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Quadrupole Non-Selective Pulse Width Optimization V.1

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Protocol status: In development
We are still developing and optimizing this protocol. We intend to publish it in June 2025.

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Abstract

Purpose
Calibration of non-selective 90° pulse width using a standard sample for a quadrupolar nucleus.

Scope
This protocol uses a standard sample with high sensitivity and low quadrupolar coupling constant. This can be a solid sample with negligible C_Q (*e.g.*, NaCl for ^{23}Na) or a solution sample (*e.g.*, 1 M NaCl for ^{23}Na).

Guidelines

This SOP should be among the first executed when investigating a new sample.

This SOP is written to the level of an experienced NMR spectroscopist who is unfamiliar with the acquisition of spectra from quadrupolar nuclei.

Calibration and validation of a central transition-selective pulse is outside the scope of this SOP; CT-selective pulses should be calibrated on the sample of interest using SOP Materials Central-Transition-Selective Pulse Width Optimization.

Materials

Definitions:

1. CT: Central Transition
2. C_Q: Quadrupolar coupling constant

Appendix:

The 360° zero crossing is less reliable in half-integer quadrupoles than in spin-1/2 nuclei. The 180° zero crossing point is preferred.

The nutation curve of will be non-sinusoidal if the relaxation delay **d1** is too short; however, the 180° zero crossing should be reliable regardless of precise relaxation time.

Safety warnings

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

Before start

This protocol is intended for a high-symmetry standard sample with low C_Q, such as NaCl for ^{23}Na. A 1 M solution of an appropriate salt can also be used.

Probe must be well-tuned and well-matched.

SOP written for Bruker spectrometers.

Expected completion time: <1 hour

Procedure

1 Open a new experiment window and load the **zg** pulse sequence.

2 Load previously known good pulse length **p1** and pulse power **plw1**.

Note

If previously known good values aren't available, a reasonable starting point is a 1 µs pulse using a power level from a nearby nucleus, e.g. ^{13}C and ^{23}Na.

Safety information

If there is no information at all on previously known pulse lengths and powers in this frequency range, consult the local expert responsible for the probe being used. If you are that expert and need guidance, consult with the probe vendor.

3 Acquire an initial spectrum and ensure that there is adequate signal. If signal is inadequate, increase the number of scans and repeat this step.

4 Open a **popt** window.

5 Array pulse width **p1** from 1.0 µs to 8 µs in steps of 0.5 µs. There should be 15 experiments.

6 Run the popt and observe the nutation curve.

7 Determine the 90° pulse length by finding the pulse length corresponding to the 180° zero crossing point and dividing it by 2.

7.1 If a more accurate determination is required, **popt** can be run again with revised start, end, and step values.

8 Set **p1** to this value.

9 If a particular radiofrequency power is desired, use the **pulse** au program to estimate the amplifier power required and optimize the pulse power **plw1** using **popt**.

9.1 Confirm the pulse power limitations of the probe prior to optimizing **plw1** and ensure that the optimization routine will not allow for the power limit to be exceeded.

10 Repeat this protocol until the pulse length and power are optimized to the level required.

Protocol references

Wasylishen, R. E.; Ashbrook, S. E.; Wimperis, S. NMR of Quadrupolar Nuclei in Solid Materials; WILEY, 2012-08-07.