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CP-Based Saturation Recovery V.2

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We use this protocol and it's working

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Abstract

Purpose

To measure the longitudinal relaxation time, T_1, of ^{1}H present in the sample, while observing non-^{1}H spin-1/2 nuclei.

Scope

This SOP should be completed after an initial 1D cross-polarization (CP) experiment has been completed, but before further experiments are performed. Upon completion of this SOP, the user will be able to set recycle delays appropriate for the purpose of later experiments, either to maximize signal per unit time, or to ensure quantitative intensities.

Guidelines

This SOP does not require a well-optimized CP condition. Once an initial CP signal is observed, this SOP can be completed prior to further CP optimization, especially if a very fine CP condition optimization is to be completed.

If acquisition of a 1D spectrum requires substantial signal averaging, this SOP may not be appropriate.

This SOP is written for acquisition using a Bruker console and TopSpin 3.6.2. It may be adapted to other TopSpin versions where appropriate.

This SOP is intended for a beginning user who has been trained on basic spectrometer safety and principles.

Materials

Definitions:

1. T_1: Longitudinal relaxation time
2. l20: Number of saturation pulse loops
3. d20: Presaturation loop delay
4. CP: Cross polarization

Appendix:

If the presaturation train is not completely effective, the recovery curve might initially decrease, and then follow the expected exponential curve. This is common behaviour and can be dealt with by discarding the initial points of the curve.

The values of the variable delay list T1_standard_array range from 0.25 to 128 s, increasing by a factor of two for each increment. This should provide adequate spacing to accurately determine T_1 values between 1 s and 64 s. If necessary, adjust the list for shorter or longer values of T_1.

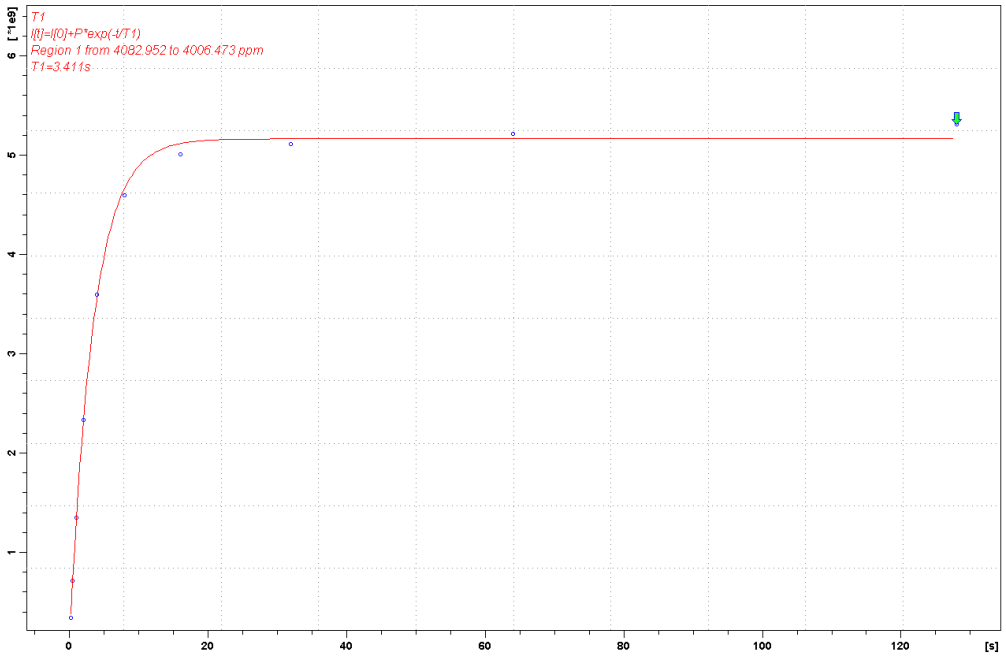

Figure 1.Example of a successful ^{7}Li saturation recovery experiment performed on a lithium silicate glass. The T_1 of this environment was found to be 3.4 s.

Safety warnings

CP-based experiments are assumed to use high-power decoupling. Therefore, **d1** should be about 20 times as long as the acquisition period **aq**, typically on the order of 1 s. A long acquisition (50 ms) protection limit is set. This protection limit should not be disabled while high-power decoupling is in use.

Before start

User is responsible for knowing the duty cycle of the probe and instrument being used.

User is responsible for knowing the maximum power limits of the probe.

Expected time to completion: 1 hour or less

Procedure

1 Prepare the experimental environment.

1.1 Create a new experiment window.

1.2 Load the **cphsatrect1** pulse program.

1.3 Set the ^{1}H excitation pulse length, **p3**, and pulse power, **plw12**, to previously optimized ^{1}H powers.

1.4 Set the ^{1}H contact pulse power, **spw0**, and the X contact pulse power, **plw1**, to previously optimized values.

1.5 Set the CP contact time, **p15**, appropriate to the sample. If the optimum value is not known, set a value of 1 ms.

1.6 Set the CP ramp shape, **spnam0**, to ramp70100.100.

1.7 Set the ^{1}H decoupling program, **CPDPRG2**, pulse length, **PCPD2**, and pulse power, **plw12**, to previously optimized values

1.8 **aq:** Set to 50 ms or less. This is required for the safety of the probe.

1.9 Set the recycle delay, **d1**, to a relatively long value, *e.g.*, 10 s.

1.10 Set the number of saturation pulse loops, **l20**, to 0.

1.11 Set the variable delay list**, vdlist,** to T1_standard_array.

2 Acquire an initial experiment. The number of scans should be as small as practicable to allow for adequate signal, ideally as low as 2.

2.1 Ensure that all expected resonances are visible. If some are not visible, either reduce the pulse length or increase **d1**, as appropriate.

3 Set the number of saturation pulse loops, **l20**, to an initial value of 10. Set the presaturation loop delay, **d20**, to an initial value of 100 µs.

4 Acquire a spectrum. There should be no signal present.

4.1 If signal is still present, increase **l20**, up to a maximum of 100, and reacquire the spectrum.

4.2 If increasing **l20** does not result in a null signal, reduce the number of loops and retry with longer presaturation loop delays **d20**.

4.3 If increasing **d20** does not result in a null signal, retry with shorter presaturation loop delays **d20**.

5 Convert the experiment to a 2D experiment window.

5.1 Move to the acqupars window via the command **eda**.

5.2 Click on the button titled “Change acquisition dimension of current data set” and change the dimension to 2D.

5.3 Set the following parameters:

1. **F1 TD**: 12
2. **FnMODE**: QF
3. **F1 SI**: 16
4. **d1**: A minimal value that respects the probe duty cycle, see Appendix.

6 Acquire the 2D experiment via the command **zg**.

7 Process the 2D experiment.

7.1 Transform the data using the command **xf2**.

7.2 Correct the F2 phasing of the slice with the longest delay period.

> Note
>
> Do not transform or phase the F1 axis.

8 To process the data, launch the TopSpin utility **t1guide** and follow the workflow in the utility.

8.1 Extract the slice with the longest variable delay, *i.e.*, the most recently acquired slice.

8.2 Integrate each resonance. Save using the option “Export Regions to Relaxation Module and .ret”.

> Note
>
> 1. If resonances are not resolved, attempt to select the regions of least overlap.
> 2. If resonances cannot be reasonably separated, integrating over all of the peaks will allow for the determination of the longest T_1 component.

8.3 Enter the relaxation window and set the Fitting Function Function Type to **uxnmrt1**.

8.4 Calculate fit for all peaks. If successful, the fitting window should resemble the example in the appendix.

9 Record the T_1 for each site. Use the longest value of T_1 for determining the recycle delay of future experiments.

Protocol references

Bruker Solid State NMR User Manual Z4D10641B, Section 16.2.4

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

CPMAS

CREATED BY

NMRFAM Facility

Preview

Example Data at 1.1 GHz: **usnan.org/ark:/83454/e115fdac66-bced-4c06-9340-0da41dfab79e**
Example Data at 900 MHz: **usnan.org/ark:/83454/e12b506b75-dce6-4c12-aa6c-6aec0f7a1253**
Example Data at 600 MHz: **usnan.org/ark:/83454/e1e037ed5b-31af-4311-982d-798c2c9ebe00**