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Spin Echo with Half-Echo Acquisition V.2



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Protocol status: Working

We use this protocol and it's working

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Abstract

Purpose

To acquire a spin echo spectrum of a spin-1/2 or half-integer quadrupole nucleus with half-echo acquisition.

Scope

This SOP is used to acquire a one-dimensional spin echo spectrum with half-echo acquisition, using a 90°-180° or 90°-90° pulse pair. It is effective at suppressing background signals from rotor or stator components and at mitigating long ringdown periods. When conducted under MAS it should be rotor synchronized. Proper rotor synchronization requires adjustment of the pre-acquisition delay to account for timing delays which may differ from instrument to instrument.

Guidelines

This sequence imposes a T_2' weighting factor on the intensities of the resonances; while typically negligible for short echo periods, this should be considered when using long echo periods.

When used for an integer spin nucleus, a solid echo (90°-90°) must be used instead of a Hahn echo (90°-180°).

Materials

Definitions:

1. MAS: Magic angle spinning
2. ν_{rf} : rf power
3. T_1 : Longitudinal relaxation time constant
4. T_2' : Homogeneous transverse relaxation time constant

Appendix:

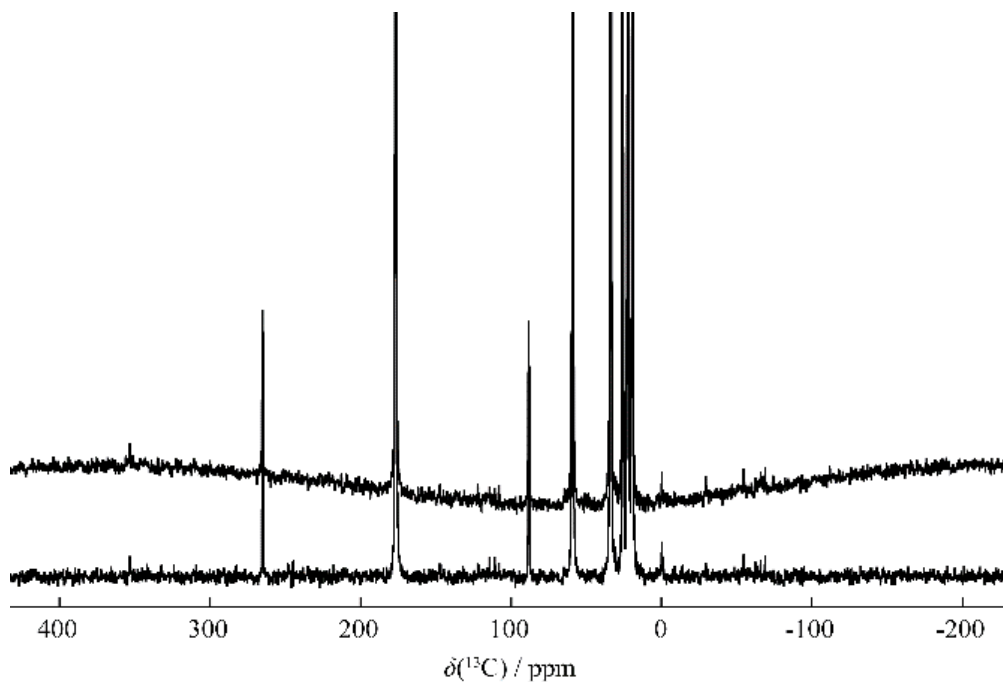


Figure 1. ^{13}C Hahn echo with correctly set value of **d7** and mis-set value of **d7**. While both spectra are correctly phased, the mis-set value leads to a baseline roll, complicating integrations and requiring post-processing.

Before start

User should be familiar with the power limitations and duty cycle of the probe being used.

Expected amount of time SOP will use: 30 minutes

Procedure

- 1 Load the pulse program **hahnecho.nmrfam**.
- 2 Set the following initial parameters:
 - 2.1 Set the 90° pulse length, **p1**, and the 90° pulse power, **plw1**, to a previously optimized 90° pulse.
 - 2.2 Set the length of the echo pulse by setting **cnst2** to either 2 (for a 180° Hahn echo) or 1 (for a 90° solid echo).
 - 2.3 Set the recycle delay, **d1**, to $1.3 \times T_1$ if optimizing for sensitivity, or $5 \times T_1$ if quantitative intensities are desired.

Note

Recall that the intensities are modulated by a T_2' weighting factor.

- 3 Set the spinning rate constant, **cnst31**, to the MAS rate in Hz.
 - 3.1 If the sample is static, i.e., not spinning, set **cnst31** to 1e6.
- 4 Set the rotor period loop counter, **l1**, to an initial value of 1.
 - 4.1 If the sample is static, set **l1** to the desired echo delay in microseconds.
- 5 Set the pre-acquisition delay, **d7**, to the initial value calculated by **del7**.

Note

If **cnst2** is changed, **d7** will need to be manually updated to account for the different echo pulse length.



6 If heteronuclear decoupling is desired, add the **-Ddec** flag to **zgoptns**.

6.1 Set the decoupling program, **CPDPRG2**, pulse length, **PCPD2**, and pulse power, **plw12**, to previously optimized values.

7 Set the acquisition time **aq** to a sufficiently long value such that the FID is not truncated.

Safety information

If high-power decoupling (>10 kHz ν_{rf}) is used, **aq** must be less than 50 ms.

8 Acquire an initial spectrum. If any background signal is sufficiently suppressed, proceed to the next step. Otherwise, increase **l1** and repeat this step.

Note

As **l1** is increased, the accuracy of the relative intensities will be reduced due to the T_2' weighting factor.

9 Observe the baseline of the spectrum. If a significant first-order phase correction (> $|60^\circ|$) is required, adjust **d7** until the required phase correction is minimized.

Note

del7 will typically overestimate **d7** by a few microseconds. Only small adjustments (1 μ s – 10 μ s) should be necessary to obtain an optimal value.

10 Increase the number of scans, **ns**, to a value which will provide adequate signal-to-noise while respecting the phase cycle.

11 Acquire the optimized spectrum.



Protocol references

Y. Nishiyama and N. T. Duong, "Practical guides for ^1H detected solid-state NMR under fast MAS for small molecules," Journal of Magnetic Resonance Open, vol. 10-11, p. 100062, Jun. 2022.

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Protocol

NAME

Saturation Recovery

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Preview

Example Data at 1.1 GHz: usnan.org/ark:/83454/e1df37eab0-5944-47f4-b1c2-3b009a7af51c

Example Data at 900 MHz: usnan.org/ark:/83454/e11ee9e85a-9dc6-46e2-b66d-e47121bc7bae

Example Data at 600 MHz: usnan.org/ark:/83454/e12383feba-4776-4118-a367-a2ee5adc2f69