Troubleshooting Shimming with Cryoprobes

Introduction

These troubleshooting instructions guide the user through a procedure with the purpose of helping him or her to achieve good shimming of a cryoprobe (CRP). This document is intended for Bruker engineers confronted with shimming problems during an installation or a service of a CRP system. It should help taking the decision whether a CRP can be shimmed or not based on the results of up-to-date shimming methods.

This shimming procedure is divided into three sections:

A. Checking the preconditions

B. Shimming the probe using topshim standard methods

C. Shimming the probe using topshim's diagnostic tools

These instructions show a general procedure that helps solving shimming issues. The procedure focuses on the standard reference samples where shimming issues have been encountered most frequently (water suppression, lineshape and sensitivity 1H), but it can be expanded to other samples as well.

Most parts of these instructions can be helpful with room temperature (RT) probes, too; although some CRP-specific items in section A need to be adapted to RT probes correspondingly or can be skipped. Sections B and C describe general shim strategies that are not explicitly restricted to CRPs.

Prerequisites for using the present instructions:

- Spectrometer hardware according to the topshim user's manual chapter 2.1. Essentially, that's an AV spectrometer or higher, with gradient system and RCB functionality.
- Spectrometer software according to the topshim user's manual chapter 2.2. Essentially that's topspin 2.0 for sections A and B of these instructions, but the methods used in section C require at least topspin 2.1.

A. Preconditions

This section describes the preconditions that must be all met before actually proceeding to the shimming procedure outlined in sections B and C, respectively. If any of the preconditions of section A is not fulfilled, fix the related issues first, as any shimming effort might be in vain otherwise.

1. Before installing the cryoprobe (CRP), do shim an RT probe. Benefits include:
   - You obtain an up-to-date start shimfile. This is needed in section B, step 1.
   - The general working condition of the console can be checked.
   - A possibly quenched cryoshim of the magnet can be identified.
   - Possible issues of this kind can be fixed before working with the CRP.
2. If dirt or liquid must be removed from the sample cavity of a 5mm CRP, follow the procedure given in the User's Manual (Z31551). Preventive cleaning is not recommended; clean only in case of problems.
3. Verify that the CRP is mounted as far up as it will go.
   Note that in case of a CRP of S1 length there will remain a gap of 10mm between the CRP's body and the shim stack's lower surface. There will remain a larger gap if a CRP of S6 length is mounted into a shim stack of S5 length.
4. Verify that the shimstack is installed at the correct height.
   In rare cases it has been observed that the shimstack has been pushed upwards e.g. when mounting a CRP, or pulled down when handling RT probes.

5. The CRP must be cooled down. The monitoring software must report that the CRP is cold; on the Cryo Cooling Unit, the "Cold" button must be lit.
6. VT setup:
   - Adjust the VT gasflow rate to the recommended value given on the limitations sheet that comes with the CRP.
   - Set the sample target temperature: If no temperature correction is effective, then set it to 298K, if a temperature correction is effective, use a setpoint of 295K. These values are intended for Bruker standard reference samples. Customer's samples might require a different target temperature.
   - Check that the VT gas heater is switched on and actually keeps the measured temperature stable at the target temperature.
   - For probes that support spinning: Make sure that spinning is stable, and the sample is not lifted or floating unsteadily. This can be monitored by checking the wobble curve on 1H. If the gasflow is too high, the wobble curve will be very unstable, as compared to the regular instability observed during sample spinning.
7. Use the correct NMR sample.
   Radiation damping is due to a high sample concentration and leads to linewidth broadening. This effect becomes prominent in 1H spectra.
   - 1H lineshape sample: Use 0.3% CHCl3 in acetone-d6 for all CRP types and all field strengths. Exception: The 1.7mm CRP needs 1% CHCl3 in acetone-d6.
   - 1H sensitivity sample: Assessing shimming with the 1H sensitivity sample is possible. Use 0.1%EB in CDCl3 for all CRP types and all field strengths.
8. Carefully clean all NMR samples before inserting them into the magnet and probe. This is absolutely essential for 1.7mm CRPs.
9. For 1.7mm CRPs only:
   - 1.7mm CRPs must not be moved or rotated in the magnetic field while cold. If this has happened, do a warmup and cooldown sequence before continuing with shimming.
   - It is absolutely essential to carefully clean each NMR sample tube before inserting them into the magnet and the probe.
   - 1.7mm NMR samples can "hide" portions of the liquid under their cap. Shake the liquid down, or use a manually operated centrifuge to get the liquid down. The filling height of a 1.7mm NMR sample must be greater than 20mm. An insufficient NMR-effective filling height can erroneously make the CRP appear to be non-shimable.
10. The NMR sample tube must not be scratched in the lower area with the liquid.
    In rare cases shimming problems have been reported that were probably due to a scratched sample tube.
11. Check that the NMR sample depth is adjusted to the CRP's standard value, or that the sample is centred with respect to the RF coils, respectively.
12. Make sure that the CRP is properly tuned and matched with the sample inserted.
13. Adjust the lock channel: Use a non-saturating lock power, and adjust all lock parameters in order to obtain a stable lock signal. Do *loopadj* to get a useful set of *lgain*, *lfilter* and *ltime*.

    Remark: With the solvent Acetone it has been observed occasionally that the lock was unstable after a *loopadj*. In a lineshape spectrum this can manifest as pronounced noise floor band up to ±25 Hz around the CHCl3 peak.
    Suggestion: Try the *lock.10*, *lock.11* or *lock.12* macros that you find on the CRP-CD that was handed out during the CRP training course.
14. VT gas leak: If the lock shows an erratic behaviour despite of good lock channel adjustments:
    - Check for possible VT gas leaks along the full VT gas supply from the source all the way to the CRP. Any leak along this chain can cause an unstable VT gas flow to the CRP, and / or cause mixing with ambient air, thereby altering the magnetic properties of the VT gas in an erratic way. Fix all leaks.

- Check that the recommended gasflow rate is actually flowing through the CRP, and is not blocked or diverted. Occasionally, it has been observed that the ATM motor unit has squeezed the VT gas tube, thereby significantly reducing the real gas flow rate through the CRP.

15. Convection inside the NMR sample: When the sample is not spinning and when the lock is unstable and / or topshim reports "not enough valid points" or "too many points lost during fit", then convection may be present. Do either one of the following:
    - Lower the NMR sample temperature.
    - Use a convection compensated topshim pulse program. See Knowledge Base entry 8699 for the details.
    - From TS 2.1 PL5 and from TS 3.0 topshim offers the option *convcomp*. Start all topshim commands from topspin's command line with the option *convcomp* added in order to compensate for convection issues. Do not replace the topshim pulse program in this case.
16. The NMR experiment that you use to assess shimming must be properly set up according to the applicable procedures. Your experiment must provide for a sufficient signal-to-noise ratio of at least 1000:1.
    If the signal-to-noise ratio is less than 1000:1 the calculation attempt of a 0.11% linewidth becomes meaningless.
17. In order to make topshim work, valid and safe 90deg high power pulses must be entered in the *edprosol* table for both 1H and 2H nuclei routed from the logical channel F1 to their respective amplifier.
18. Hint: In order to better see what *topshim* is doing, open first *topshim gui* from topspin's command line, and consult the Results tab to see the actions and results of topshim.

B. Standard shimming method

This section describes a standard shimming procedure similar to the one used in the final test of the NMR probe. As no shims are manually set, the resulting shim set is determined by software only. This procedure requires that all preconditions listed in section A are met.

1. *rsh* of an up-to-date start shimfile.
2. Insert the water suppression sample, tune the CRP and lock.
3. Set all high-order shims to 0 (zero).
    - We recommend setting to zero all onaxis shims from 5^{th} order on (i.e. z5 and higher).
    - We recommend setting to zero all offaxis shims from 4^{th} order on (i.e. xz3, yz3, xyz2, (x2-y2)z2 and higher, plus x3z and y3z).
4. Do topshim in the following sequence:
    - *topshim 3d ordmax=4,3*
    - *topshim 3d z6*
    - *topshim cal*
    - *topshim z6*
    - Save the resulting shimfile. This is your reference shimfile from which you'll start all subsequent work in this section B.

    Remark to steps 2 – 4:
    If your CRP is not specified for water suppression, use the ASTM sample instead. The *topshim 3d* commands in step 4 must then be expanded by the additional parameter *astm*.
5. Insert now the sample for which you would like to improve the shimming. Tune the CRP, and lock.
6. Do *topshim* (without further options).
    Do your NMR experiment that requires the good shimming.
    Assess the result. See below for further hints about evaluating the result.

7. If the result is not yet good enough: Do either
   - *topshim lshump*, or
   - *topshim ss*.

   Repeat your NMR experiment and assess the result.
8. If the result is not yet good enough: Do either
   - *topshim ordmax=6* (for CRPs that can do with a BOSS 2 shim system), or
   - *topshim ordmax=8* (for CRPs that benefit from a BOSS 3 shim system).

   Repeat your NMR experiment and assess the result.
9. If the result is not yet good enough: Do either
   - *topshim ordmax=6* [*lshump* or *ss*] (for CRPs that can do with a BOSS 2 shim system), or
   - *topshim ordmax=8* [*lshump* or *ss*] (for CRPs that benefit from a BOSS 3 shim system).

   The brackets [*lshump* or *ss*] mean that you must use either one of the options given therein. Repeat your NMR experiment and assess the result.
10. For future reference, save the shimfile with which you have obtained the best result with your NMR experiment in steps 6 through 9.
11. When you're finished and satisfied with the shimming result:
    Repeat the lock channel adjustment described in section A, item 13. Save the result for the solvent used in the *edlock* table.

Suggestions for assessing the result of your shimming effort with Bruker standard NMR reference samples:

- 1H lineshape sample (CHCl3 in Acetone-d6):
  lb = 0. Determine the hump and the resolution with *humpcal* and *hwcal*, respectively. The FID should show an envelope with a smooth exponential decay. Set o1p slightly off resonance to see this (e.g. to 7.9ppm). Two features deserve your special attention:
  1. Any nodes in the first ~3s of the FID indicate a multiple peak spectrum – this can often be improved by slightly adjusting the low order z-shims up to z3.
  2. The very beginning of the FID should ideally appear slightly rounded off – this indicates a narrow hump. If, on the contrary, the beginning of the FID shows a pronounced spike then the hump region will be broad, and probably not meet specs. This problem can be addressed with high order onaxis shims. You can use higher ORDMAX limits and topshim options as stated above to correct for this problem.
- 1H sensitivity sample (0.1% EB In CDCl3):
  To assess the shimming with the 0.1%EB sample, set the parameter *si* to 128k (instead of the std 16k), then transform the data using *fp* (i.e. without line broadening).
  The mid peak of the triplet near 1.25ppm should show a very good resolution. Measure the full width at half maximum (FWHM) using *hwcal*. The better the achieved resolution the higher the resulting sino. Typical FWHM results may be around 0.50Hz or less, while some CRPs can achieve results of the order of 0.30Hz.
- 1H water suppression sample (2mM Sucrose in 10% D2O + 90% H2O):
  There are two major aims when shimming the water suppression sample in view of optimum results in the presat water suppression experiment:
  1. The residual water line should be narrow. This allows for a better water suppression, and hence for increasing the maximum receiver gain *rg* to optimise the sino without saturating the receiver.
  2. The splitting of the anomeric proton at 5.4ppm should be optimised, as this will boost the sino value without adversely affecting the water suppression.

Recommendations:

1. The residual water line is dominated by the high order onaxis shims, mostly z5 and z6 for a CRP that needs BOSS 2, or z7 and z8 if the CRP requires a BOSS 3 shim system. You can use higher ORDMAX limits and topshim options as stated above to address this issue.
2. The splitting will not be optimum after topshim. This is due to the temperature gradient that is inherent to all CRPs. Start the *gs* mode, observe the immediately transformed spectrum, zoom on the DSS signal around 0ppm. Slowly change the z shim, unit by unit, thereby maximising the amplitude of the DSS signal. This will optimise the splitting of the anomeric proton as well. Direct observation of the anomeric proton, however, is not recommended due to phasing issues that are often observed in the *gs* mode close to the residual water line. – Typically, a change of about +15 units of z shim will do the job with 5mm CRPs. This number depends on the probe diameter, the diameter and shape of the NMR tube, and on the gas flow rate. The improvement that can be achieved with this correction of the z shim is usually more pronounced with higher field strengths.

   Remark: If the magnet has been charged with opposite polarity with respect to the Bruker convention, then the required change of the z shim will have the opposite sign as well.

C. Shimming with topshim's diagnostic tools

Sometimes, the standard shimming methods described in section B do not yield sufficiently good results. In this case, the methods presented here show how to analyse the results that topshim has achieved, and take manual corrective actions to achieve a more homogeneous B_0 field distribution. Basically, the 1d field maps acquired by topshim will be interpreted, and corrections to high order onaxis shims will be applied manually to obtain a better result.

C.1. Theory of shimming with topshim's diagnostic tools

For the method presented here, two basic topshim commands are used:

topshim map:

This acquires a 1d field map of the current B_0 field distribution. No shimming is done. No further topshim options are necessary.

topshim plot:

This command executes the usual 1d shimming iterations, and on top of that it records the B_0 field distribution with each shimming iteration. Any further topshim option can be added as usual.

Topshim creates datasets in a user directory named "topshimData", see Fig. 1. Each topshim command overwrites previously existing data in topshimData. Therefore, you are responsible to save any data that you would like to keep.

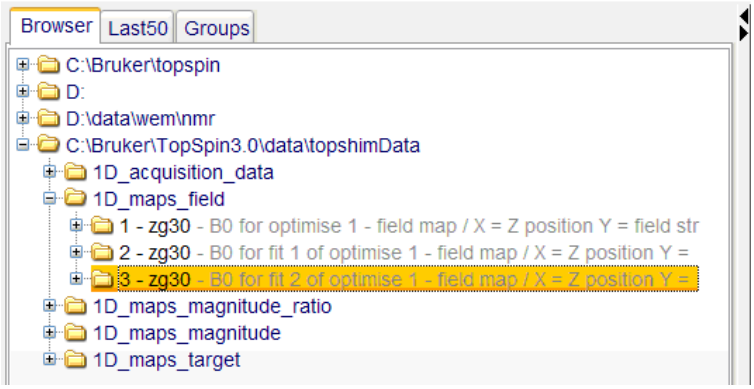

Fig. 1. Locate the topshimData directory in topspin.

The user topshimData reveals several experiments, depending on what topshim has done in the last run. After executing *topshim* with the option *map* or *plot*, there will be an experiment named "1D_maps_field". Here we find the B_0 field deviation as a function of the position z along the sample,

with z=0 referring to the magnetic centre. The B_0 field deviation is calibrated in mHz (milli-Hertz, cf. title), therefore always use absolute scaling in topspin when interpreting such data.

Topshim map displays the current B_0 field graph in expno 1, while *topshim plot* may create several expnos, depending on the number of iterations that topshim has decided to do. The highest expno shows the current B_0 field graph (see Fig. 1).

Fig. 2 shows the B_0 field graph with a quite well shimmed central part, but pronounced side lobes. The consequence of such a profile on a lineshape spectrum will be a fairly decent half width (determined with *hwcal*), because the central part, from where most of the signal amplitude is collected, shows small deviations from the ideal B_0 field distribution. The side lobes, however, will contribute to NMR signal amplitude shifted with respect to the central resonance frequency. This will promote a broad hump as will be revealed with *humpcal*.

Consider now the case of a presat water suppression experiment. A field profile that resembles the one shown in Fig. 2 will not only lead to a broadened residual water line. The upper and lower portions of the NMR sample that exhibit a shifted resonance frequency with respect to the central portion cannot be well suppressed with the presat sequence, and will therefore produce superelevated contributions to the left and / or right wings of the residual water line in the presat spectrum, making the residual waterline appear wider and taller. As a consequence, the receiver gain must be reduced in order to avoid saturation of the receiver / ADC chain, which in turn impairs the achievable signal to noise ratio.

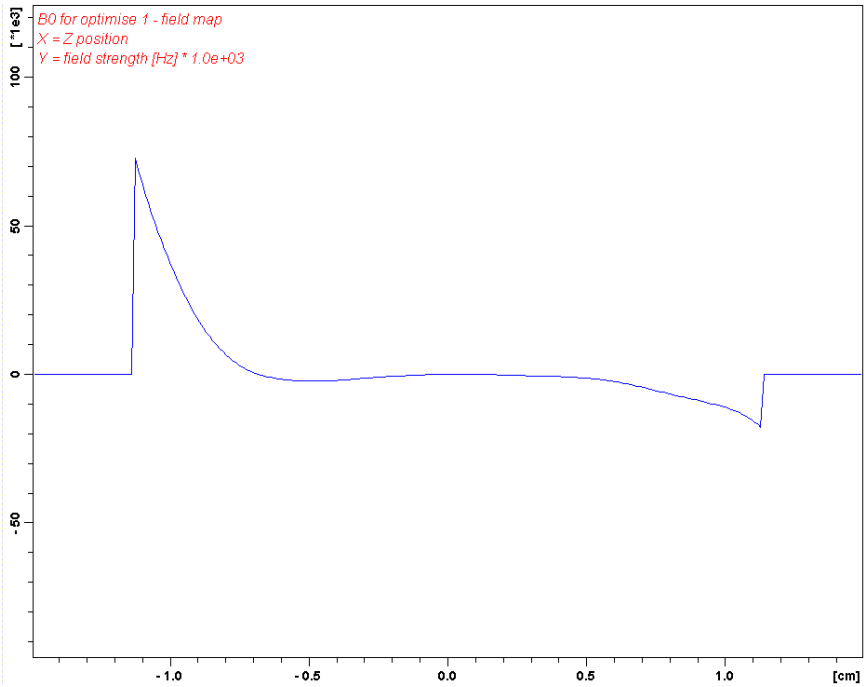

Fig. 2. A B_0 field graph with lobes to the end of the active NMR sample length.

It is the goal of shimming to obtain a small B_0 field deviation not only in the centre, but over the full active length of the NMR sample. Drawn to the same scale, the resulting B_0 field deviation after shimming might look like in Fig. 3. With respect to the examples given above, the lineshape spectrum will benefit from a narrower hump region. The water suppression spectrum will exhibit a narrower residual water line, and, because the receiver gain can be increased, the sino will also improve.

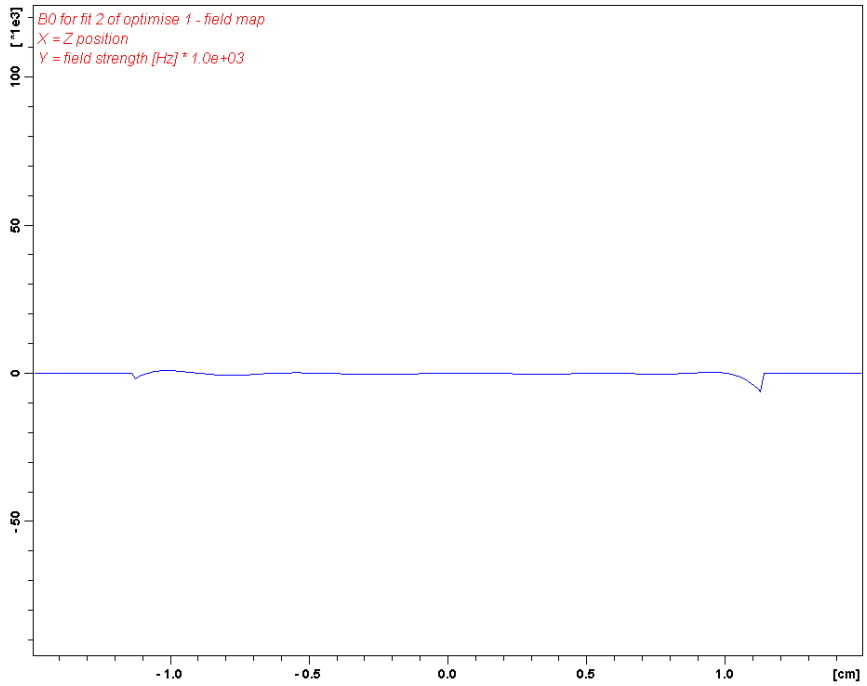

Fig. 3. A B_0 field graph with a good shim over the full active NMR sample length.

The ends of the active NMR sample volume are dominated by the two highest order onaxis shims. In order to understand how these shims can be used to correct the side lobes from Fig. 2 and make them "flat" as shown in Fig. 3, it is essential to understand what pattern these shims create in a B_0 field graph.

In case of a BOSS 2 shim system, the highest order onaxis shims are z5 and z6; with a BOSS 3 shim system these are z7 and z8. The principles and methods explained here, however, remain the same.

Fig. 4 shows the B_0 field graph of Fig. 3, but z5 has been changed by +1000 units.

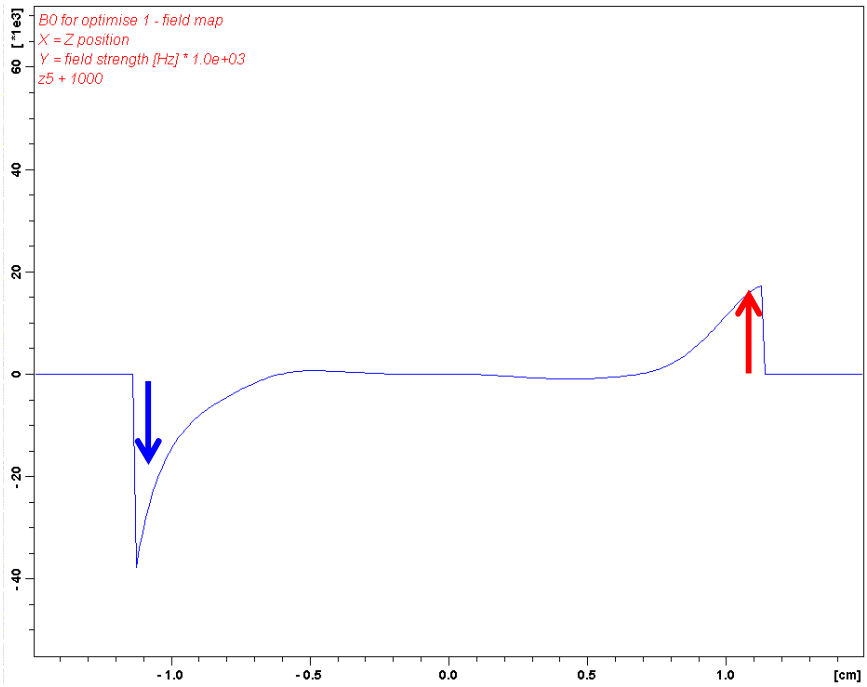

Fig. 4. B_0 field graph of Fig. 2, but with z5 increased by +1000 units.

All odd order onaxis shims (z1, z3, z5 and z7 if available) show this behaviour: The more any odd order shim is increased the higher the resulting lobe on the right hand side. On the left hand side, another lobe will develop to the *downside* by about the same amount (arrows in Fig. 4). When an odd order onaxis shim is reduced instead, the resulting lobes will also reverse their sign: The right hand side lobe goes down while the left hand side lobe goes up. The higher the order of the shim, the

further away from the centre the change will take effect; the z1 shim creates a linear tilt from end to end, whereas z7 will work only on the very end to either side. *Odd* order shims alter the B_0 field graph in an *antisymmetric* way.

Fig. 5 shows the B_0 field graph of Fig. 3, but now z6 has been changed by +7000 units.

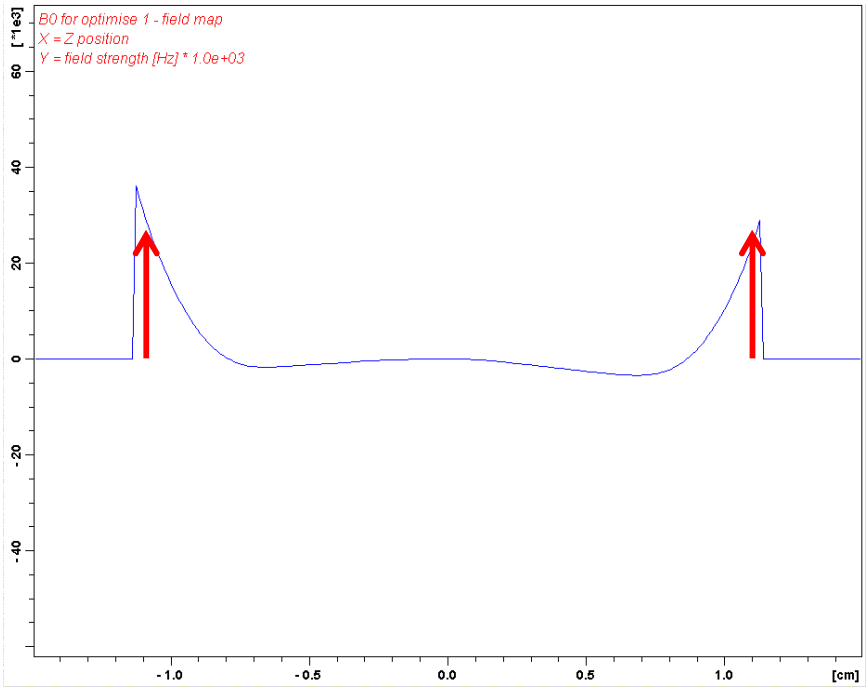

Fig. 5. B_0 field graph of Fig. 2, but with z6 increased by +7000 units.

All even order onaxis shims (z2, z4, z6 and z8 if available) show this behaviour: The more any even order shim is increased the higher the resulting lobe on the right hand side. On the left hand side, another lobe will develop to the *upside* by about the same amount (arrows in Fig. 5). When an even order onaxis shim is reduced instead, the resulting lobes will also reverse their sign: Both lobes will go down. The higher the order of the shim, the further away from the centre the change will take effect; the z2 shim creates a parabola, whereas z8 will work only on the very end to either side. *Even* order shims alter the B_0 field graph in a *symmetric* way.

Remark: If the magnet has been charged with the polarity opposite to the Bruker convention, then the B_0 field will respond also with the opposite sign to changes of a shim. E.g. if z6 is increased, both lobes go down instead of up.

With the knowledge of how the high order shims affect the B_0 field graph, a shimming strategy can be derived easily:

- Acquire a *topshim map* or use the latest B_0 field graph from the last *topshim plot*.
- Use the highest even order shim to get a completely antisymmetric B_0 field graph, and then correct it with the highest odd order shim.
- Use the highest odd order shim to get a completely symmetric B_0 field graph, and then correct it with the highest even order shim.

Typically recommended changes of these shims are about 500 units for z5, 1000 units for z6, 3000 units for z7 or z8 to see an effect.

- After a shim change, we recommend doing a *topshim plot* (not *map*), as this rectifies the lower order shims up to z5. High order shims are usually not "pure"; e.g. z7 carries often a considerable amount of z1. If you're working with z5 manually, then you should limit topshim to z4: *topshim plot ordmax=4*.

As visible from Fig. 4 and 5, shim changes are usually not perfectly antisymmetric or symmetric. Therefore, it is often needed to apply odd and even order shim changes in an iterative manner until the B_0 field graph levels out in the desired way, without any big side lobes remaining.

Remark: Which pair of shims z5 / z6 or z7 / z8 is the suitable one to work with?

- Probes that work with a BOSS 2 shim system will usually respond mostly to z5 / z6. Do the manual optimisation with these two shims. When done, and if you have a BOSS 3 shim system available, you can do another round using z7 and z8. This often gives you even better results.
- Probes that need a BOSS 3 shim system: Usually, work with z7 and z8. Some probes need also a manual touch up of z6 (to cure symmetric features that are not located at the very end of the active NMR sample area). Adjust z6 in steps of about 1000 units, do the usual optimisation with z7 and z8; the *topshim plot* commands in between will take care of all lower order shims from z1 to z5.

C.2. Shimming procedure using topshim's diagnostic tools

It is assumed that

- the preconditions from section A are all fulfilled, and
- the automated shimming method in section B has been done, but has not yet produced the desired results in view of shimming your current NMR sample.

Go through the following procedure that uses topshim's diagnostic tools and manual corrections:

1. *rsh* of the best shimfile obtained with the procedure in section B.
2. Insert the sample for which you would like to improve shimming. Tune the CRP and lock.
3. Execute *topshim plot*, or
   topshim plot tunea (recommended when the sample is measured nonspinning).
4. In topshimData, open the experiment 1D_maps_field, and display the highest expno. This shows the current B_0 field deviation.
5. Analyse these data according to the instructions given in section C.1.
   - If not yet finished, then manually apply the shim changes determined. Repeat from step 3 until the criteria for good shimming are met.
   - If the criteria for good shimming are met, continue with step 6.
   - If the criteria for good shimming are not met and the shimming iterations do not yield any improvement any more, continue with step 8.
6. Run your NMR experiment. The result obtained now is optimised with respect to shimming. Save the final shimfile for future reference.
7. Repeat the lock channel adjustment described in section A, item 13. Save the result for the solvent used in the *edlock* table.
   Shimming of the CRP is completed at this point.
8. (Continued from step 5) This step applies only if shimming has not been successful and the shimming iterations did not yield any improvement any more.
   - Insert the water suppression sample, tune the CRP and lock.
   - Do *topshim 3d plot*.

   Remark: If your CRP is not specified for water suppression, use the ASTM sample instead, tune the CRP and lock, and then do a *topshim 3d plot astm*.
   - Immediately compress the complete topshimData folder into one single zip file.
   - Upload this zip file to the ftp server of Bruker Switzerland: ftp.bruker.ch (anonymous login) /NMR/incoming/.
   - Send an email to cryoprobe.service@bruker-biospin.ch: Give the exact probe ID (both part and serial numbers), add the problem description, give the filename of your uploaded topshimData folder, and give your contact details.

- Bruker Switzerland will analyse your data, and give you advice how to proceed with this probehead.

END OF DOCUMENT