magnet to the magnet's top, where the residual field and its gradient is canceled out by a pair of Helmholtz coils. Low field homogeneity is within 0.5 gauss. The ^{1}H signal is observed at high field as a free induction decay (FID) after a 90° pulse. At low field the sample can be irradiated by a digitally tuned RF coil in the 300 kHz–7 MHz range. The sample has to be maintained at low temperature ($\sim$30 K) to avoid relaxation via thermal motion of methyl groups in biomolecules. For this purpose field cycling equipment is placed in a variable temperature dewar (4–300 K). We plan to use solutions of biomolecules in standard cryoprotective buffer, containing $\sim$30% glycerol. Preliminary results on the quadrupole resonance of natural abundance ^{17}O in the cryoprotective buffer and of natural abundance ^{11}B in a protease inhibitor at 50 mM are presented.

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