

Automatic Shimming for Solid-State NMR

Abstract

Bruker’s MAS iProbes have been designed with special consideration of the requirements that arise from 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. Here, 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 possible, making solid-state NMR more efficient and easier to conduct.

A shimming routine for MAS probes is implemented in TopSpin starting from version 4.3. Traditional shimming routines for liquid samples rely on gradient and can thus not be used in most solid-state applications. Bruker’s automatic MAS shimming routine uses an adamantane sample, which is Bruker’s reference sample for CPMAS probe shimming, and relies on a ¹³C decoupling acquisition.

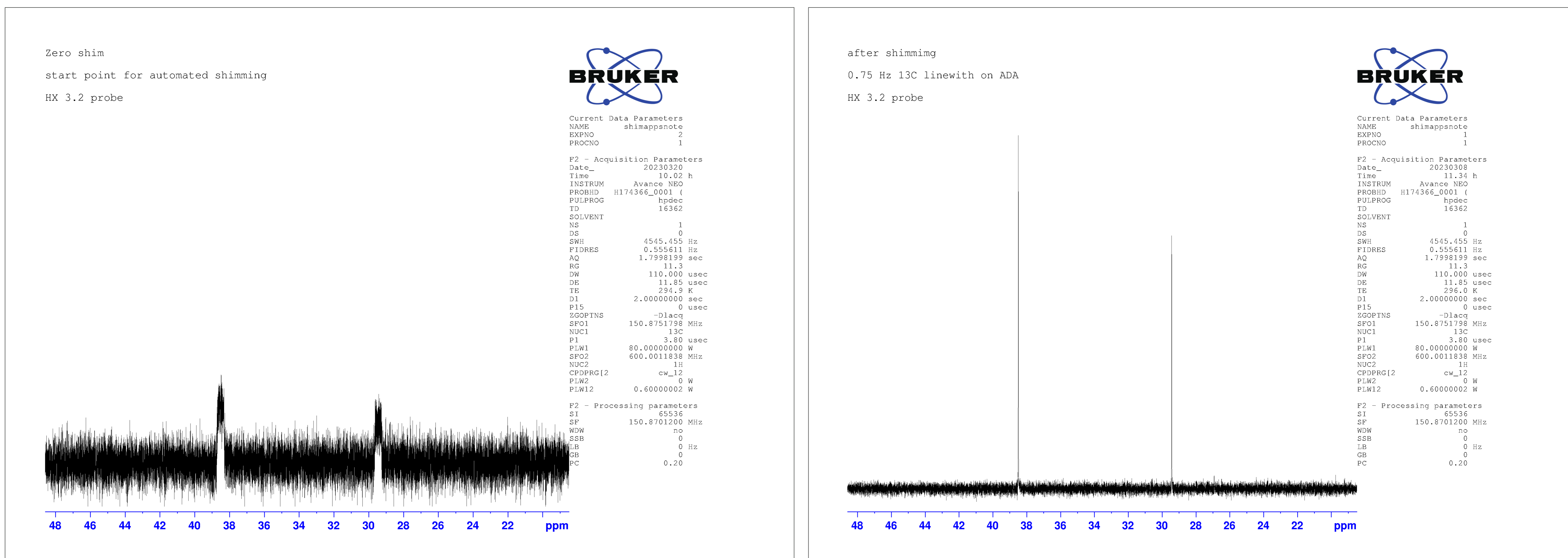


Figure 1: ¹³C lineshape spectrum of an adamantane sample with all room temperature shims set to zero (left) and after the successful completion of the MASshim routine (right). The shimming procedure took less than 10 minutes. It was performed with a 600 MHz 3.2 mm HX CPMAS iProbe and at a MAS rotation rate of 15 kHz, using low power decoupling at ¼ of the rotation speed.

The shimming process can be easily started from the TopSpin command line. For instance, with the probe being mounted as shown in Figure 2 on the left, the following command shim the probe until a linewidth of 7 Hz FWHM is reached:

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MASshim full --FWHM 7.0 --angle 0
```

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

Table 1: TopSpin commands for the MAS shimming routine

Parameter	Mandatory or Optional	Typical value	Description	Example
full	Mandatory		Uses the full set of room temperature shims that are relevant for MAS	MASshim full
quick			Speeds up the shimming process by using a reduced set of room temperature shims, with a minimum impact on shim quality.	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 room temperature shim system	MASshim full --angle 40

Table 2: Parameters for the MASshim command



Figure 2: The orientation of the MAS probe to the RT shim system is a parameter of MASshim (left: angle 0, middle: angle 90, right: angle 45).

Conclusion

- Starting from TopSpin version 4.3, a new set of commands will be available for automatic shimming in solid-state NMR.
- With these new commands, full automation is now possible for solid state NMR experiments, including automatic tuning and matching, automatic adjustment of the magic angle, and automatic sample changes with the MAS shutte.
- The MASshim algorithm can be conveniently used in combination with IconNMR.
- The results achieved with the automatic algorithm are typically better than the results achieved with manual shimming. This also translates into significant time savings for the spectroscopist.