NMR

Automation for Low-Gamma Nuclei

The New CPMAS Low-Gamma iProbe

Innovation with Integrity

Expand Your Solid-State NMR

Bruker's new Low-Gamma iProbe defines a new benchmark for sensitivity and efficiency in solid-state NMR. Engineered for nuclei with low gyromagnetic ratios - including ^{15}N, ^{14}N, and others - this probe enables researchers to achieve exceptional performance in demanding applications.

The Low-Gamma iProbes feature a standard tuning range from ^{109}Ag to ^{15}N and are available with maximum MAS spinning speeds of 15 kHz (4 mm) or 24 kHz (3.2 mm), providing flexibility for a wide range of experimental needs.

These new probes are available for 400 to 600 MHz Systems and can be equipped with a 3.2 mm or 4 mm MAS stator. The 4 mm MAS probe is ideal for routine spectroscopy on sensitivity-limited nuclei, while the 3.2 mm MAS probe supports spinning rates up to 24 kHz, offering greater flexibility and improved spectral resolution.

All of our new CPMAS iProbes support automated magic-angle adjustment using the ^{79}Br resonance of KBr, which is the preferred method for routine magic angle calibration. For low-gamma iProbes with a tuning range to ^{15}N, the magic angle can be adjusted using the ^{14}N resonance of ammonium dihydrogen phosphate (ADP). This is a highly sensitive method for precise magic-angle adjustment.

Key Benefits

Our new Low-Gamma solid-state NMR probe, built on the iProbe platform, delivers broad application versatility while fully leveraging the advantages of our automated, ready-to-use probe architecture. It is fully compatible with MAS Shuttle insert-and-eject functionality and offers

- **Superior Sensitivity for Low Gamma Nuclei:** Achieve high signal to noise ratios and significantly reduce experimental time.
- **High Power Handling:** Enables short pulses for broadband excitation.
- **ATMA – Automatic Tuning & Matching:** Supports automatic retuning at each frequency step during static wideline spectroscopy and allows unattended switching between nuclei for extended experiments.
- **MAS Shuttle Automation:** Facilitates remote, automated sample exchange.
- **Wide Temperature Range:** Ensures reliable variable temperature operation up to 200 °C through optimized gas flow control and enhanced thermal stability

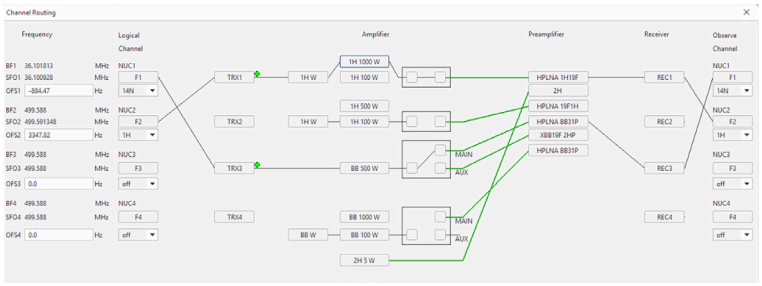

Figure 1: Routing for the experiment.

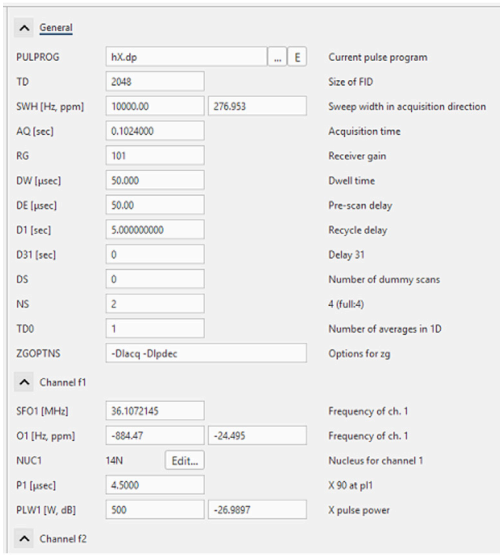

Figure 2: All pulse parameters can be loaded with a click through the PROSOL table.

The experiment is setup by loading the parameter set ^{15}N DPMAS or if available ^{14}N DPMAS. When loading the ^{15}N DPMAS parameter set, change the nucleus to ^{14}N. The parameter set uses the pulse program hX.dp.

With the flag -Dlpdec, decoupling is defined as a low power decoupling which sets the ^{1}H-spin decoupling field to 1/4 of the MAS rate, 3 kHz in the current case. The decoupling program is swftppm or swftppm_12 which is a frequency swept TPPM variant that delivers the best results for low power decoupling.

When the magic angle is far off, we see a splitting of the ^{14}N resonance as shown in figure 4.

Adjusting the stator orientation leads to a narrowing of the split resonance.

When the orientation seems close, a single resonance can be observed as seen in figure 6, it is recommended to shim the probe. Shimming can lead to resonance splitting re-emerging, which requires further adjustment of the stator orientation. Such and iterative approach guarantees a well setup low-gamma iProbe. A line width of approximately 7.5 to 8 Hz should be achieved.

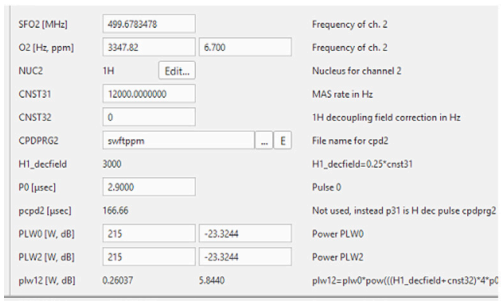

Figure 3: Parameter setup for the experiment.

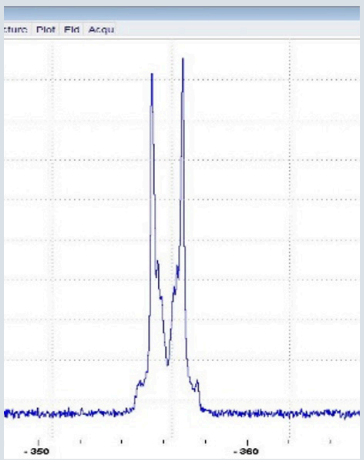

Figure 4: ^{14}N resonance when the stator is not well aligned with the magic angle.

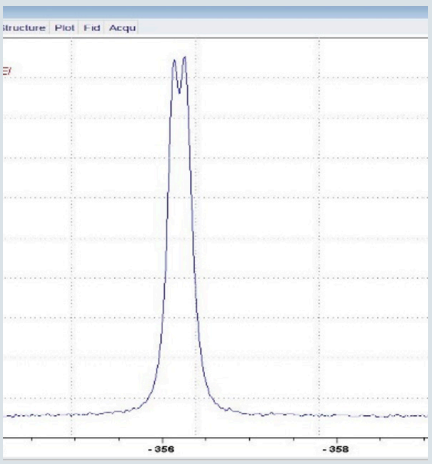

Figure 5: The resonance splitting is reduced as the stator orientation approaches the magic angle.

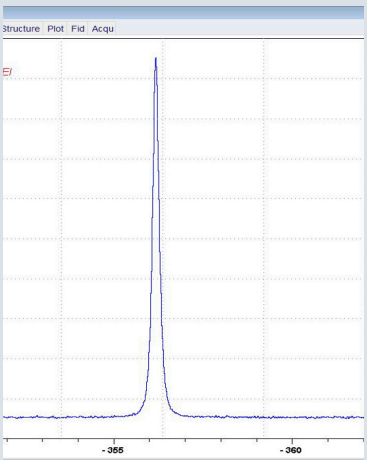

Figure 6: One resonance without splitting.

When the resonance is optimally narrow and a single resonance, both, the shim file as well as the stator's orientation need to be stored.

A distinguishing feature of our iProbe series is the integrated ATMA system, which is especially valuable in the low-gamma regime because it enables fully automated execution of ultra-wideline experiments. These experiments are used to measure very large CSA tensors, commonly for nuclei such as ^{14}N or ^{35}Cl in pharmaceutical formulations.[1] To capture the complete spectrum, the excitation frequency is stepped across the broad powder pattern to ensure uniform excitation. The individual stepwise acquisitions are then concatenated to reconstruct the full wideline spectrum.

The pulse sequence is shown in figure 7. It consists of an adiabatic excitation pulse followed by a Carr-Purcell-Meiboom-Gill (CPMG) echo train. The total length of the echo train is given by the parameter N. A sample with a long enough T2 times, the echo train will boost the sensitivity significantly as compared to a standard 90° excitation.

To use the sequence all we need is a calibrated 90° pulse in the range of 28 kHz B_1-Field. This corresponds to ~ 9 us pulse length. To test the setup of the WCPMG sequence an ideal test sample is "glycine" or "glycine HCl".

Table 1 shows the typical experimental details for a parameter set using the CPMG sequence.

After acquisition of the first spectrum, you need to move the carrier, O_1, by approximately 250 kHz, which is 10 * the spike separation, determined by the separation of the echoes in several steps to acquire the full static pattern. After each offset change, you need to retune the probe. Using our iProbes you can use ATMA for automatic tuning and matching before running the next spectrum, which makes the whole process easy and fully automated.

Figure 8 shows the result after the glycine ^{14}N wideline pattern was acquired stepwise (multicolor overlay) and also after summing up all contributions to the final wideline spectrum. After summing up all contributions using topspin "sola" a simulation can be calculated to extract and confirm tensor information.

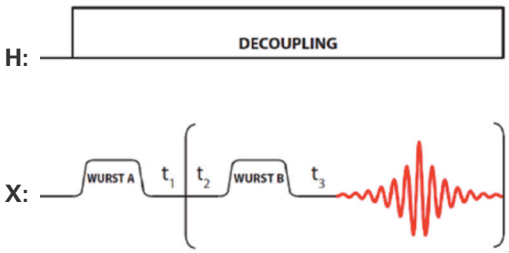

Figure 7: Echo Train sequence, incl ^1H decoupling. The number of echoes in the train is defined by "N".

Sample	Glycine
^1H decoupling	Approx. 50 kHz
Sweep width	2 MHz
D_6	100 us
D_3	Depends on probe, use to adjust spike separation to an even number
L_{22}	Try 50 echoes
P_{11}	100 us, 1000 kHz sweep
Sp_1	Power level at approx. 28 kHz
N_s	1024
D_1	1 s
O_1	-250 kHz (or 100 * spike separation)

Table 1: Start parameter for the setup.

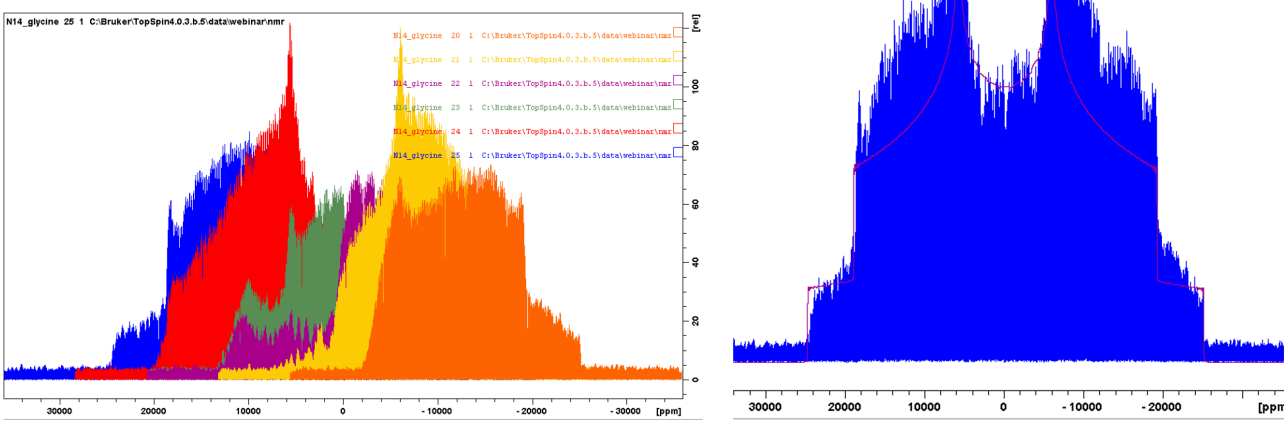

Figure 8: Result after detecting the full pattern.

[1] Hirsch, D.A., Rossini, A.J., Emsley, L., & Schurko, R.W. ^{35}Cl dynamic nuclear polarization solid-state NMR of active pharmaceutical ingredients. Phys. Chem. Chem. Phys., 2016,18, 25893-25904. https://doi.org/10.1039/C6CP04353D

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CPMAS Probes
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