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Practical aspects of shimming a high resolution magic angle spinning probe

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Abstract

High resolution magic angle spinning (HRMAS) has become an extremely versatile tool to study heterogeneous systems. HRMAS relies on magic angle spinning of the sample to average out to zero magnetic susceptibility differences in the sample and to obtain resonance linewidths approaching those of liquid state NMR. Shimming such samples therefore becomes an important issue. By analyzing the different sources of magnetic field perturbations present in a sample under MAS conditions, we propose a simple protocol to obtain optimum shim settings in HRMAS. In the case of aqueous samples, we show that the lock level cannot be used as a reliable indicator of the quality of the shims at high spinning speeds. This effect is explained by the presence of temperature gradients imparted by the sample rotation.

Keywords: HRMAS; Shimming; Magnetic susceptibility differences; Temperature gradients

1. Introduction

High resolution magic angle spinning (HRMAS) has become a ubiquitous tool to study heterogeneous systems endowed with sufficient dynamics by NMR [1–9]. The domain of application of HRMAS is extremely diverse and includes the analysis of molecules issued from solid phase synthesis [10,11], molecules in membrane environment [12,13], swollen polymers, mesoporous materials [14], cells [15–18], and even biopsies [19–25]. While the dynamics of some of the molecules present in such system is comparable to molecules in solution [26,27], the inherent sample heterogeneity precludes the obtaining of high resolution NMR spectra under static conditions. The main role of magic angle spinning (MAS) is therefore to average out internal magnetic susceptibility differences present in such samples [28,29]. Concomitantly, MAS also averages out to zero residual dipolar interactions that might be present in the sample provided that their intensity is smaller than the spinning speed [30].

When using HRMAS to study highly dynamic systems such as metabolites in a biopsy or true liquids, proper shimming is of the uttermost importance to analyze the fine structure present in these spectra. The requirements become similar to what is expected of a regular high resolution liquid state probe. In this article, we analyze the different factors that affect the quality of shimming in a high resolution MAS probe. Using some theoretical considerations that are confirmed by experimental observations, we propose a simple protocol to obtain shims of optimum quality on different samples in different solvents.

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2. Hardware considerations

The following description pertains mainly to Bruker HRMAS probes, however, many of the general features presented below also apply to other manufacturer's probes. HRMAS experiments are usually performed on standard liquid-state NMR spectrometers equipped with a dedicated probe that allows to spin the sample at the magic angle. The radio-frequency circuits of these probes are designed to withstand only the power classically available on liquid-state NMR spectrometers. Therefore, they cannot be used to perform typical solid-state type experiments like cross-polarization. They are usually fitted with a gradient coil generating a linear field gradient along the magic angle axis that allows to perform coherence selection experiments [1]. A HRMAS probe is designed to minimize the different sources of magnetic field perturbations in close proximity to the sample in order to obtain resonance linewidths similar to those obtained on a standard liquid-state high resolution probe. The sample is contained in a 4 mm ZrO_2 rotor that is able to withstand the strong centrifugal forces created by high spinning speeds. To optimize the sensitivity of the probe, the rotor is fitted with Teflon inserts to define a 50 μl volume that matches exactly the detection volume of the solenoidal coil. This experimental set-up allows to detect all the material contained in the rotor which means that the whole sample contributes to the detected signal, a situation which is in contrast to classical high resolution probes where typically only half of the sample is detected. Despite the presence of several sample/rotor interfaces in the coil detection volume, the NMR lineshape is not affected by these magnetic perturbations provided the sample is rotated at the magic angle (see detailed explanations below).

3. Shimming the residual field distortions in a HRMAS probe

When placed in a homogeneous B_0 magnetic field, even a well designed HRMAS probe will disturb the magnetic field lines. Like in high resolution liquid state NMR, the resultant field distortions have to be corrected using the shim system of the spectrometer. The shimming procedure differs however by the fact that the correction gradients have to be applied along the MAS axis [31]. A linear combination of the usual shims found on a high resolution NMR instrument must therefore be used. For a HRMAS probe with a stator positioned in the (x,z) plane of the laboratory frame, inhomogeneities of the B_0 field along the rotation axis can be corrected up to the third order by using the zonal shims $\boldsymbol{B}_{Z^1}^{\mathrm{MAS}}$, $\boldsymbol{B}_{Z^2}^{\mathrm{MAS}}$ and $\boldsymbol{B}_{Z^3}^{\mathrm{MAS}}$. The radial inhomogeneities do not require any corrections since they are averaged out by the rotation. This averaging process is similar to what is found in a classical high resolution sample where slow sample spinning leads to an improvement of the linewidth. The zonal shims of the MAS frame are related to the classical laboratory frame shims by the following relations [31]:

$$\begin{aligned}
\boldsymbol{B}_{Z^1}^{\mathrm{MAS}} &= \frac{1}{\sqrt{3}}\boldsymbol{B}_{Z^1}^{\mathrm{LAB}} - \frac{\sqrt{2}}{\sqrt{3}}\boldsymbol{B}_{X}^{\mathrm{LAB}},\\
\boldsymbol{B}_{Z^2}^{\mathrm{MAS}} &= \boldsymbol{B}_{(X^2-Y^2)}^{\mathrm{LAB}} - 2\sqrt{2}\boldsymbol{B}_{ZX}^{\mathrm{LAB}},\\
\boldsymbol{B}_{Z^3}^{\mathrm{MAS}} &= -\frac{2}{3\sqrt{3}}\boldsymbol{B}_{Z^3}^{\mathrm{LAB}} - \frac{1}{\sqrt{6}}\boldsymbol{B}_{Z^2X}^{\mathrm{LAB}} + \frac{5}{\sqrt{3}}\boldsymbol{B}_{Z(X^2-Y^2)}^{\mathrm{LAB}} - \frac{5}{3\sqrt{6}}\boldsymbol{B}_{X^3}^{\mathrm{LAB}}.
\end{aligned} \tag{1}$$

Considering the small volume occupied by a HRMAS sample, the set of shims described in Eq. (1) is sufficient to shim a sample spinning at the magic angle in a correctly designed HRMAS probe. For a static sample, tesseral shims in the MAS frame have to be considered as well [31]. From a practical point of view, the correct setting of the shims $\boldsymbol{B}_{(X^2-Y^2)}^{\mathrm{LAB}}$, $\boldsymbol{B}_{Z^3}^{\mathrm{LAB}}$, and $\boldsymbol{B}_{Z(X^2-Y^2)}^{\mathrm{LAB}}$ is crucial to obtain a correct lineshape.

This section introduces some important concepts that are essential to shim a sample in rotation at the magic angle. We now turn to a more practical aspect and try to answer the following basic question: How different is the optimum shimming between two different samples? To answer this question, we need to examine in details the averaging process of magnetic susceptibilities by MAS.

4. General considerations on the averaging on magnetic susceptibilities differences under MAS in a heterogenous sample

The averaging of magnetic susceptibility differences present in a heterogeneous sample by MAS is an essential aspect of HRMAS spectroscopy. The additional magnetic fields created by volume elements of different magnetic susceptibilities can be treated as dipolar fields [26,32,33] that vanish when the sample is rotated at the magic angle. Strictly speaking, this averaging to zero by MAS is only true if the differences in magnetic susceptibilities are reasonably small and if the magnetic susceptibilities are isotropic. These two conditions are usually fulfilled in HRMAS samples. Consequently, the contribution of volume elements of different magnetic susceptibilities to the width of the NMR resonance vanishes and only spinning sidebands, at frequencies multiple of the spinning frequency, remain in the spectrum. The fact that inhomogeneous bulk magnetic susceptibility can be efficiently removed by MAS was demonstrated both experimentally and theoretically by Doskocilova et al. [28], VanderHart [34], and Garroway [29] in the case

of liquids and solids with random orientation. More recently, Barbara [35] used arguments based on electromagnetic calculations to explain this averaging process of MAS.

5. Averaging of magnetic susceptibilities present at the sample/rotor interfaces under MAS

The previous section explains how MAS efficiently removes the line broadening due to magnetic susceptibility gradients present inside heterogeneous systems. MAS plays also a very important role in averaging out magnetic susceptibility gradients present at the sample/rotor interface. A typical HRMAS arrangement is such that the whole sample is contained within the detection volume of the solenoidal coil. This is a remarkable feature since this experimental set-up allows the whole sample to be detected and allows for the highest sensitivity. Under these experimental conditions, magnetic susceptibility jumps exist at the sample/rotor interface along the long axis of the rotor and at the top and bottom of the sample. Typically, these interfaces are between a solvent containing heterogeneous substances and the rotor material (ZrO_2 and Teflon for the insert). On a static sample, these magnetic susceptibility jumps give rise to important local magnetic field gradients which are uncorrectable with the macroscopic shim system of the spectrometer. MAS has the very unique property of being also able to average out these magnetic susceptibility to zero. This property can be explained using arguments similar to those developed in the previous section. The consequence is that the rotor material, which is the source of the magnetic perturbations, is rotating at the same time as the sample and its effect on the sample vanishes. This very important property of MAS predicts that the optimum shim in HRMAS is sample and solvent independent provided that the rotor is always at the same position in the probe and that the volume detected is always the same. In particular, a reproducible position of the stator at the magic angle is of prime importance. This theoretical finding is supported by experimental results since one optimum set of shims is found to be sufficient for a variety of solvents and samples (data not shown). A further consequence of these ideas is that the resolution of the spectrum of a given sample should be independent of the filling ratio of the rotor. Indeed, MAS should be able to compensate the differences in magnetic susceptibilities present at the liquid/air interface. This is experimentally demonstrated in Fig. 1 which shows the 1D ^1H spectrum of a sucrose sample recorded in a 50 μl rotor filled completely with 50 μl of solution and one only half-filled with 25 μl of solution. These data show clearly that the resolution of the spectrum is not affected by the air bubbles present in the sample.

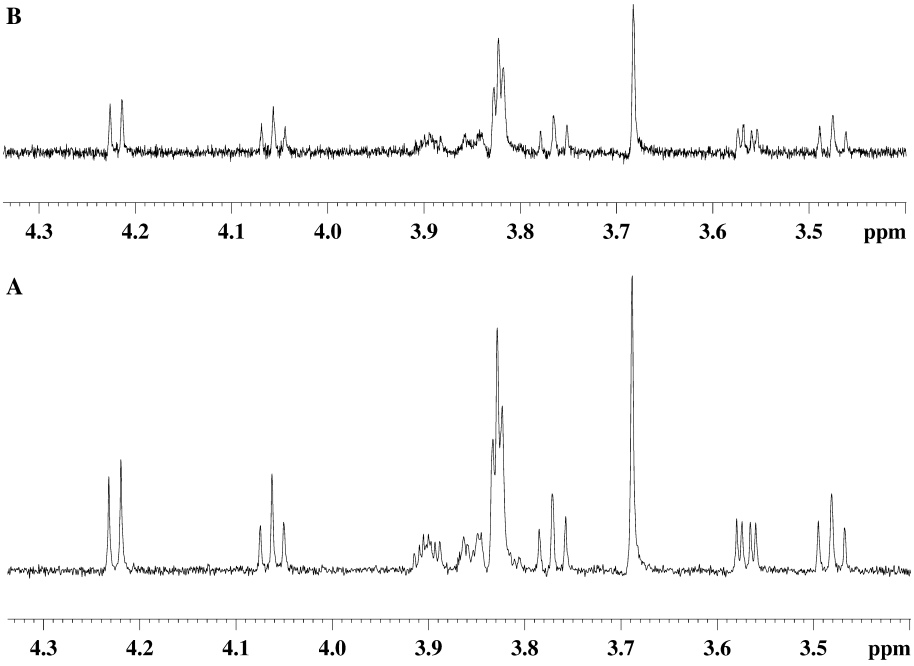

Fig. 1. 1D proton spectra of a 2 mM sucrose sample in D_2O recorded at 4 kHz on a Bruker Avance 700 MHz equipped with a 4 mm $^1H/^{13}C/^2H$ HRMAS gradient probe. The sample container is a 4 mm rotor fitted with Teflon inserts delimiting an active volume of 50 μl. (A) Fifty microliters of the sucrose solution were placed in the rotor. The volume under observation is filled with the sucrose solution. (B) Twenty-five microliters of the sucrose solution were placed in the rotor. The volume under observation contains 25 μl of the sucrose solution and 25 μl of air.

6. Temperature gradients and shimming on water samples

Since 2D experiments are frequently performed in HRMAS, the spectrometer is usually run in the locked mode with samples in deuterated solvents. The customary habit is therefore to shim a sample using the lock level as an indicator of the quality of shimming. In HRMAS, this protocol can lead to a strong deterioration of the quality of the spectra when used on water samples. This effect was observed experimentally on numerous occasions and can be explained by the following arguments: when a sample is in rotation at fairly high speeds (say 4–10 kHz), the air bearing used to rotate the sample produces some local heating due to friction against the walls of the rotor. This localized heating will create temperature gradients inside the HRMAS sample. For a sample in water, the consequences can be quite severe since the ^{1}H chemical shift of HDO is strongly temperature dependant (−11.9 ppb/°C) [36]. Measurements carried out on the water signal of an unlocked sucrose sample lead to a temperature dependence of the water frequency of about −7 Hz/°C at 700 MHz. Remembering that the intrinsic half-width of a $CHCl_3$ sample in acetone is inferior to 1 Hz, it is clear that a distribution of a few degrees can create a substantial line broadening of the water resonance. Slice selective images recorded with selective pulses in the presence of gradients along the axis of the rotor show clearly such a frequency dependence of the water signal. At a speed of 8 kHz, if the frequency of the water signal at the center of the rotor is set arbitrarily to 0 Hz, a value of about −10 Hz is found for the water resonance at the two extremities of the rotor (data not shown). These data show clearly that the temperature is the highest at the center of the rotor and then decreases when moving towards the edges along the axis of the rotor. These results corroborate similar measurements carried out in solid state NMR using lead nitrate [37]. The deuterium signal, and therefore the lock, is affected in a similar manner by the temperature gradients. At high speed, shimming by optimizing the lock level will therefore essentially compensate the broadening due to the temperature gradients. The net result is that the lock level increases significantly and the water resonance, which has the same temperature dependence, also becomes sharper. However, the solute, which is the compound of interest in the rotor, usually has a much smaller temperature dependence and will therefore be deshimmed if the lock level is increased. Fig. 2 shows the 1D ^{1}H spectrum of 2 mM sucrose in D_2O recorded at different spinning speeds from 1 to 9 kHz and back to 1 kHz. Clearly, increasing speeds create stronger temperature gradients in the sample. The HDO resonance at 4.76 ppm is well shimmed at 1 kHz. It then degrades continuously to reach a maximum line width at 9 kHz. On the other hand, the anomeric proton of the same sucrose sample (Fig. 3) which has a much lower frequency dependence on temperature exhibits the same line shape throughout the experiment. The effect is clearly reversible since decreasing the spinning speed leads to the same original spectrum. A point of practical interest is that these temperature gradients can be attenuated by cooling down the air bearing of the system and regulating the temperature.

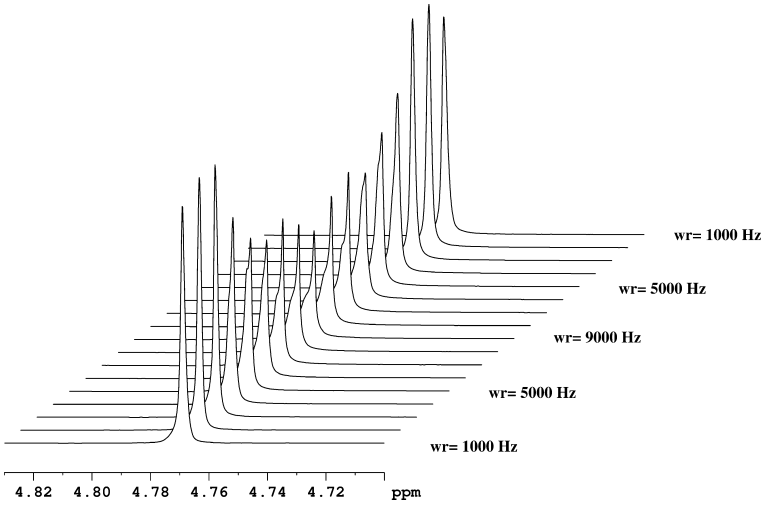

Fig. 2. 1D proton spectra of the HDO resonance of a 2 mM sucrose sample in D_2O recorded at different spinning speeds, starting at 1 kHz and going to 9 kHz and back in 1000 Hz increments. Spectra were recorded on a Bruker Avance 700 MHz equipped with a 4 mm ^{1}H/^{13}C/^{2}H HRMAS gradient probe. The spectrometer was locked on the D_2O resonance. The sucrose sample was placed in a 4 mm rotor fitted with Teflon inserts delimiting an active volume of 50 μl. The lineshape of the HDO resonance is affected by the temperature gradients generated by the sample rotation. The line broadening is due to the strong temperature dependence of the ^{1}H HDO chemical shift (−11.9 ppb/°C).

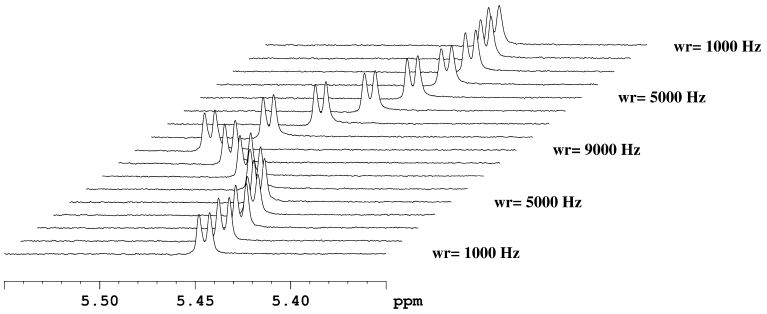

Fig. 3. Same spectra as the ones of Fig. 2 but showing the lineshape behavior of the anomeric proton of sucrose as a function of the rotor spinning speed. The chemical shift changes with the spinning speed because of the increase in temperature in the sample imparted by the rotation. Since the spectrometer is locked on the D_2O resonance, the water frequency remains at the same position whereas the frequency of the anomeric proton changes. The crucial point of this experiment is that the lineshape is not affected by the rotation and the resulting temperature gradients because of the low temperature dependence of the anomeric proton chemical shift.

7. Conclusions and recipes for shimming a HRMAS probe

The magnetic perturbations affecting the ultimate resolution of a HRMAS sample can be divided in two different categories: perturbations external to the rotor system and perturbations originating from the rotor system (rotor + sample). The main differences between these two sources of perturbation is that the former is not modulated by MAS while the latter is averaged out to zero by MAS. This means that for a given NMR probe, proper shimming has to be used to correct external, non MAS-modulated perturbations, on a chosen sample. As is customary in NMR, a 1% $CHCl_3$ in acetone is usually a very good candidate. Once good shimming is obtained on this sample, the resulting set of shims can be used for all the other samples provided that (a) the new samples are exactly in the same position and (b) the detection volume is the same. The second condition is necessarily true if the rotor used is always of the same type and if the Teflon inserts are positioned at the same position. Any additional magnetic perturbations introduced by a solvent with a different magnetic susceptibility should be removed by MAS. It is clear that fine tuning of the main shims Z^1, X^1, $X^2 - Y^2$, and XZ^1 can still be performed. Caution has to be taken when working with water samples, like cells or biopsies, since the temperature gradients present in the rotor will not always allow the use of the lock level as a reliable measure of field homogeneity. In that case, fine shimming directly on a sharp resonance in the spectrum provides the best results.

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