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Spin Echo with Whole-Echo Acquisition V.1

In 1 collection

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Protocol status: In development
We are still developing and optimizing this protocol, but it should be functional. We hope to solicit feedback primarily on clarity and usability. We intend to publish it in June 2025.

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Abstract

Purpose
Acquisition of a Hahn-echo spectrum with whole-echo acquisition from a quadrupolar nucleus.

Scope
This protocol is part of a standard array of experiments to be run on a new sample. It can also be used as preparation for MQMAS experiments, particularly those involving split-t_1 acquisition. This protocol can also be used when there is a significant probe or rotor background observed in the spectrum.

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. MQMAS: Multiple-quantum magic angle spinning
2. MAS: Magic angle spinning
3. CT: Central Transition

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 pulse **p1** and power **plw1** to a central-transition-selective 90° pulse.

Note

If acquiring data on a spin-1/2 nucleus, ignore all references to central-transition selective and use a 90° pulse.

3 Set **cnst2** to 2 to produce 180° CT-selective echo pulse, or to 1 to produce a 90° CT-selective echo pulse.

Note

If the observed nucleus is integer-spin, **cnst2** must be set to 1.

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 a T_2' weighting factor.

5 If collecting a MAS spectrum, set **cnst31** to the MAS rate. If collecting a static, i.e., not spinning, spectrum, 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 1 µ.

Note

This initial value will ensure that the whole echo is acquired, but might allow for residual FID to bleed through for small values of **l1**.

8 Set **ns** to a multiple of 16.

9 Acquire an initial spectrum.

10 Process the spectrum according to procedure for full-echo acquisition.

10.1 This involves setting a large first-order phase correction **phc1**.

10.2 Apodization can be applied using Gaussian, sine bell, or squared sine bell functions.

10.3 If half-echo acquisition is desired, left-shift the spectrum until the echo top is the beginning of the FID. With half-echo acquisition the large first-order phase correction **phc1** is not required and exponential apodization can be used.

11 Increase **l1** if necessary to either remove background signals or to prevent residual FID from the excitation pulse.

11.1 If removing background signal from the spectrum, increase **l1** until the background is no longer observed.

11.2 If testing for MQMAS with split-t_1 acquisition, increase **l1** until residual FID is no longer overlaps with the whole echo.

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

13 Acquire the optimized spectrum.

Protocol references

Duer, M. Solid-State NMR Spectroscopy: Principles and Applications; Blackwell Science: Malden, MA, 2002.

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Preview