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Quadrupolar Non-Quantitative Bloch Decay V.1

In 2 collections

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Alexander L. Paterson[1]

[1]National Magnetic Resonance Facility at Madison (NMRFAM), University of Wisconsin-Madison, Madison, WI, United States

NMRFAM Facility
UW-Madison

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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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Keywords: Non-Quantitative MAS Bloch Decay: 23Na, 1d nmr spectra of quadrupolar nuclei, acquisition of 1d nmr spectra, 1d nmr spectra, quadrupolar nuclei, quadrupolar nucleus, 1d mas spectrum, quadrupolar non, nuclei, nucleus

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Abstract

Purpose
To acquire a 1D MAS spectrum on a quadrupolar nucleus, optimized for sensitivity with non-quantitative intensities.

Scope
The acquisition of 1D NMR spectra of quadrupolar nuclei with non-quantitative intensities allows for greater sensitivity per unit time than acquisition with quantitative intensities. This SOP should be followed when quantitative intensities are not required. If quantitative intensities are required do not use this protocol.

Materials

Definitions:
1. CT: Central Transition

Appendix:
Using the tip angles above will result in the maximum sensitivity for resonances within the CT-selective limit with non-zero intensity for the non-selective limit. These angles should ensure that all resonances are observed, but they will not be homogeneously excited and relative intensities will not be reliable.

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.

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

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

Procedure

1 Load pulse sequence **zg**.

2 Set the relaxation time **d1** to 1.3 × T_1, where T_1 is the largest value previously measured.

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

4 Using a previously optimized 90° non-selective pulse length corresponding to pulse power **plw1**, calculate a pulse length corresponding to the nuclear spin number of the observed nucleus:

	Nuclear Spin Number	Tip Angle	Factor
	3/2	45°	0.50
	5/2	30°	0.33
	7/2	22.5°	0.25
	9/2	18°	0.20

Note

Using the tip angles above will result in the maximum sensitivity for resonances within the CT-selective limit with non-zero intensity for the non-selective limit. These angles should ensure that all resonances are observed, but they will not be homogeneously excited and relative intensities will not be reliable.

5 Set the pulse length **p1** to the value calculated in Step 4.

6 Acquire the spectrum until the signal-to-noise ratio is satisfactory for all resonances.

Protocol references

Vega, A. J. Quadrupolar Nuclei in Solids. John Wiley & Sons, Ltd 2010-03. https://doi.org/10.1002/9780470034590.emrstm0431.pub2.

Protocol

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