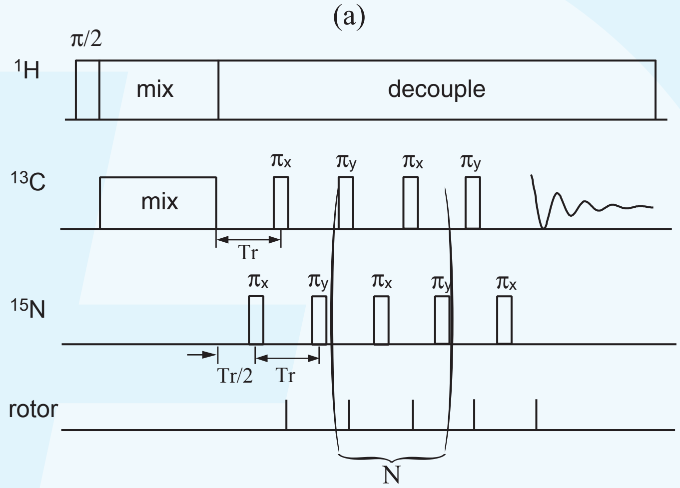
1. Introduction

Rotational-**E**cho **DO**uble-**R**esonance nuclear magnetic resonance (REDOR) is a magic-angle spinning (MAS) experiment for measuring internuclear distances between heteronuclear spin pairs through dipolar interaction.[1, 2] The spin pair consists of two enriched sites, e.g., ^{13}C and ^{15}N in the same molecule. In the REDOR experiment, the sample is spun about the magic angle and cross-polarization from protons is applied to enhance the signal intensity. The ^{13}C and ^{15}N dipolar interaction is averaged due to MAS, thus removed, and reintroduced by a set of rotor-synchronized π pulses on both ^{13}C and ^{15}N which results in a signal intensity reduced spectrum.

2. Pulse sequence

^1H_90: 4 µs
Mixing *rf* field: 50 kHz
^{13}C_180: 8µs
^{15}N_180: 18µs
^1H decoupling: 55 kHz
Phase cycling for π pulses: xy

(a)



(b)

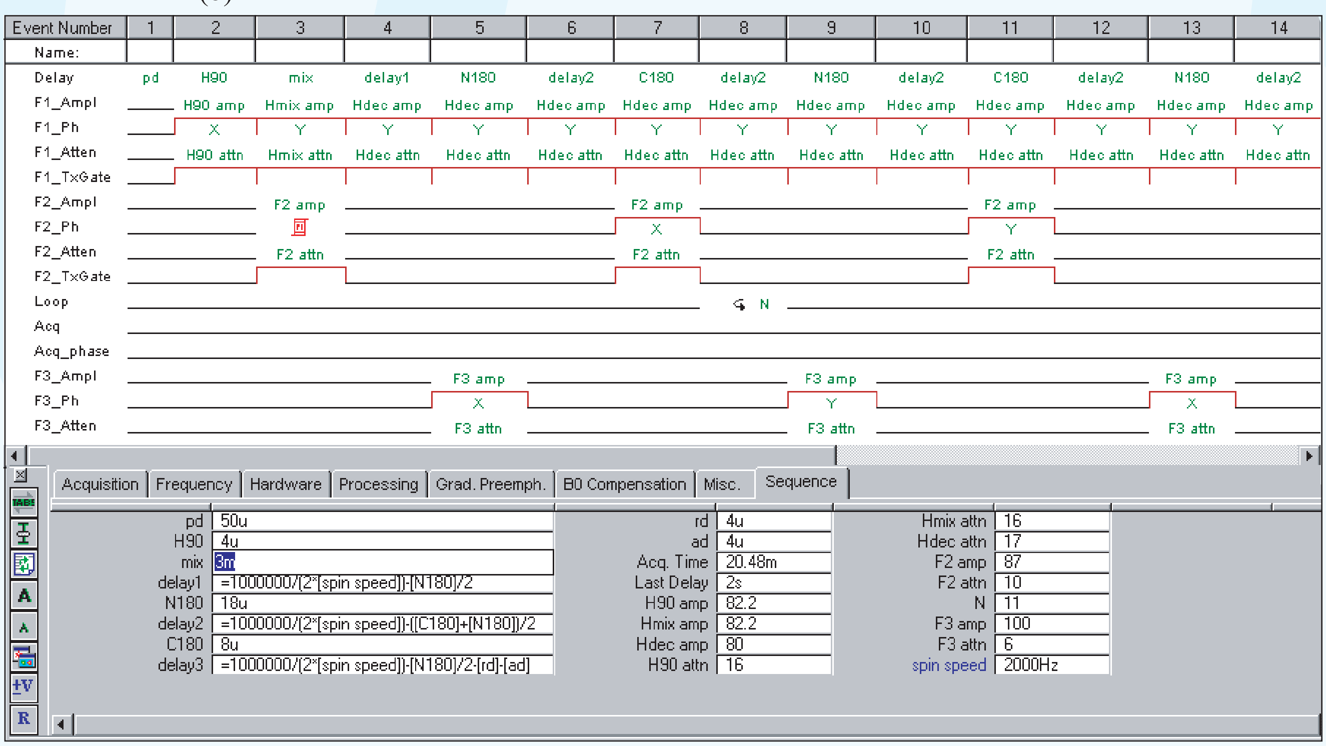


Fig. 1a: REDOR pulse sequence. b: Actual sequence in the NTNMR sequence editor. Delays between π pulses are calculated conveniently by means of the mathematical expressions in the dashboard(Fig.1 b). Update automatically occurs upon entry rotor speed and π pulse width.

3. Experiments

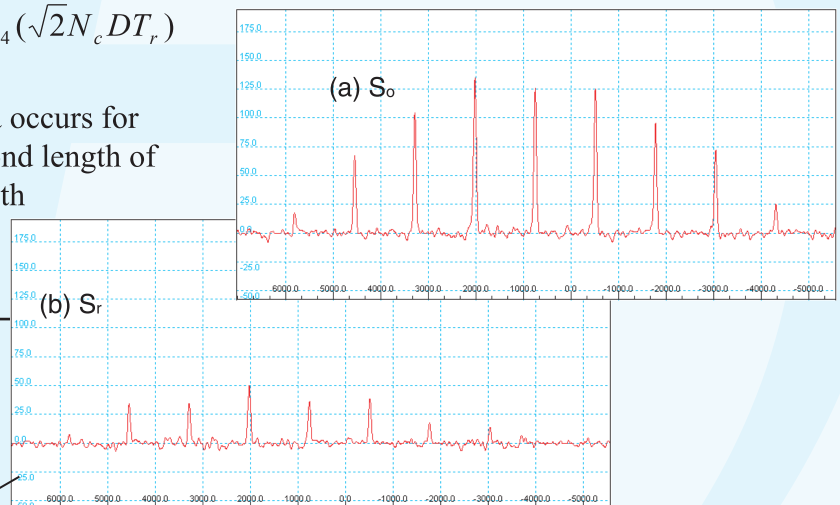
Sample preparation: 1 portion of glycine-1-^{13}C, ^{15}N (>98% Cambrige Isotope Labortories, Inc.) and 9 portions of natural abundance glycine dissolved in 50^{o}C water. The solution is left standing at room temperature and air. Crystals precipitate after a few hours. Spectra were aquired using a 7 Tesla spectrometer and a Doty 5 mm HXY triple resonance MAS probe. The number of rotor cycles during the $^{13}C/^{15}N$ dipolar evolution period was up to 32. The rotor speed was 1.3 kHz.

4. Results

Figure 2 shows the spectra aquired at the end of the 8th rotor cycle as shown in the pulse sequence in Fig.1, without (a) and with (b) π pulses. The ratio of the total intensity of all sidebands in the REDOR spectrum (S_r) to the corresponding spectrum without π pulses (S_o) is plotted as function of $\lambda_d = N_c D T_r$ (Fig.2c). N_c represents the number of rotor cycles, D the dipolar constant, and T_r the rotor period. The solid line in Fig.2c is calculated according to [2]:

$$\frac{S_r}{S_0} = \frac{\sqrt{2}\pi}{4} J_{1/4}(\sqrt{2} N_c D T_r) J_{-1/4}(\sqrt{2} N_c D T_r)$$

The best fit to the experimental data occurs for D = 195 Hz corresponding to the bond length of 2.50 Å. This compares favorably with the X-ray structure (2.486 Å)[3]



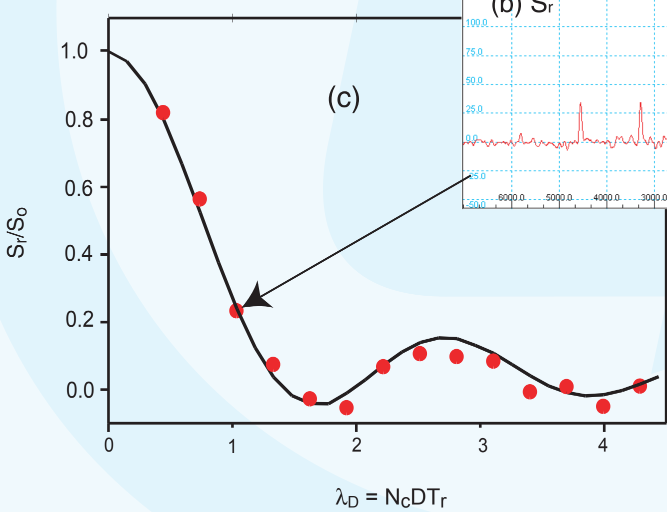


Fig. 2 (a) Full-echo, and (b) dephasing spectrum with two rotor cycles and (c) ratio of REDOR (Sr) to full-echo intensity (So)

5. References

(1) Gullion, T., J. Schaefer, *J. Magn. Reson.* **81,** 196 - 200, 1989.
(2) Gullion, T., *Concepts in Magn. Reson.* **10,** 277-289, 1998.
(3) Legros, J.-P. A. Kivick, A., *Acta Cryst., Sect. B* **36**, 3052, 1980.