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CRAMPS V.2

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Protocol status: Working

We use this protocol and it's working

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Abstract

Purpose

To obtain one-dimensional high-resolution MAS spectra of systems with broadening originating from significant homonuclear dipolar coupling, *i.e.*, ^1H and ^{19}F .

Scope

This protocol should be followed when attempting to obtain high-resolution 1D ^1H or ^{19}F spectra when ultrafast (> 60 kHz) magic angle spinning is unavailable or impractical. This protocol can require substantial optimization and produces spectra where the chemical shifts are affected by a scaling factor; if ultrafast MAS is available, it should be seriously considered before proceeding with this protocol.

Guidelines

This protocol uses the LG4 homonuclear decoupling sequence, which may not provide the strictly optimal resolution increase in all spinning regimes. However, it should perform quite well in most spinning regimes and has a very homogeneous scaling factor, making it a robust default choice. If the resolution provided by LG4 is insufficient, consult with your facility manager about other decoupling sequences.

Homonuclear decoupling benefits strongly from rf homogeneity. "Centre-packing", *i.e.*, restricting the sample to the central third of the rotor, will often result in improved resolution when using this protocol. Consider the filler material carefully to avoid the inadvertent introduction of background signals.

Temperature control of the sample via VT is important to stabilize tuning and minimize sample heating from the high-power rf pulses. It is recommended even for temperature-stable samples, as long as spinning stability is not negatively impacted.

Materials

Definitions:

1. MAS: Magic angle spinning
2. CRAMPS: Combined rotation and multiple pulse spectroscopy
3. rf: Radiofrequency
4. VT: Variable temperature
5. τ_c : Decoupling cycle length
6. τ_r : Rotor period

Appendix:

In the case of a low-sensitivity sample, the CRAMPS optimization can be performed on a reference compound prior to running the experiment on the sample of interest. However, it is critical to not change the transmitter offset between the reference compound and the sample of interest; doing so can cause errors in chemical shift referencing.

If the sweep width is wide and the decoupling element (**p10** and associated compensation pulses) is long, spurious spectra can appear in the window. These are an artifact of insufficient sampling of the FID and can be ignored.

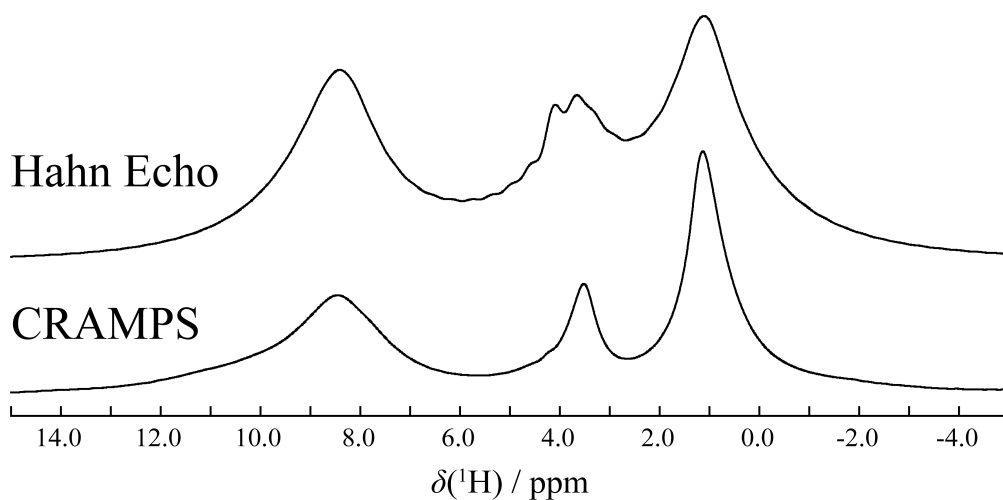


Figure 1. ^1H Hahn echo (top) and CRAMPS (bottom) spectra of alanine.

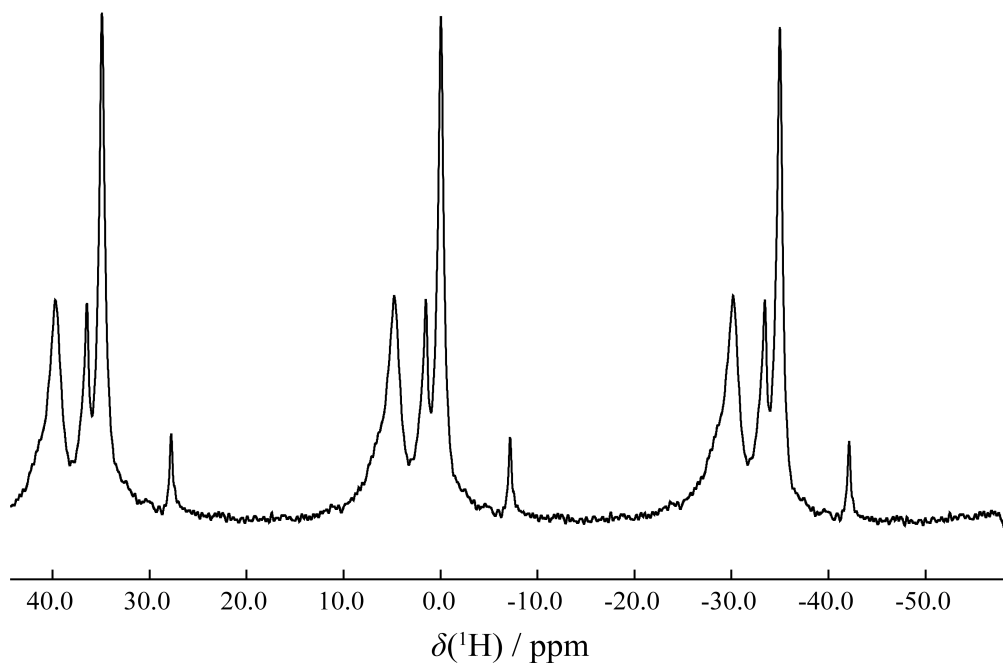


Figure 2. ¹H CRAMPS spectrum of alanine during optimization. Note the wide spectral window. Only the peaks around 0 ppm are real; the rest are artifacts due to limited sampling of the FID.

Before start

This protocol directly optimizes power levels, and hence is intended for intermediate or advanced users. The maximum safe probe power must be known before using this protocol.

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

User is responsible for knowing the maximum spinning rate of the probe and rotors being used.

This SOP has only been tested on Avance III HD spectrometers, and may not function on older systems. As of 2025-01-01, it is known to not function on Avance NEO spectrometers.

Expected amount of time SOP will use: 2 hours

Procedure

- 1 Load the sample into the probe and achieve stable spinning, temperature, and tuning.
- 2 Acquire an initial zg spectrum to ensure that the transmitter frequency, **o1** or **o1p**, is set to a reasonable initial guess that does not overlap with any obvious peaks.
- 3 Load the **dumbo.fmp** pulse sequence
- 4 Run the **lg4** AU program using the following initial values:
 - 4.1 Number of points for each LG4 cycle: 120
 - 4.2 Number of cycles in the shape file: 3
 - 4.3 Alpha value: 70
 - 4.4 Offset angle for the shape: 0
- 5 Set initial values:
 - 5.1 Set the 90° pulse length, **p12**, and pulse power, **plw12**, to previously optimized values.
 - 5.2 Set the decoupling pulse power, **plw13**, to 0 W.
 - 5.3 Set the compensation pulse power, **plw1**, to 0 W.

- 5.4 Set the decoupling pulse length, **p10**, to the shortest allowable value. This may be between 10 μ s and 20 μ s, depending on your spectrometer.
- 5.5 Set the pre-pulse length, **p14**, to 0.1 μ s.
- 5.6 Set the first compensation pulse length, **p22**, and second compensation pulse length, **p21**, to 0.1 μ s.
- 5.7 Set **p8**, an additional acquisition delay period, to 0.
- 5.8 Set the number of acquisition points, **l11**, to 16.
- 5.9 Set the phase constants (**cnst21**, **cnst22**, **cnst25**) to 0.
- 5.10 Set **cnst31** to the MAS spinning rate in Hz. This is only used for calculating ratios, not for pulse or delay timings.
- 6 Ensure that the acquisition time, **aq**, is within the probe safety and duty cycle limit. An initial value of 10 ms is recommended.
- 7 Acquire an initial CRAMPS spectrum with the above initial guesses. Ensure that the entire spectrum is comfortably within the spectral window; the peak positions may change as parameters are optimized. This spectrum should not be particularly different than the initial one-pulse experiment.
- 8 Enable decoupling by setting **plw13** to a low value, *e.g.*, 10 W. Save this processed spectrum using **wrp** and refer back to it if comparisons are needed to evaluate performance.
- 9 Optimize the transmitter offset, **o1**, in coarse steps (*e.g.*, 500 Hz for ^1H) to avoid overlap with resonances of interest.

Note

This will likely be on the right side of the spectrum, close to but not overlapping with the rightmost peak.



- 10 Ensure that the axial peak is sufficiently suppressed. This step can be repeated any time the axial peak exceeds the intensity of the most intense real peak.
 - 10.1 Begin by optimizing the pre-pulse length, **p14**, from 0.1 μs to 1.5 μs in steps of 0.25 μs
 - 10.2 Then optimize the pre-pulse phase, **cnst25**, from 0° to 360° in steps of 30°.
 - 10.3 If necessary, repeat the optimization using smaller step sizes and a smaller search range.
 - 10.4 Any time the axial peak is more intense than the most intense real peak, the pre-pulse phase and pre-pulse length should be refined.
- 11 Iteratively optimize the decoupling elements. This may require several loops.
 - 11.1 Coarsely optimize the decoupling pulse length, **p10**, from as short as allowed to 100 μs in steps of 5 μs . The optimum pulse length will result in maximum resolution.
 - 11.2 Select the pulse length range that provides the best resolution. Finely optimize **p10** over this range in steps of 1 μs , again selecting best resolution.
 - 11.3 Note that if the decoupling cycle time ($\tau_c = \mathbf{p9} + \mathbf{p10}$) synchronizes with the rotor period (such that $\tau_c/\tau_r = n/2$), resolution will be strongly reduced.
 - 11.4 Once **p10** is optimized, optimize the decoupling pulse power, **plw13**, from 1 W to the probe maximum power in steps of approximately 10% of the probe maximum power.

Note

Be cautious of overly-long pulse lengths; serious artifacts can result.

Safety information

Be absolutely sure of the probe maximum power before proceeding.



- 11.5 Select the pulse power range that provides the best resolution. Finely optimize the **plw13** over this range in steps of approximately 5% of the probe maximum power.
- 11.6 Optimize the transmitter offset in fine steps (*e.g.*, 200 Hz for ^1H).
- 11.7 Return to step 9 and repeat all optimization steps with smaller step sizes. Continue until a satisfactory spectral resolution is found. [⇒ go to step #9](#)
- 12 Optimize the acquisition window elements.
 - 12.1 Adjust the number of acquired complex points, **l11**, from 8 to 32 in steps of 2.
 - 12.2 Adjust the receiver delay, **p8**, from 0 μs to 1 μs in steps of 0.1 μs .
- 13 Optimize the compensation pulse phases. First set the compensation pulse power, **plw1**, to the same value as **plw13**, and the compensation pulse lengths, **p22** and **p21**, to 0.3 μs .
 - 13.1 Optimize the phase adjustment of the second compensation pulse, **cnst21**, from 0° to 360° in steps of 5° . Optimize for spectral resolution and not the behaviour of the axial peak.
 - 13.2 Optimize the phase adjustment of the first compensation pulse, **cnst22**, from 0° to 360° in steps of 5° . Optimize for spectral resolution and not the behaviour of the axial peak.
- 14 Optimize the compensation pulse lengths.
 - 14.1 Optimize the length of the second compensation pulse, **p21**, from 0.1 μs to 1.2 μs in steps of 0.1 μs .
 - 14.2 Optimize the length of the first compensation pulse, **p22**, from 0.1 μs to 1.2 μs in steps of 0.1 μs .
 - 14.3 Iterate between these two steps, reducing the step size and search range each time, until optimal resolution is achieved.



- 15 Fine-tune the pre-pulse length, **p14**, and phase, **cnst25**, over a narrow range with small step sizes, until the axial peak is optimally suppressed.
- 16 Acquire a high-quality spectrum with sufficient S/N.
- 17 Correct for the chemical shift scaling factor.
- 17.1 If the MAS spectrum has two sufficiently resolved peaks:
 1. Compare the distance between the two peaks in the MAS spectrum with the distance between the two peaks in the CRAMPS spectrum to obtain the scaling factor.
 2. Divide the acquisition spectral width (**s swh**) by the scaling factor and Fourier transform the FID.
 3. Calibrate the chemical shift of the CRAMPS spectrum using the chemical shifts of the MAS spectrum
 4. If the MAS spectrum does not have sufficiently resolved peaks, acquire a CRAMPS spectrum of a reference compound (e.g., α -glycine) using *exactly* the same parameters as the sample of interest.



Note

Exactly is not an understatement. Do not adjust the probe tuning; if the reference compound requires the tuning to be adjusted, choose a different reference compound.

Protocol references

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Protocol

NAME

Saturation Recovery with Half-Echo Acquisition

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Preview

Example Data at 900 MHz: usnan.org/ark:/83454/e1aba32d00-eae4-4f2f-9290-da5b2a6ffb30

Example Data at 600 MHz: usnan.org/ark:/83454/e156f805af-438c-4f36-b011-25becc7eff58