1. *Introduction*

$T_{1\rho}$ is the spin-lattice relaxation time in the rotating frame. The NMR signal intensity (M) is measured as a function of the spin-lock duration τ. $T_{1\rho}$ is obtained by fitting the equation:

$$M = M_\infty + (M_0 - M_\infty)e^{-\tau/T_{1\rho}}, \tag{1}$$

where M_0 is the initial magnetization, and M_∞ is the magnetization when the spin system and the lattice reach a quasi-equilibrium during the spin-lock ($M_\infty = 0$ at resonance).

2. *Pulse sequence*

Figure 1 shows the measurement of $T_{1\rho}$ in two situations: (a) Single-resonance: nuclei are excited by a 90° pulse and then spin-locked for a time τ; (b) Double-resonance: a rare spin is excited by cross-polarization from protons and then spin-locked for a time τ.

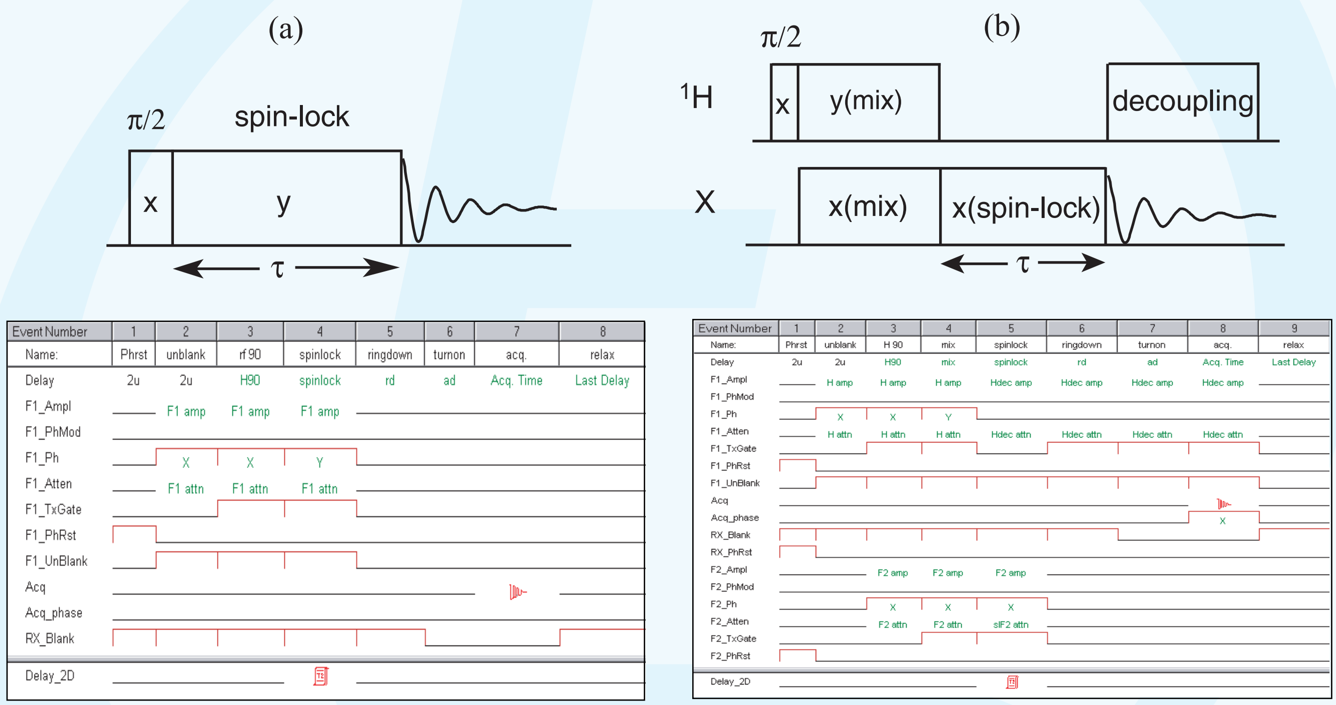


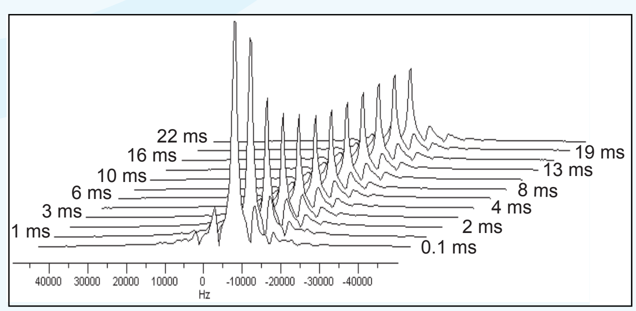
Fig. 1 Sequences for measuring $T_{1\rho}$ in the NTNMR sequence editor.

3. *Experiments and Results*

$T_{1\rho}$ is measured by a series of experiments with different spin-lock durations, i.e. as a 2D-experiment, with a 2D delay table containing a set of spin-lock durations.

Sample 1: Hexamethyl Benzene

Fig. 2. A stacked plot of proton spectra of hexamethyl benzene at different spin-lock durations. The sample was spun at 5.1 kHz. The spectra were obtained with the sequence shown in Fig. 1a. The 90° pulse width is is 2.3 µs. The spin-lock power level is 108 kHz.



Sample 2: 2-^{13}C, ^{15}N-glycine

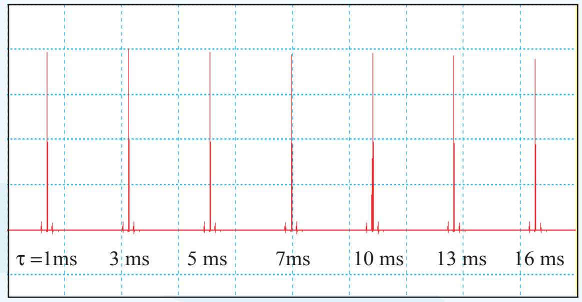


Fig. 3. An array of ^{13}C spectra of 2-^{13}C, ^{15}N-glycine with different spin-lock durations. The sample was spun at 6.8 kHz. The spectra were obtained with the sequence shown in Fig. 1b. The 90° pulse width is 3 μs and the spinlock power level is 56 kHz.

$T_{1\rho}$ Calculation:

1. Phase the spectra.
2. Put the cursor on the desired peak for $T_{1\rho}$ calculation.
3. Open the "Data Analysis" window (Fig. 4a) from the "Tools" menu.
4. Set the "X-values" to τ–table, and the "Y-values" to "Real" and "Intensity".
5. Click "Fit|Add" to open a dialog window for entering the mathematical expression (Fig. 4b).
   □ Enter Eq.1, set M_∞ = [p1], $M_0 - M_\infty$ = [p2], and $T_{1\rho}$ = [p3] together with their initial values. Click "OK" to exit the window.
6. Click the "Draw" button. The result will appear in the window as shown in Fig. 4a.

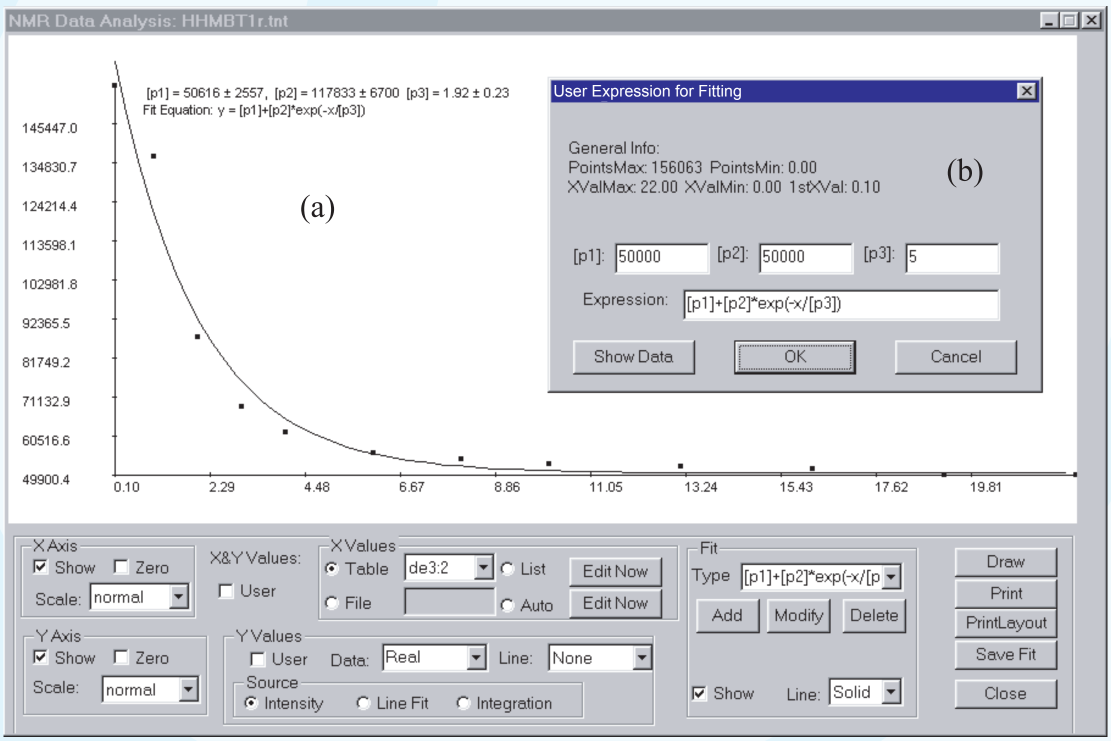


Fig. 4. a: $T_{1\rho}$ fitting for hexamethyl benzene in the "NTNMR Data Analysis" window. Fitting results: $T_{1\rho}$ = 1.92 ± 0.23 ms. b(insert): The dialog window for entering the desired mathematical expression and the initial values of fitting parameters.

4. References

(1) Slichter, C.P., "Principles of Magnetic Resonance", Springer-Verlag, 1990, p.242 - 246.
(2) Bovey, F.A. and Mirau, P.A.,"NMR of Polymers", Academic Press, 1996, p. 81 - 83.