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Quadrupolar Quantitative Bloch Decay V.1

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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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Abstract

Purpose
To acquire a 1D Bloch decay spectrum under magic angle spinning with quantitative intensities.

Scope
The acquisition of 1D NMR spectra of quadrupolar nuclei with quantitative intensities requires particular care due to the complex dependence of nutation on C_Q. This SOP should be followed when quantitative intensities are required. If quantitative intensities are not required, the sensitivity of the Bloch decay experiment can be improved by making different choices in pulse length and **d1**.

Guidelines

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. C_Q: Quadrupolar coupling constant

Appendix:
Using the tip angles above will result in a maximum integrated intensity deviation of 5% between a perfectly non-selective excitation and a perfectly selective excitation. As perfectly selective excitation is unlikely using these powers, the real deviation will likely be lower. This limit of 5% only applies to the homogeneity of the excitation and not other possible sources of error, e.g., background subtraction.

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 **zg**.

2 Set the relaxation time **d1** to 5 × 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	18°	0.20
	5/2	11°	0.12
	7/2	8°	0.09
	9/2	6°	0.66

Note

These tip angles ensure a worst-case quantification error of 5% between a site with a C_Q of 0 MHz and a site with a very large C_Q. In most cases the error will be much less.

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

6 Set the acquisition time **aq** to a sufficiently long value such that the FID is not truncated.

7 Ensure that the central transition approaches the infinite-spinning limit by optimizing the MAS rate.

7.1 Obtain a preliminary spectrum using a stable MAS rate.

7.2 Increase the MAS rate by a safe increment.

Note

If the MAS rate cannot be safely increased, consider changing to a probe with a smaller rotor diameter. Quantification cannot be assured if increasing the MAS rate increases the line width.

7.3 Compare the two spectra. If the linewidth has not changed, go to step #8

7.4 If the spectrum at higher MAS rate has a higher linewidth, go to step #7.2 .

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

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

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

11 Apply the satellite transition intensity correction described in Massiot *et al.*

Protocol references

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

Massiot, D.; Bessada, C.; Coutures, J. P.; Taulelle, F. A Quantitative Study of ^{27}Al MAS NMR in Crystalline YAG. Journal of Magnetic Resonance (1969)1990, 90 (2), 231–242. https://doi.org/10.1016/0022-2364(90)90130-2.

Protocol

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Saturation Recovery for Quadrupolar Nuclei

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Quadrupole Non-Selective Pulse Width Optimization

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