Jun 26, 2025 Version 2

Hahn Echo for High-Gamma Nuclei V.2

In 1 collection

DOI
https://dx.doi.org/10.17504/protocols.io.14egn63zzl5d/v2

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DOI: https://dx.doi.org/10.17504/protocols.io.14egn63zzl5d/v2

Protocol Citation: Alexander L. Paterson 2025. Hahn Echo for High-Gamma Nuclei. **protocols.io** https://dx.doi.org/10.17504/protocols.io.14egn63zzl5d/v2Version created by **NMRFAM Facility**

Protocol status: Working
We use this protocol and it's working

Created: May 28, 2025

Last Modified: June 26, 2025

Protocol Integer ID: 219030

Keywords: Materials High-Gamma Spin-1/2 Spin Echo, spin echo spectrum, echo acquisition, hahn echo, dimensional spin echo, gamma nuclei purpose, proper synchronization, nuclei, background signals from rotor, stator component, gamma, timing delay, rotor

Funders Acknowledgements:
National Science Foundation
Grant ID: 1946970

Abstract

Purpose

To acquire a spin echo spectrum of highly sensitive high-gamma (*i.e.*, ^{1}H, ^{19}F) spin-1/2 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° 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 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.

Both ^{1}H and ^{19}F spectra frequently suffer from significant background signals, both from the probe and sometimes from within the rotor itself (*e.g.*, caps and seals). Echoes are very effective for reducing background signals from outside the coil, but much less effective for signals originating inside the coil.

Materials

Definitions:

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

Appendix:

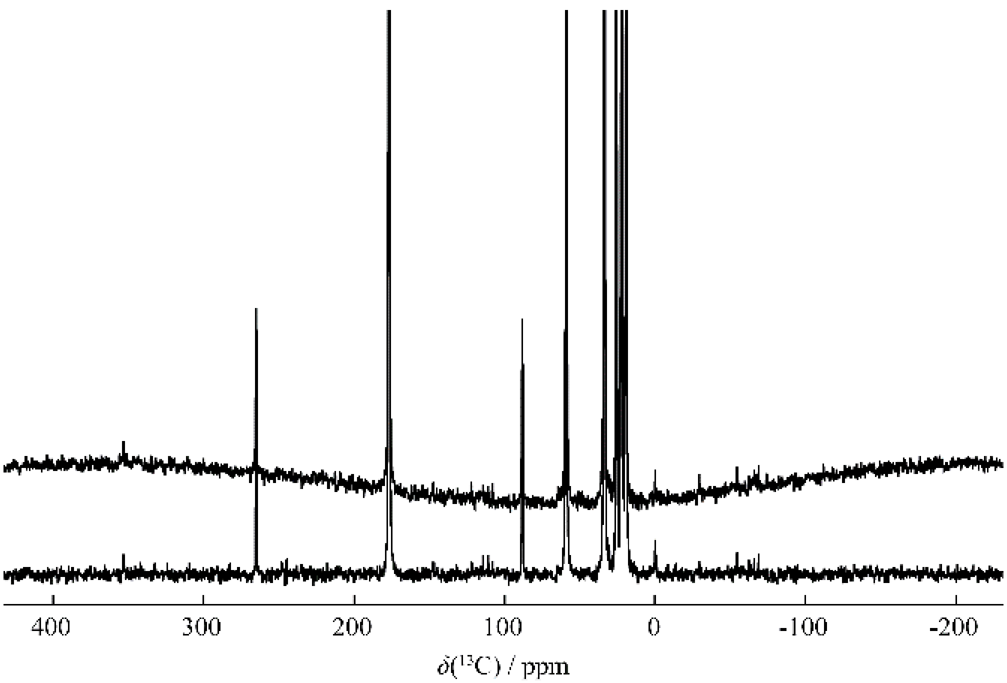

Figure 1. ^{13}C Hahn echo with correctly set value of **d7** and mis-set value of **d7**. While both spectra are correctly phased, the mis-set value leads to a baseline roll, complicating integrations.

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 **hahnecho_shortcycle.nmrfam** pulse program.

2 Set the 90° pulse length, **p1**, and the 90° pulse power, **plw1,** to previously optimized values.

3 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).

4 Set the recycle delay**, d1**, to 1.3 × T_1 if optimizing for sensitivity, or 5 × T_1 if quantitative intensities are desired.

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

5 Set the spinning rate constant, **cnst31**, to the MAS rate in Hz.

5.1 If the sample is static, set **cnst31** to 1e6.

6 Set the rotor period loop counter, **l1**, to an initial value of 1.

6.1 If the sample is static, set **l1** to the desired echo delay in microseconds.

7 Set the pre-acquisition delay, **d7**, to the initial value calculated by **del7**.

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

9 Set the number of scans, **ns**, to an initial value of 3.

10 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.

11 Observe the baseline of the spectrum. If a significant first-order phase correction (> |60°|) 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 µs – 10 µs) should be necessary to obtain an optimal value.

12 Increase the number of scans, **ns**, to a value which will provide adequate signal-to-noise while respecting the phase cycle (3×*n*).

13 Acquire the optimized spectrum.

Protocol references

Y. Nishiyama and N. T. Duong, “Practical guides for ^{1}H detected solid-state NMR under fast MAS for small molecules,” Journal of Magnetic Resonance Open, vol. 10-11, p. 100062, Jun. 2022. DOI: **10.1016 j.jmro.2022.100062**

Protocol

NAME

Saturation Recovery with Half-Echo Acquisition

CREATED BY

NMRFAM Facility

Preview

Example Data at 1.1 GHz: **usnan.org/ark:/83454/e1f1857934-0e88-4ff6-990b-438ca1af7cc2**
Example Data at 900 MHz: **usnan.org/ark:/83454/e1b4d6922f-21ab-47d5-a7d0-09c8cdc1293b**
Example Data at 600 MHz: **usnan.org/ark:/83454/e1fb6faae8-9d3e-4476-9fe0-00b7c45dca35**