NMR

Automated Shimming for MAS Probes

Innovation with Integrity

Introduction

Here we present a shimming routine for MAS probes that is implemented in TopSpin version 4.3. Traditional shimming routines for liquid samples rely on gradient shimming and thus cannot be used with most solid-state NMR probes. The presented automatic MAS shimming routine uses an advanced algorithm, shimming on the FID of an adamantane sample which is Bruker's reference sample for CPMAS probe shimming. The routine relies on a ^{13}C decoupling acquisition at high MAS rates. The routine works for Bruker's routine solid-state NMR probes for WB and SB magnets. The routine ideally complements the automation features of Bruker's CPMAS iProbes.

An additional benefit of the shimming routine especially for new solid-state NMR customers is that it references the B_0-Zeeman field of the magnet to the 38.48 ppm resonance of adamantane, relative to TMS.

Bruker's MAS iProbes have been designed with special consideration of the requirements for solid state automation, remote operation and ease of use. Features like automatic tuning and matching (ATMA), the automatic adjustment of the magic angle and automatic sample changes are all supported by Bruker's MAS iProbes. In this application note, we describe another important feature, namely the implementation of an automatic shimming algorithm. In combination with Bruker's renowned IconNMR software, fully automated solid-state NMR experiments are now possible, thereby increasing the productivity of solid-state NMR analysis, and the ease of using solid-state NMR for everyone.

TopSpin Commands

The commands available for the MAS shimming routine are the following:

Command	Description	Parameters
MASshim	Starts the MAS shimming process	full quick --FWHM --angle
haltshim	Stops the shimming process at any time	

The parameters for MASshim fulfill the following functions:

Parameter	Mandatory or Optional	Typical value	Description	Example
full	Mandatory		Uses the full set of room temperature shims that are relevant for MAS. This option should be used if the probe is not shimmed and needs a new initial shim.	MASshim full
quick	Mandatory		Speeds up the shimming process by using a reduced set of room temperature shims, with a minimum impact on shim quality. This option should be used if the probe has been shimmed before already and just needs a quick iteration.	MASshim quick
--FWHM	Optional	7.0	Stops the shimming process once a certain linewidth (FWHM) has been achieved, e.g. 7 Hz.	MASshim full --FWHM 7.0
--angle	Optional	0; 90; 40	This parameter describes the angle between the stator in the MAS probe and the Y-axis of the room temperature shim system.	MASshim full --angle 40
--skip_TuneMatch	Optional		The shimming routine runs ATMA to ensure the best results of the routine. In case the probe is already fully setup, this options will skip it.	MASshim full --skip_TuneMatch
--s	Optional	adamantane	This option defines the sample used for shimming. At the moment only admantane can be selected and is also defined as default if this option is not used.	MASshim full --s adaman-tane

One can use more than one option with the MASshim command, e.g.

> **MASshim full - -FWHM 7.0**

Please note that in case "FWHM" is not defined, the shimming routine will run continuously until the command haltshim is called; thus if "angle" is not defined, a dialog box will open and the value for the "angle" parameter must be entered manually. When the MAS shimming routine is used within IconNMR, it is thus strongly recommended to use the optional "FWHM" and "angle" parameters to ensure that a clear stop criterium is defined and to guarantee an uninterrupted automation workflow.

Orientation of MAS iProbes

The parameter "angle" describes the angle of the stator's rotor axis in the MAS probe in the room temperature shim system's X-Y coordinates. It is not necessary to measure the angle accurately. In practical applications, it is typically enough to work with one of three values: 0, 90 or 40 degrees.

0: Y gradient is the relevant component
90: X gradient is the most relevant gradient
40: the stator is aligned at app. 40° to the Y-axis

Values for the "angle" parameter for some typical orientations of an iProbe are shown in Figure 1. Note that for probes which are not based on the iProbe platform, the orientation of the stator inside the probe might be different. For such probes, Figure 1 might not be valid. Also note that it is only necessary to enter values between 0 and 90 degrees, and the same value can be used for the "angle" parameter even if the probe is mounted 180° rotated from what is shown in Figure 1. All Bruker WB probes have the angle 90.

-- angle 0

-- angle 90

-- angle 90

-- angle 40

Figure 1: Recommended values for the "angle" parameter for some typical orientations of MAS iProbes.

Application Example

The following example illustrates the power of the MASshim algorithm. Figure 2 shows the ^{13}C spectrum of an adamante sample with all room temperature shims set to zero. The lineshape is clearly not within specification.
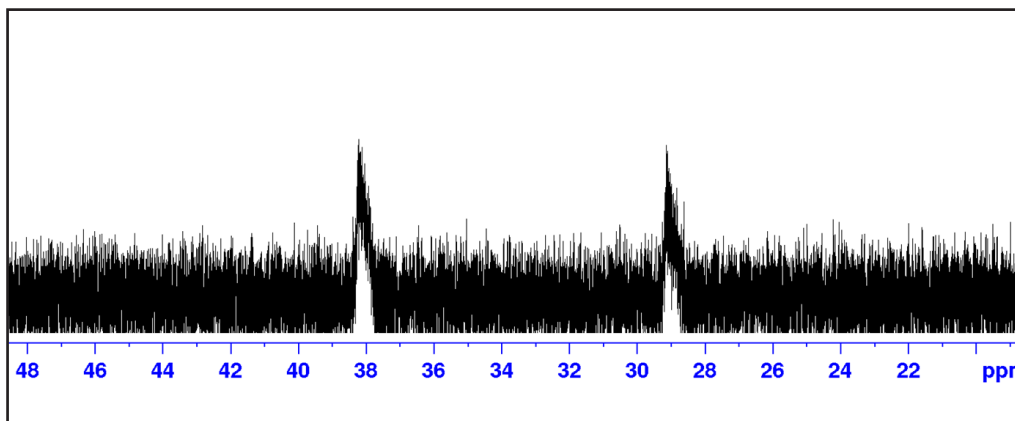

Figure 2: With all room temperature shims set to zero, the lineshape (^{13}C adamantane) is not within specification

If the orientation of the MAS probe with respect to the room temperature shim system is known, the shimming process can be easily started from the TopSpin command line. For example, if the probe is mounted in the X-direction, the following command can be used:

> **MASshim full --FWHM 7.0 --angle 0**

This command prompts TopSpin to shim the probe until a linewidth of 7 Hz FWHM is reached and to only use shim gradients which are oriented along the Z and the X-axis. The shimming routine will display a message once the probe is shimmed successfully. Figure 3 shows the lineshape spectrum which was achieved by the shimming algorithm in the application example presented here.

The shimming procedure shown in Figures 2 and 3 took less than 10 minutes. It was performed with a 600 MHz 3.2 mm HX CPMAS iProbe and at a MAS rotation rate of 15 kHz, using low power decoupling frequency swept TPPM decoupling at 3.75 kHz, ¼ of the MAS rotation frequency.
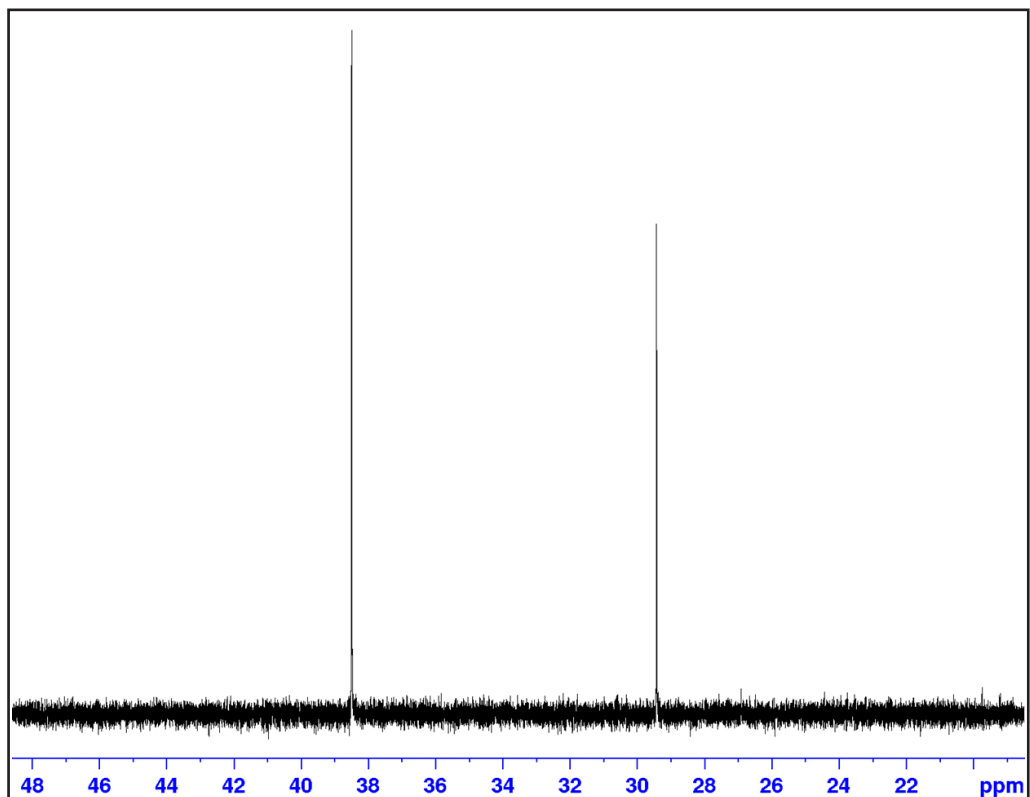
More stringent linewidth targets can of course always be set, e.g. 2 Hz instead of 7 Hz:

> **MASshim full --FWHM 2.0 --angle 0**

It is possible to stop the shimming routine before the target linewidth has been reached by typing

> **haltshim**

This will halt the routine and set the best shim values that were found during the shimming process. Remember, if MASshim is called without the FWHM option, the routine will run continuously until it is stopped with the haltshim command.

Procedure if the Angle Parameter is Unknown

If the orientation of the stator inside the MAS probe with respect to the room temperature is not known, the following procedure can be executed:

1. Create an hpdec dataset (rpar SETUPSHIM) with adamantane running at MAS > 10 kHz
2. Load rf-power parameters with "getprosol"
3. Start the continuous acquisition mode by typing gs in the command line
4. Type "BSMSDISP" in the TopSpin command line
5. Set the X shim to 10,000 units and observe the shim reaction in the live display, then reset X to 0
6. Set the Y shim to 10,000 units and observe the reaction in the live display.

The parameter that should be used with the "angle" option can be determined from the result of the above procedure:

1. Only the X shim caused a reaction: angle is 90
2. Only the Y shim caused a reaction: angle is 0
3. Both the X and the Y shim caused a reaction: angle is between 0-90 and can be set to 40 in the initial step.

As an example, if both the X and the Y shim caused a reaction (i.e. case 3), the shimroutine can be called with the following command:

> **MASshim full - -FWHM 7.0 - -angle 40**

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