
SOLID-STATE NMR

# Manually Shimming a CPMAS NMR Probe Using Adamantane

## Setting up your CPMAS probe for routine work

Innovation with Integrity

### Introduction

Precise shimming of a solid-state Nuclear Magnetic Resonance (NMR) probe is essential for obtaining high-quality spectra with optimal resolution and signal-to-noise ratio. This application note outlines the manual adjustment of shim currents using adamantane as a reference substance, without employing gradients. Adamantane is an ideal standard due to its chemical and structural properties: it exhibits sharp, well-defined resonances and is chemically inert. In this application note, we describe how to optimize the probe shim manually; note that there is also a fully automatic shimming routine for iProbes, which is described in a separate document.

To manually optimize the shim currents for a solid-state NMR probe using adamantane as a standard, the pulse sequence "HPDEC" is used. Here, $^{13}C$ is excited with a 90° pulse, and during acquisition, low power proton decoupling is applied to achieve the best resolution. This application note describes the procedure based on an Avance NEO system and TopSpin >4.0, but most of the procedures will also work on older instruments.

### As a prerequisite for this procedure, the following is needed:

- A MAS NMR probe compatible with the operating frequency of the NMR spectrometer
- Adamantane powder homogeneously packed into a zirconia rotor of the desired diameter (e.g., 4 mm), rotating with at least 7 kHz, ideally 15 kHz
- The probe must be properly tuned and matched on both the $^{1}H$ and $^{13}C$ channel
- A prefilled prosol table for at least $^{1}H$ and $^{13}C$ pulses. Otherwise, the correct calculation of the various power levels cannot be guaranteed
- Basic knowledge about the general TopSpin functionality (e.g. display modes, command line etc.)
- The probe has a well adjusted magic angle

After the rotor packing and probe mounting, load the correct parameter set within TopSpin. To do so, use the command:

```
rpar SETUPSHIM
```

Confirm to load all selected settings. Afterwards, please assign a new pulse program to the dataset by typing

```
pulprog hpdec
```

followed by the command

```
getprosol
```

Now the dataset is fully prepared and has power levels and pulse lengths set correctly for long-term low power decoupling.

In the example case described here, starting from a configuration where all shim currents are set to zero, the spectrum in Figure 1 can be seen. The probe is not very well shimmed with a resolution of approximately 30 Hz FWHM. The peak width can be measured by typing the following command into the command line of TopSpin:

```
peakw
```

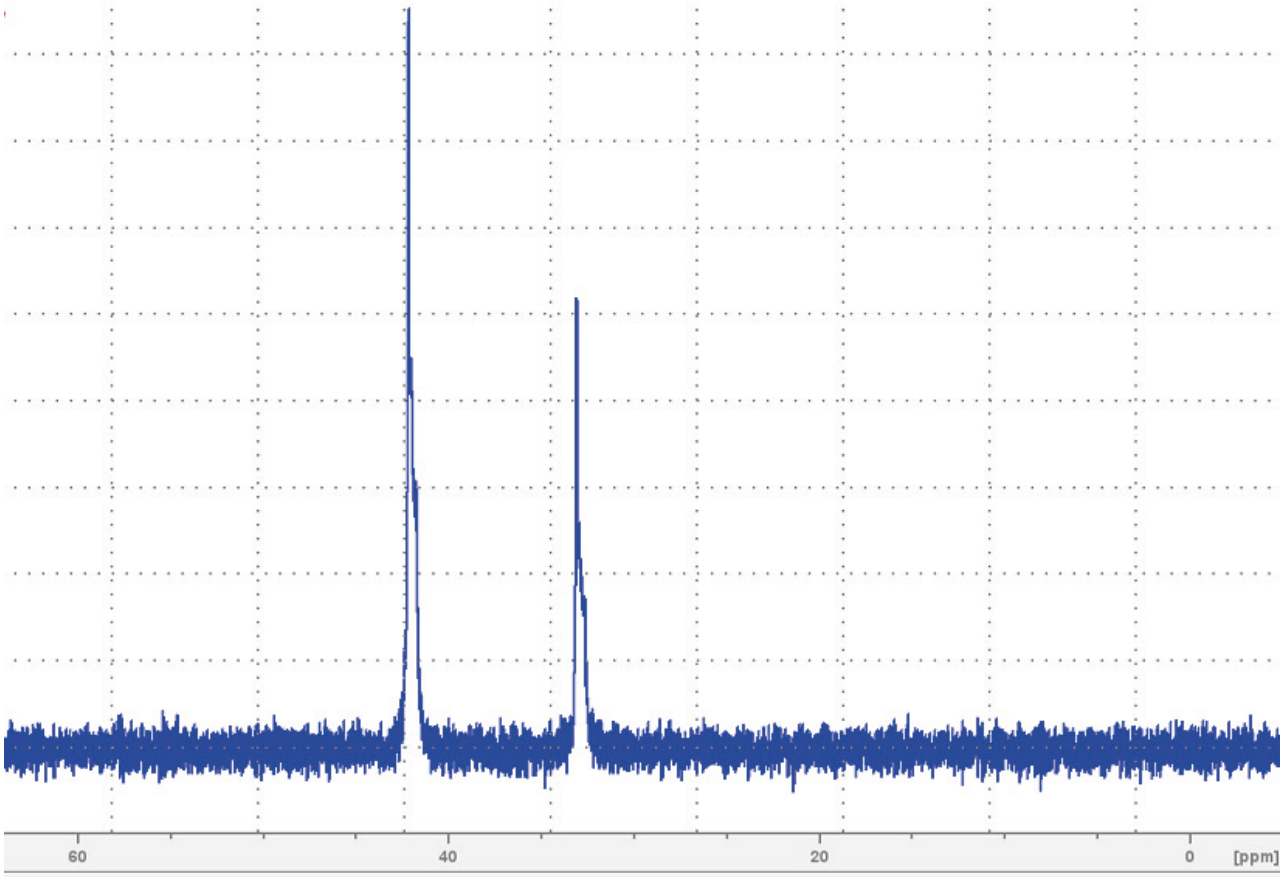


**Figure 1:** Adamantane spectrum with zero shims; FWHM approx. 30 Hz

Now that it has been confirmed that the probe needs to be shimmed better, the following procedure can be applied:

```
gs
```

This activates the continuous scan mode.

Afterwards, please switch on real-time Fourier transformation by pushing the realFT button in the graphical user interface to see the adamantane $^{13}C$ signal.

Now by using the command

```
bsmsdisp
```

the shim current adjustment panel can be opened from TopSpin. In this panel, please select the shim tab as indicated in figure 2.

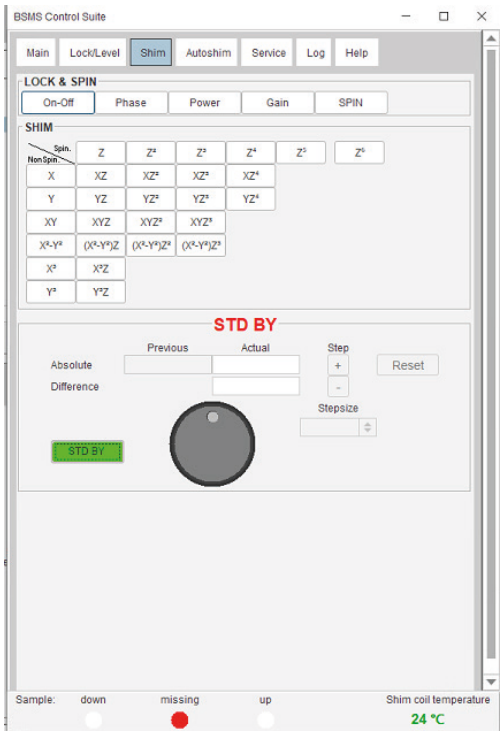


**Figure 2:** Bsmsdisp with the shim tab selected

Manual shimming is usually done iteratively, following the four steps outlined below:

1. **Adjust the Z shim:**
   - Start by optimizing the Z shim by systematically increasing or decreasing the units while monitoring the signal intensity and linewidth of the adamantane resonance.
   - Goal: Maximize signal intensity and minimize the full width at half maximum (FWHM).

2. **Optimize the transverse shims (X, Y):**
   - Adjust the X and Y shims to improve homogeneity in the lateral dimensions.
   - Select the correct X- or Y-shim or alternate between X and Y adjustments to achieve the optimal peak resolution.

3. **Iterative optimization:**
   - Repeat the process (Z, X, Y, etc.) until no further improvements in signal intensity or linewidth are observed.

4. **Cross-term shims (XY, XZ, YZ):**
   - If available, fine-tune these parameters to eliminate residual inhomogeneities.

In the following, we illustrate the key steps of the procedure above in more detail. Select the "Z" shim, set the step size to app. "500" and zoom to one of the adamantane peaks to see how the shim setting is influencing the lineshape (see figure 3).

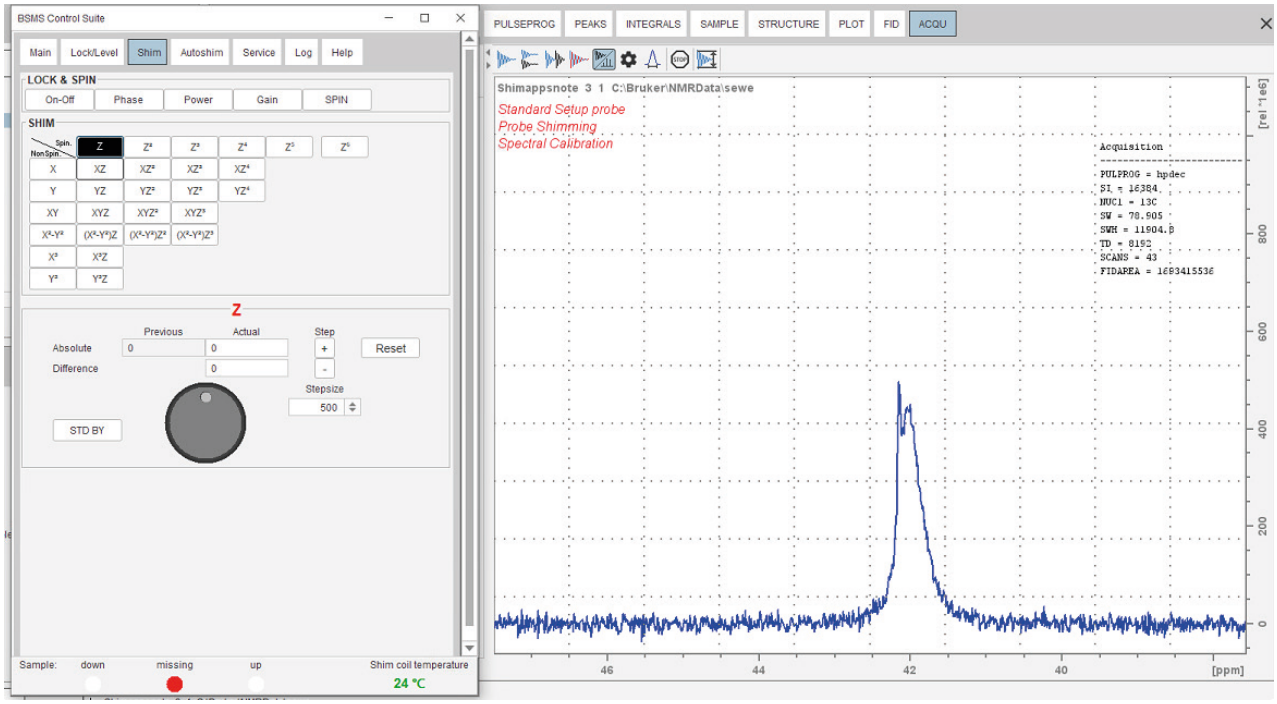


**Figure 3:** Z shim selected, with step size "500" and the adamantane signal correctly centered in the display

Now start to maximize the signal intensity by modifying the "Z" shim value. In the example (figure 4), the setting had to be adjusted to -44000 units.

Please note: If the signal raises over the edge of the display, use the "/2" intensity button to bring it back into into full view before continuing to adjust the "Z" shim.

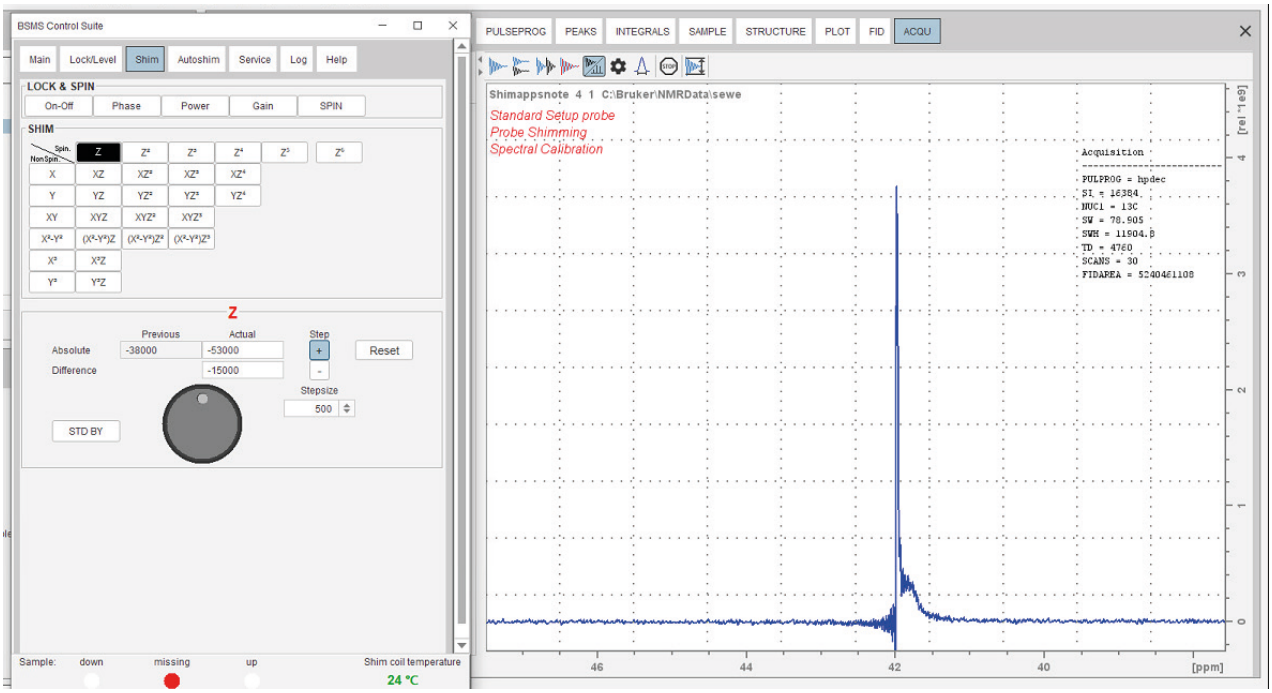


**Figure 4:** Z shim adjusted to maximum signal intensity

With this adjustment, the peak width was brought down to approximately 4 Hz, but the peak is not very symmetric at the foot. To compensate this, select the X or Y shims for further adjustments.

Please note: For the transverse shims, a much larger step size of 5000 should be used, since their influence on the homogeneity is much less pronounced.

Follow the same procedure as for the Z shim to optimize the peak width further to to optimize the peak width further and to maximize intensity.

With most probes, good shimming according to the procedure described here will lead to an adamantane linewidth of < 10 Hz and a symmetric lineshape.

To summarize, manual shim optimization without gradients requires patience and a systematic approach. Adamantane is an ideal shimming material as its sharp resonances provide quick and precise feedback. When performed correctly, this procedure yields high-resolution solid-state NMR spectra.

After the lineshape has been set correctly, the following command can be used to store the archieved shim to the hard disk.

```
wsh NAMEHERE
```



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