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Quantitative Bloch Decay for Spin-1/2 Nuclei V.3

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

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Abstract

Purpose

To acquire a 1D MAS spectrum of a spin-1/2 nucleus with quantitative intensities.

Scope

The acquisition of 1D NMR spectra of spin-1/2 nuclei with quantitative relative intensities requires attention to be paid to the T_1 relaxation of each resonance. This SOP should only be followed when quantitative relative intensities are required. If only lineshapes or chemical shifts are required, the **d1** relaxation period can be reduced in duration.

Guidelines

If there is a significant rotor or probe background, an echo sequence may be more appropriate.
The quantitative intensities described here are relative intensities, not absolute intensities. Determining absolute intensities, sometimes known as spin counting, is outside the scope of this SOP.

Materials

Definitions:

1. T_1: Longitudinal relaxation time constant

Safety warnings

High-power decoupling can cause damage to a probe if applied incorrectly. Ensure that the decoupling power and duration are set within safe limits.

Before start

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

User should be familiar with the maximum safe spinning speed of the probe and rotor being used.

Accurate T_1 relaxation times and calibrated pulse lengths must have been obtained prior to starting.

The expected amount of time to completion is highly sample-dependent and cannot be accurately estimated ahead of time.

Procedure

1 Load pulse sequence **hpdec**.

2 Set the relaxation time **d1** to 5 × T_1, where T_1 is the largest value previously measured.

3 Set the pulse length **p1** and pulse power **plw1** to a previously optimized 90° pulse.

4 Set the ^{1}H decoupling program, **CPDPRG2**, pulse length, **PCPD2**, and pulse power, **plw12**, to a previously optimized ^{1}H decoupling condition.

4.1 If no ^{1}H decoupling is required, set the pulse power **plw12** to 0 W.

5 Set the acquisition time **aq** to a sufficiently long value such that the FID is not truncated, but less than 50 ms if decoupling is used.

Safety information

Applying high-power decoupling for more than 50 ms can cause physical damage to the probe.

Safety information

Ensure that the relaxation time **d1** is long enough to respect the duty cycle of the probe. This typically requires **d1** to be at least 20x longer than **aq**.

6 Adjust **ns** to effectively make use of available experimental time while achieving sufficient signal-to-noise ratios.

Note

If a good number for **ns** cannot be estimated, spectra can be acquired in blocks and added via **fidsum**.

7 Process the spectrum while paying careful attention to background subtraction and baseline correction if necessary.

8 Obtain the relative intensities of the resonances via integration if well-resolved, or deconvolution if not.

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. https://doi.org/10.1016/j.jmro.2022.100062

Protocol

NAME

Saturation Recovery

CREATED BY

NMRFAM Facility

Preview

Example Data at 1.1 GHz: **usnan.org/ark:/83454/e1d5056070-adc9-42a0-8c4c-0dd64ea732d4**
Example Data at 900 MHz: **usnan.org/ark:/83454/e1dee19c5b-b543-42d6-a01d-3f8e3dd3856d**
Example Data at 600 MHz: **usnan.org/ark:/83454/e18ed3ed7b-c9d3-4afa-8ce0-d1664b865868**