

NMR SOFTWARE

Using KBr as Test Sample for Optimizing and Setting the Magic Angle Position for a CPMAS Probe

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Basic setup, CPMAS, routine work, MAS, TopSpin, magic angle, iProbe

Innovation with Integrity

To perform solid-state NMR experiments successfully, a well-configured system is crucial. One of the most critical parameters of an Magic Angle Spinning (MAS) probe is the magic angle setting, which is essential for achieving the highest possible resolution. This Application Note provides information on which TopSpin setup can be used to control and readjust the magic angle. As a prerequisite, it is assumed that:

- The prosol table is correctly filled. OR
- The user has knowledge about the correct pulse length's and power pairs for the used MAS probe.

To perform these experiments, it is necessary to have a KBr-filled rotor with a diameter that corresponds to your probe. The spinning speed for the angle setting should be 5 kHz for most probes, while for smaller diameters (1.3 mm, 0.7 mm, 0.4 mm), the speed should be 40 kHz.

Regularly checking the magic angle position is essential for performing high-sensitivity experiments using MAS. If the position is not correctly adjusted, it needs to be recalibrated using "KBr" as a reference compound. The magic angle is considered optimal if the line width of the main peak in the rotating KBr spectrum has a line width similar to that of a spinning sideband, i.e. within +/-10% of the full width at half maximum (FWHM) of a selected spinning sideband. Traditionally, this involves comparing the center band to a sideband at a distance of 20 or 40 kHz. The TopSpin software includes a parameter set called "SETUPMAS," which can be accessed using the command "rpar SETUPMAS all," as illustrated in Figure 1.

| rpar SETUPMAS all | | |
|---------------------|-------------------|--------------------|
| Spectrometer Status | Amplifier Control | Acquisition Status |
| On ✓ | | no acquisition |

Figure 1: Set up the experiment by reading the predefined parameter set

Once “getprosol” is called, the dataset is prepared, and a first reference spectrum can be acquired using “zg,” as shown in Figure 2

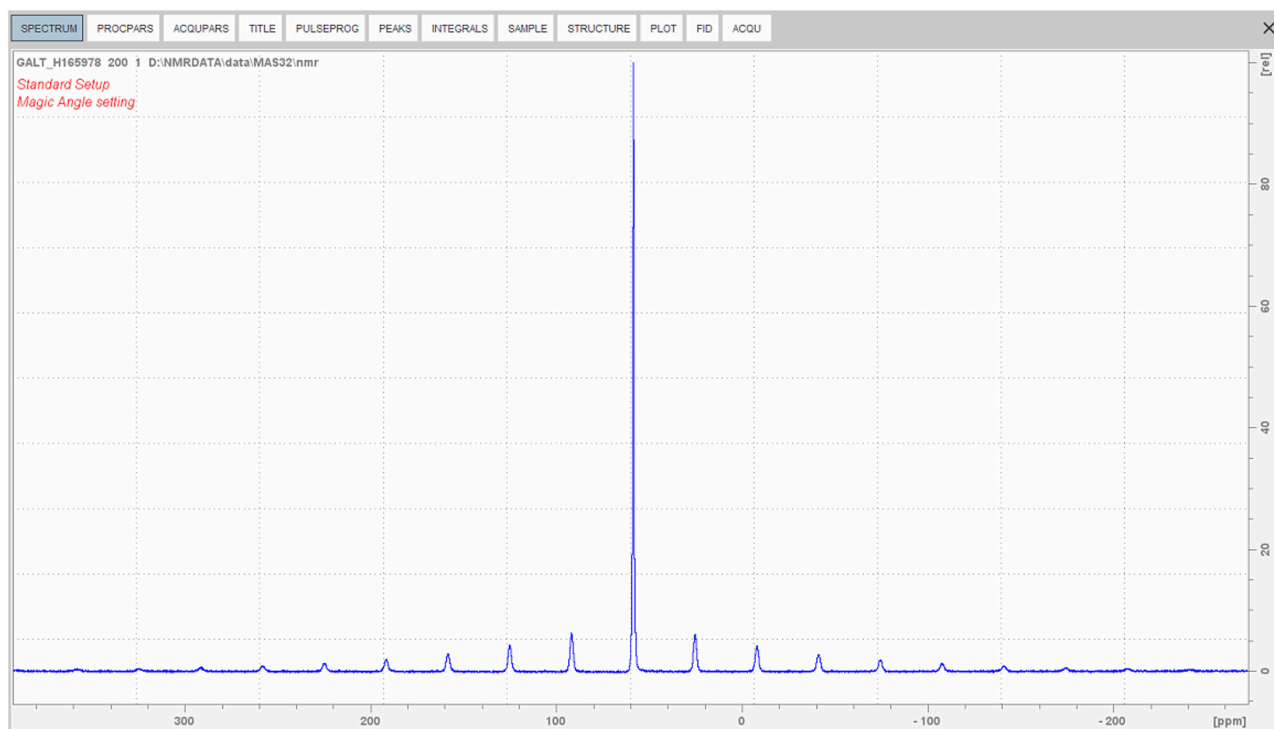


Figure 2: KBr spectrum as detected using the predefined parameter set

In this case, since the above defined $\pm 10\%$ criteria is not fulfilled, it is evident that the probe has an incorrect magic angle position and needs to be readjusted. To confirm this, you can use the AU program “Angle_Check,” while the rotor is still running at your previously selected MAS speed, as shown in Figure 3. The program evaluates the KBr spectrum and writes a stability statement into the title of your experiment.

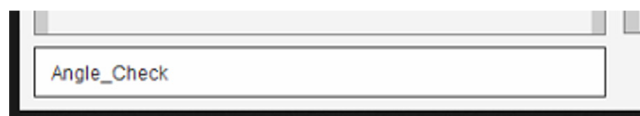


Figure 3: AU program for checking the $\pm 10\%$ criteria for an optimal angle setting

There are two options available to recalibrate the angle of your MAS probe, depending on the type of probe you are using:

1. For iProbe-based probes, an automated routine is available.
2. For non-ATMA probes, a manual procedure for readjustment is necessary.

In the case of iProbe-based probes, the automated angle adjustment feature can be found in the corresponding tab of the MASDISP software (as shown in Figure 4). Once the routine is completed, the optimum arrangement is saved in the probe’s permanent memory by pressing the “Save” button, which shows up after the routine is finished.

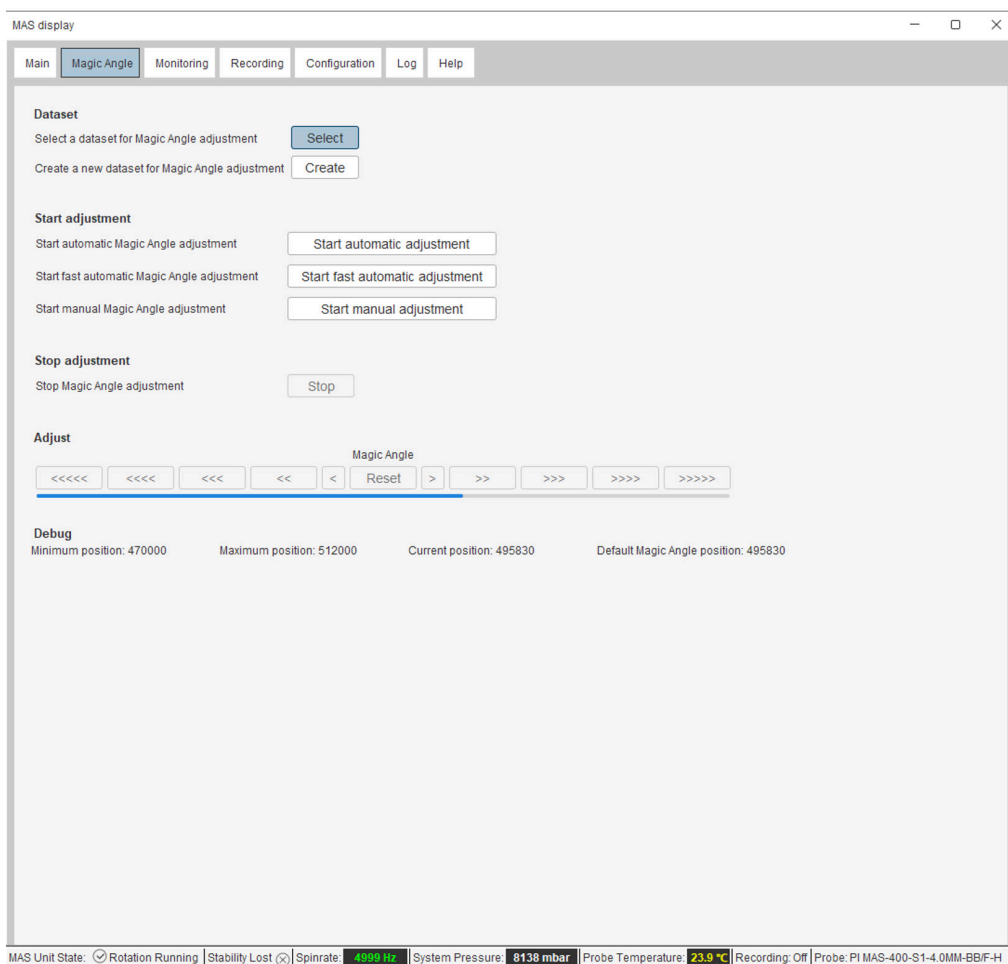


Figure 4: In the “MAS display,” the tab “Magic Angle” is shown, if an iProbe is connected. The button “start automatic adjustment” calls the routine to readjust the angle position based on the rotating reference sample.

For non-ATMA based probes, a fully automated routine is not available, and the angle needs to be readjusted manually by turning the magic angle knob at the probe bottom. To do so, load the parameter set as described above, fill in the corresponding pulse lengths/power pairs, and type “gs” in the open dataset to start the live acquisition. Next, the length of the ^{79}Br FID or the intensity of the spinning sideband pattern of the KBr spectrum needs to be optimized. To adjust the magic angle, it is necessary to observe the live acquisition on the PC screen while turning the magic angle adjustment rod at the probe base. In recent probe designs, this rod has a black end marked with an “A” for Angle. The magic angle is considered adjusted properly when the FID persists for the longest time, or when the real-time fourier transformation provides the highest spinning sideband intensities.

After the adjustment is complete and you are satisfied with the results, type “stop” and acquire a new reference spectrum using “zg” to store the final result in the dataset. In both manual and automated cases, another call of the “Angle_Check” program will confirm whether a good setting has been reached or if readjustment is necessary.

The criterion for a well-set angle position is the FWHM comparison between the center peak and the spinning sideband at a distance of 20 or 40 kHz. If these two peaks have the same linewidth within an error margin of $\pm 10\%$, the KBr-based angle setting is considered accurate enough to perform ^{13}C -based CP experiments. However, for experiments with quadrupolar nuclei, it is necessary to use setup samples with higher accuracy compared to KBr, such as Rubidium nitrate or Rubidium sulfate.

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