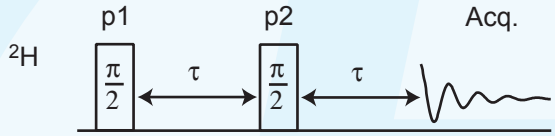
1. Introduction

A solid (quadrapolar) echo refocuses dipolar and quadrupolar couplings. It is generated by a 90° pulse applied at a time τ after the 90° excitation pulse (see Fig. 1a). The two 90° pulses must be 90° out of phase. The echo maximum is at a time τ after the second pulse. The echo delay τ should be smaller than the inverse coupling strength.

2. Pulse sequence

(a)



Phase cycling: P1 P2 Acq.

x	y	x
x	$\bar{y}$	x
y	x	y
y	$\bar{x}$	y
$\bar{x}$	y	$\bar{x}$
$\bar{x}$	$\bar{y}$	$\bar{x}$
$\bar{y}$	x	$\bar{y}$
$\bar{y}$	$\bar{x}$	$\bar{y}$

(b)

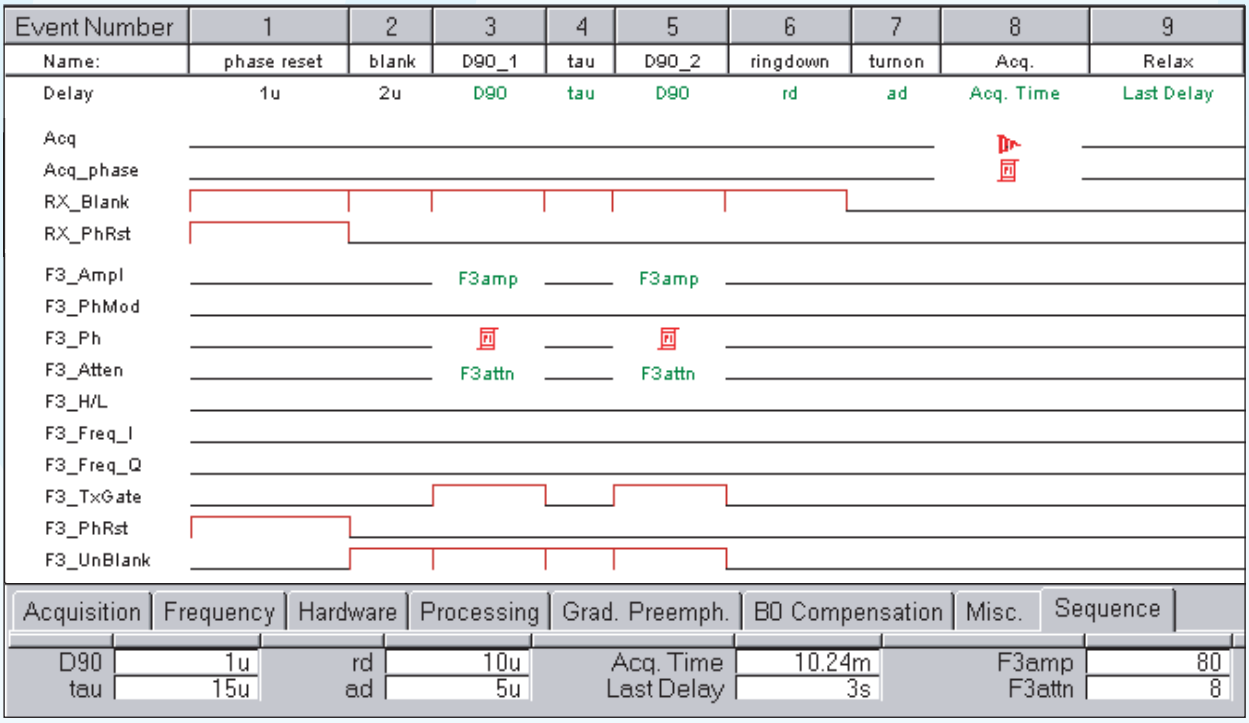


Fig. 1a: Solid echo pulse sequence. The phase cycle on the right side removes spurious signals that are generated if the flip angle of the second pulse deviates from 90°. b: the actual pulse sequence in the NTNMR sequence editor.

3. Experiments

Sample: Hexamethylbenzene (HMB)-D_{18} or glycine-2, 2-D_2, ~20 mg
90° pulse width: 1 µs
Solid-echo delay: 15 µs
Spectrum window: ±50 kHz (HMB); ±500 kHz (glycine)
Recycling Delay: 3s (HMB); 15s (glycine)
Number of scans: 1024 (HMB), 4096 (glycine)
Magnet: 7 Tesla
Console: Discovery HF3
Probe: Home-built probe (original design by prof. Richard. J. Wittebort)

4. Results

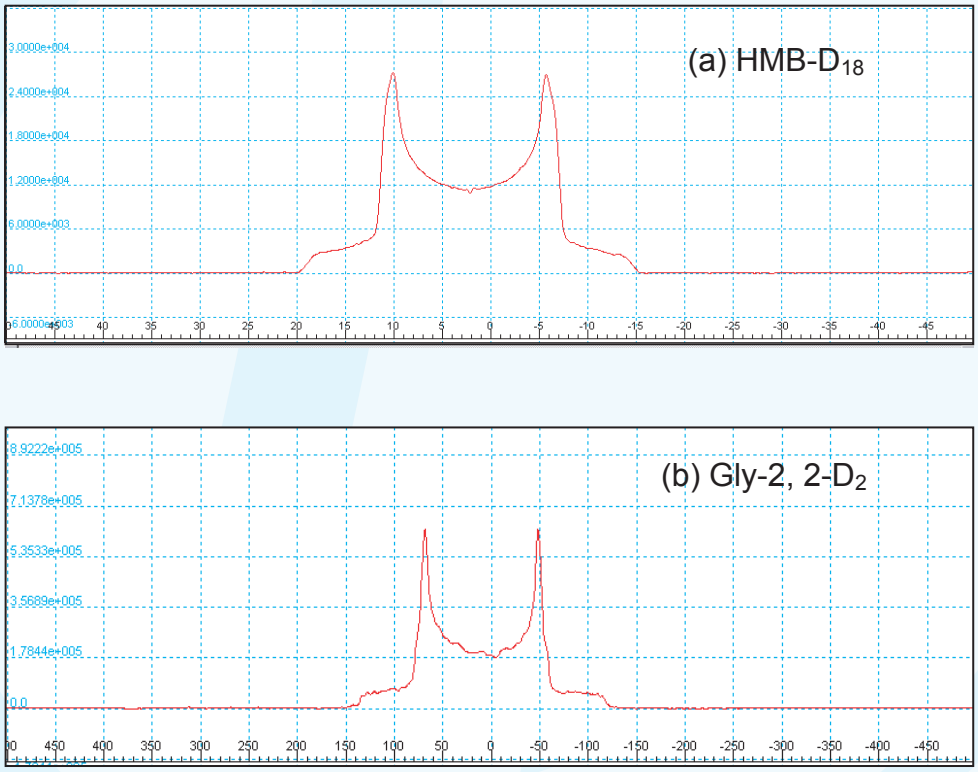


Fig. 2: ^2H spectra of HMB-D_{18} (a) and glycine-2, 2-D_2 (b) obtained with sequence shown in Fig. 1.

5. References

(1) K. Schmidt-Rohr & H. W. Spiess, "Multidimensional Solid-State NMR and Polymers" Academic Press Inc. San Diego, CA, 1999, p.63.
(2) E. O. Stejskal and J. D. Memory, "High Resolution NMR in the Solid State", Oxford University Press, New York, 1994, p. 92.