A compact permanent magnet for microflow NMR relaxometry

Dmytro Polishchuk¹, Han Gardeniers *

Mesoscale Chemical Systems Group, University of Twente, 7500 AE Enschede, the Netherlands

Keywords: Low-field NMR, Relaxometry, Permanent magnet, NMR-on-a-chip, Flow measurements, Microflow

1. Introduction

Nuclear magnetic resonance (NMR), by far the most informative and versatile analytical technique used in chemistry, (bio-)medicine, food, and environment, is currently meeting drastic miniaturization [1–4] thanks to the advances in CMOS electronics [5–7] and (micro-)probe fabrication [8,9]. An emerging concept of miniature NMR sensors – NMR-on-a-chip [10–12] – is giving a promise of on-demand and on-line NMR applications, while substantially reducing the size, weight and cost of the equipment and hence making it portable and accessible.

One of the key components of NMR sensors is the source of required static magnetic field – magnets – which is required to be compact, lightweight, and providing sufficient field strength and homogeneity [13]. Superconducting magnets (SCM), commonly used in NMR studies and demanding cryogenic temperatures for operation, are bulky, energy consuming and too complex for portable NMR applications. Permanent magnets, on the other hand, are power independent and operating at room temperature, however, providing relatively weak fields, ranging from 0.5 to 2.0 T (compared with up to 28 T for commercially available superconducting magnets). Superconducting magnets are a rather demanding task because of the strong sensitivity to minor displacements, non-uniform magnetization, material imperfections and fragility. Therefore, the pursuit for a suitable permanent magnet design as compact, efficient, simple in assembly and tuning as possible and with little or no electrical shimming is still ongoing.

In this work, we aimed at a compact permanent magnet system specifically designed for micro-fluidic flow measurements based on the NMR relaxometry principle. NMR relaxometry measures relaxation times of the nuclear spin system after excitation with an rf pulse – longitudinal, $T_1$, or transverse, $T_2$ – with respect to the direction of the static magnetic field. Special spin-echo sequences, such as classic Carr-Purcell-Meiboom-Gill (CPMG) sequence [19,20], enable NMR relaxometry in relatively inhomogeneous magnetic fields – hundreds of parts per million (ppm) compared to < 0.1 ppm required for low-resolution NMR spectroscopy. Therefore, our target field homogeneity was < 200 ppm over a cylindrical volume of 1 mm i.d. and 20 mm long, whereas keeping the magnet length<10 cm (sensing volume length 25–50 % of the total magnet length). Design and fabrication of such magnets, hardly possible a few decades ago, are facilitated nowadays by a number of modern materials and technologies, which we employ here: superior permanent magnet materials [21], such as families...
of Nd-Fe-B and Sm-Co materials giving strongest magnetic fields and good thermal stability, improved CNC (Computer Numerical Control) manufacturing machines with tolerances down to 10 μm (or better) and, finally, fast and reliable FEM (Finite-Element Method) numerical simulations.

Here we design and fabricate a compact (3.5x3.5x8.0 cm³) and simple in assembly permanent magnet which demonstrates sufficiently strong (0.5 T) and uniform (<200 ppm) magnetic field suitable for microfluidic flow rate measurements in the sensing volume up to 40 μL (1.2 mm i.d. × 20.0 mm). Using this magnet, we measure actual flow rates in a range from 20 ml/min down to 0.001 ml/min, which can be of interest for micro- and nano-fluidic applications. To the best of our knowledge, this is the first report demonstrating standard NMR flowmetry measured with such a compact system specifically designed for microflow measurements (with the exception of the spectrometer electronics, which nowadays can also be minimized to mm size [5–7]).

2. Magnet Design: Approach, prior experiments, and optimized prototype

2.1. Approach and prior experiments

Our approach is based on the fact that pieces of soft magnetic material (e.g. magnetic stainless steel, as used in this study) placed inside the airgap and adjoining to the two permanent magnets lead to a much more uniform magnetic field [22,23]. To obtain actual quantitative characteristics, a simple experiment was designed, as depicted in Fig. 1a. It compares magnetic field (Z-component of magnetic flux density, B_Z, measured with a transverse Hall probe) in the center of the airgap along lateral axis X in between the two permanent magnets (each made of three 7x7x40 mm³ SmCo bar magnets) for two cases – when the soft-magnetic inserts (stainless steel, grade 410) are (i) inside or (ii) outside of the bore. As seen in Fig. 1b, when inside, the 1-mm-thick steel inserts improve the field homogeneity in the volume of interest (2 mm in diameter) by around 15 times, whereas the field strength somewhat decreases by around 35% (from 425 mT to 280 mT in the center). It is noteworthy that these experimental results are in remarkable agreement with the simulated results obtained for the same geometry and tabular material parameters using the COMSOL Multiphysics® software [24]. This assured us in reliability of our computer 3D model for the magnet design, which was confirmed later for other even more complex geometries.

The simulations also give an explanation to the observed substantially improved field homogeneity: owing to the soft magnetic properties, the steel inserts effectively redistribute the magnetic flux in the airgap’s center (see inset in Fig. 1a). This distribution of flux lines is determined by the magnets’ shape, induced magnetization direction and overall system geometry. Since this problem is complex to solve analytically, the FEM modeling, based on solving Maxwell’s equation for the magnetic vector potential at each point [25], is indispensable and accurate enough for performing numerical experiments with different magnet designs.

Another experiment was designed for studying the effect of the steel inserts on the field homogeneity in the longitudinal direction – parallel to the long side of the bar magnets, axis Y in Fig. 1c. In order to make B_Y as uniform as possible along the airgap, we have come up with an idea of somewhat shorter inserts: in such a way it is possible to effectively manipulate the magnetic flux in the airgap by exposing parts of the permanent magnets at the ends. This leads to higher B_Z at the ends and thus can compensate the decrease in field strength off the airgap center. Fig. 1d shows experimental curves of the B_Z change (ΔB_Z) on the Y axis in the airgap center (X, Z = 0) for different lengths of the steel inserts. These data demonstrate over 2 orders of magnitude improvement in the field homogeneity in the central part (50% of L_max = 40 mm), which was obtained for the optimal length of the steel inserts (33.6 mm, 84% of L_max). Inset in Fig. 1d shows how the field uniformity can be tuned after magnet assembly by displacing the steel inserts. Interestingly, by such fine tuning, it is possible to achieve sufficient field homogeneity in a region somewhat off the magnet center even for not optimal insert length: for example, the curve for the ~0.05 mm off-center displacement demonstrates ΔB_Z < 50 ppm in a rather long interval of 8 mm (20% of L_max). Such off-center position is beneficial for flow measurements as the opposite longer part of the magnet can be fully used for pre-magnetizing the inflowing liquid [26].

We would like to stress that, in general, magnet optimization by computer simulation alone is not sufficient, as for field changes of the order of 100 ppm or less, as they exist in our volume of interest, the effect of imperfections in the magnets and their exact arrangement in the assembly can become substantial. Therefore, the actual real-world magnet requires finding the optimal value experimentally in the vicinity of the simulated one (in our case, the insert length).

2.2. C-shape magnet

Based on our experimental findings and COMSOL® simulations, we propose an optimized C-shape magnet design depicted in Fig. 2a,b. The permanent magnets are SmCo (4x15x80 mm³ blocks), the soft-magnetic insert plates and the external flux-guide part (yoke) are made of magnetic stainless steel 410. As the magnetic flux from the permanent magnets is mostly enclosed in the yoke, this substantially increases the field inside of the airgap (from around 200 mT to 490 mT) and decreases undesirable stray field outside. Fig. 2c shows the field line of the general safety standard of 0.5 mT, which is very close to the magnet and thus can satisfy safety requirements. The “C” shape allows access from the side, and the 5-mm airgap has enough space for additional electronics (such as shimming elements, if better field uniformity is desired) or microfluidic fixtures inside. By decreasing the airgap height, it is possible to achieve even higher magnetic field, which can be exploited for further field increase and/or miniaturization. Optimal length of the steel inserts leads to a substantial improvement of the longitudinal field homogeneity, as seen in Fig. 2d. These results are fully consistent with the behavior presented in Fig. 1d and discussed therein. Fig. 2d also shows a curve for the configuration without steel inserts in the airgap: the curve has local irregularities, which is a notorious problem usually attributed to the nonuniform magnetization distribution in the permanent magnets [27]. The steel inserts effectively smooth out these local irregularities, though their footprint (much weaker in magnitude) can be still seen in case of our 1-mm steel inserts; cf. insert to Fig. 2d. Fig. 2e compares lateral (vs X) and vertical (vs Z) magnetic field variations (in the magnet center, Y = 0) for the configurations with and without steel inserts: for the latter case (bottom panel in Fig. 2e), besides much larger field inhomogeneity, the characteristic field maximum/minim in the X/Z-dependences are offset from the initial central position. What is more, if one of the magnets is rotated by 180°, the curve acquires very steep slope (see Fig. 2f) indicating that the magnets have a substantial magnetization gradient in the longitudinal direction. This gradient is compensated in the default configuration. Same behavior was observed for a few different sets of magnets from the same batch; sets 1, 2, 3 in Fig. 2f. The optimized steel inserts have improved the field homogeneity for five made prototypes with different permanent magnet sets to the same extent as presented here, indicating high reproducibility of
the results. Therefore, even in case of such imperfect permanent magnets, our design can provide sufficient field homogeneity.

3. NMR experiments

3.1. NMR field mapping

Fig. 3a shows an FID (Free Induction Decay) curve obtained for deionized water using our C-shape magnet prototype \( (L_{\text{ins}} = 70 \text{ mm}) \) and a solenoid coil probe (sensing volume 1.6 mm i.d., 1.8 mm long) after an optimal 12 \( \mu \text{s} \) excitation pulse \( (P90) \). The decay time of the FID, \( T_2^* \approx 0.1 \text{ ms} \), is much shorter than the relaxation time of water at room temperature, \( T_2 \approx 2.5 \text{ s} \), as the consequence of the field inhomogeneity. Fast Fourier Transform (FFT) of the FID gives a resonance peak with central resonance frequency \( f \approx 20.8 \text{ MHz} \), giving \( B \approx 490 \text{ mT} \). The obtained full linewidth at half maximum, FWHM \( \approx 200 \text{ ppm} \), which is in this case the quantitative characteristic of the field distribution in the sensing volume, is meeting desired specifications even though the used probe had much larger i.d. than the targeted 1.0 mm.

Fig. 3b demonstrates how \( f \) (black) and FWHM (orange) change when the probe is moved along the magnet airgap (along axis Y; \( X, Z = 0 \)). Fig. 3c shows the change in \( f \) and FWHM in the lateral \( X \)-direction at \( Y = -5 \text{ mm} \). These NMR data remarkably reproduce what was obtained using the Hall probe; cf. Fig. 2c.

Employing even smaller coil probe (sensing volume 0.6 mm i.d., 0.5 mm long) with copper sulfate water solution as the medium (its \( T_1 = 50 \text{ ms} \) enables faster measurements), it was possible to obtain a 3D field map in the region of interest. Fig. 3d shows the 3D region where \( |f - f_0| < 50 \text{ ppm} \) (or equally \( |\Delta B| < 50 \text{ ppm} \)); Fig. 3e,f give selected 2D slices of this region in \( YZ \) (panel e) and \( XZ \) (panel f) planes. Such field mapping gives more detailed information on the field distribution in the region of interest and confirms sufficient field homogeneity of our C-shape magnet.

3.2. Test flow measurements

Since our C-shape magnet was designed for micro-flow measurements and its size is defined by the desired sensing volume (1 mm i.d., 20 mm long), we have fabricated an NMR probe having a long solenoid coil with the sensing volume 1.6 mm i.d. by 20 mm long. Fig. 4b shows the probe with the coil wound around a 2.0 mm o.d. glass tube and soldered to a PCB with single-ended impedance matching circuit. All the components are mounted on a 3D-printed holder of the sizes matching the airgap so that the probe can be easily slid into and fixed at the right position inside the magnet. Such a probe design allows quick and easy replacement of probes of different coil sizes.

Here we give an example of flow rate measurements based on NMR relaxometry by measuring \( T_2 \) relaxation curves for various flow rates; more details about the measurement method are in Ref. [28]. The \( T_2 \) curves undergo changes for different flow rates because of the outflow of the excited spins from the sensing coil volume. In the used CPMG pulse sequence (Fig. 4a), P180 pulses have the same pulse duration as P90 (40 \( \mu \text{s} \)) but twice larger amplitude and twice longer time between the pulses. Because of the choice of such pulse sequence, the first spin-echo peak is lower

![Fig. 1. Effect of soft-magnetic (stainless steel 410) inserts on field homogeneity in the airgap between two permanent magnets. (a) Two experimental settings when the 1-mm-thick inserts are inside (IN) or outside (OUT) the airgap and simulated distributions of magnetic field lines in transverse plane XZ with respect to the long side of the permanent magnet bars \( (7 \times 7 \times 40 \text{ mm}^3) \). (b) Measured and simulated change in Z-component of magnetic flux density, \( \Delta B_z \), in lateral direction (axis X) in the airgap center \((Y, Z = 0)\). (c) Experimental setting for studying \( B_z \) homogeneity in the longitudinal direction (axis Y). (d) Corresponding longitudinal \( \Delta B_z \) in the center of the airgap \((X, Z = 0)\) for different insert lengths, \( L_{\text{ins}} = 40, 34.0, \) and 33.6 mm or 1.0, 0.85, and 0.84\( \times L_{\text{mag}} \), where \( L_{\text{mag}} = 40 \) mm is the magnet length. Insert shows the change in \( \Delta B_z \)-vs-\( Y \) \((L_{\text{ins}} = 33.6 \text{ mm})\) when adjusting the insert off-center displacement along \( Y \) (values in mm).](image-url)
Fig. 2. Optimized C-shape permanent magnet. (a) Physical prototype and its main components. (b) Assembled magnet with aluminum fixtures for simple assembly and tuning the longitudinal field homogeneity (by displacing the inserts with side screws; parameter Δ in panel a). (c) XZ cross-section view of the equipotential B field contours obtained by the FEM simulations (slice at Y = 0, magnet center). (d) Longitudinal field change ΔB_Z vs Y in the center of the airgap (X, Z = 0) for different insert lengths L_{ins} = 80, 74, 72, and 70 mm and without inserts. (e) Lateral (vs X) and vertical (vs Z) ΔB_Z in the magnet center (Y = 0) for L_{ins} = 72 mm (top) and without inserts (bottom). (f) Longitudinal ΔB_Z vs Z for the magnet without inserts for different sets of permanent magnet blocks (set 1, 2, 3) and for the case when one of the two blocks in each set is rotated by 180° in XY plane (Y direction is changed to −Y). Def. marks the default orientation of the blocks.

Fig. 3. FID measurement and NMR field mapping. (a) FID measured for deionized water using a solenoid coil probe (sensing volume 1.6 mm i.d., 1.8 mm long) and corresponding FFT resonance peak (insert). (b), (c) Relative change in peak central frequency (black squares; left tick axis) and peak FWHM (orange diamonds; right tick axis) versus longitudinal Y position (X, Z = 0; panel b) and lateral X position (Y, Z = 0; panel c). (d) 3D surface of region with |f - f_0| < 50 ppm, where f_0 is the average frequency in the volume of interest (|X, Z| < 0.5 mm, Y = (-5, −25). (e-f) Selected 2D slices in planes YZ (X = 0; panel e) and XZ (panel f). The map was obtained using a small coil with sensing volume 0.6 mm i.d. and 0.5 mm long and copper sulfate water solution inside. X, Y, Z = 0 correspond to the airgap center. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)
in amplitude than the second one. Maximums of the spin-echo peaks build a $T_2$ curve, examples of which measured for deionized water and different flow rates are shown in Fig. 4c. The static relaxation curve fits perfectly by exponential decay function $\exp(-t/T_2)$ with $T_2 = 2.25$ s, which is close to the look-up table value for water at room temperature.

It is important to note that the time between $P_{180}$ pulses (2\tau) in our measurements was rather short, $\tau = 0.1$ ms, whereas choosing more typical $\tau = 1$ ms decreases (effective) $T_2$ to around 1 s (not shown). This is the consequence of a finite field homogeneity and interdiffusion of spins to the regions of slightly different field strength in the sensing volume. Choosing $\tau = 0.1$ ms effectively suppresses the effect of spin diffusion on our measurements.

The flow component can be extracted from the measured relaxation curves by dividing them by the static curve (for better SNR, by its exponential fit), as shown in Fig. 4d. Only the first part of the normalized curves (top half) is linear, whereas the second part (bottom half) is tailing off. Such behavior is typical for a laminar flow, which is the case for thin capillaries, and explained by the flow velocity profile: the flow velocity is maximum in the center (2 times of the average flow speed) and is getting slower in radial layers closer to the capillary wall. It means that, when the majority of the excited spins in the central part is out of the sensing volume, the slowly flowing spins at the wall are still contributing to the signal.

To correlate obtained curves with corresponding flow rates, it is helpful to fit the linear part of the curves and find a correlation parameter — e.g. time $t_0$, when the linear fit crosses the time axis, is a convenient parameter to use. Within such approach, from Fig. 4d, it is well seen that the lower flow rate determination is limited by the increased noise at later times (the noise is technically amplified by dividing experimental curves by values close to 0), whereas the upper flow rate is limited by a substantial decrease in the initial amplitude. The latter is owing to the fact that inflowing spins do not have enough time to align along the $B_0$ field of the magnet, i.e. the upper flow rate is limited by the premagnetization length of the magnet (in our case, since the probe was shifted to one end, this length is around 40 mm, 0.5$L_{\text{mag}}$).

Fig. 4e shows obtained $1/t_0$ versus set flow rates for two probes of different lengths (4 mm and 20 mm) and i.d. (0.6 mm and 1.6 mm, respectively). The horizontal offset between the curves is because the actual linear flow velocities are different for same flow rates; the flow velocity is higher in capillaries with smaller i.d. The larger 20-mm probe allowed measurements of flow rates up to 20 ml/min (flow speed 165 mm/s), whereas the smaller 4-mm probe extended the range down to 0.001 ml/min (0.058 mm/s). Overall, these two probes cover the flow-rate range over 4 orders of magnitude. These data illustrate how choosing an appropriate probe size makes it possible to work in a desired flow rate range.

The data sets for the 4-mm probe (marked by different color tones in Fig. 4e,f) were obtained using a syringe pump supplied with syringes of different sizes for optimal pump functioning in different flow rate ranges. The relative deviations from linear fits, shown in Fig. 4e, having the average less than 5 %, can be further decreased by averaging, temperature stabilization, and improving data processing.

It is noteworthy that presented data were obtained using single-shot measurements in the ambient environment at room temperature without any temperature stabilization or temperature compensation. Temperature variations, and especially temperature gradients over position, can affect the magnetic field strength and its homogeneity, since the temperature coefficient of the used magnetic materials is around 200–300 ppm/K. However, in the current design, large temperature gradients are avoided by the one-piece steel yoke having high thermal conductivity and thus quickly redistributing the heat over the whole magnet. Moreover, our simulations demonstrate that the temperature variations affect the field strength but leave the field homogeneity basically unchanged.

4. Discussion and conclusions

The proposed C-shape magnet design, efficient and simple in assembly, can be a starting point for further improvements, some of which we discuss here on the basis of our simulations. For example, by increasing the height and width of the SmCo magnets and the yoke, resulting in a magnet with total size not larger than 6x6x8 cm³, the field strength of 1 T can be achieved, which e.g. can quadruple the SNR. On the other hand, as seen in Fig. 2b, the
magnetic flux is mostly focused in certain regions of the yoke: our numerical studies (not shown) indicate that optimizing the yoke shape can lead to at least 25% additional decrease in size and weight.

The key component behind the much-improved field homogeneity in our magnet design is the magnetic steel inserts having a shape of simple rectangular flat plate. By tapering an inner side of these plates in a special manner, it would be possible to come up with even better field homogeneity or, alternatively, with same field homogeneity but much smaller magnets. This approach is somewhat similar to the methods based on shaped pole pieces [29], which can be also 3D printed [30]. However, employing such tapering to construct a magnet with considerably better homogeneity (below 10 ppm) can be problematic owing to the high sensitivity to manufacturing and material imperfections (cf. Fig. 2c-e). Our simulations indicate that 10-µm errors in the shape or position of such tapered inserts lead to noticeable field distortions. On the other hand, our numerical experiments show that a few-ppm field homogeneity, but less sensitive to fabrication and material imperfections, is possible with a bit more complex design based on thick prism-like pole pieces combined with a specific permanent magnet arrangement (work in progress; will be reported elsewhere).

Our test flow measurements demonstrate that the magnet is suitable for sensing flow rates in a wide range, from 0.001 to 20 ml/min. The measurements of high flow rates are technically limited by the premagnetization length. In fact, the premagnetization section can be made separately (having even simpler and more compact design) and when long enough can potentially extend the range up to 100 ml/min. At the same time, the main magnet can be made at least twice shorter (<40 mm) while preserving sufficient field homogeneity (≤200 ppm) in the desired sensing volume (roughly 1 mm i.d., 20 mm long; 0.5 lmag). The low range limit is essentially determined by miniaturizing NMR probes. Owing to the design scalability, the magnet can be made even more compact for smaller probes.

In summary, we have proposed and demonstrated a scalable magnet design suitable for micro-fluidic flow measurements based on the NMR relaxometry principle. The sensing volume and broad range of flow rates are in the desired range of micro- and nanofluidic applications. Its compact form factor, simplicity in assembly and tuning are key for developing portable (multi-phase) NMR-based flow meters for laboratory, field and industrial settings. Furthermore, this approach can be advanced for designing compact permanent magnet systems for low-resolution NMR spectroscopy.

Data availability

Data will be made available on request.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: [Han Gardeniers reports financial support was provided by Dutch Research Council (NWO)].

Acknowledgements

This work is (partly) funded by the Dutch Research Council (NWO) through the research programme FLOW+, project number 15025. The authors thank Jankees Hogendoorn, Lucas Cerioni, Marco Zoeteweij, and Koert Kriger of Krohne New Technologies BV, Eren Aydin of Delft University of Technology, and Rob Dierink of the technical center (TCO) at the University of Twente for their input and support.

Appendix A. Materials and methods

Magnet materials and assembly

The assembled magnet prototypes have been built using custom-made permanent magnets SmCo17 (Schallenkammer Magnetysteme BmbH, Germany) and magnetic stainless steel 410 (Salomon’s Metalen B.V., the Netherlands): the inserts were laser-cut from a 1-mm thick plate, and the yoke was CNC machined from a 60-mm diameter round bar. The inner bed for the inserts and SmCo magnets as well as the side fixtures were made of aluminum. The prototypes were manufactured at the Technical Center for Education and Research (TCO) of the University of Twente.

Field mapping

The magnetic field inside the magnets were measured using a magnetometer (Teslometer 3002, Project Elektronik GmbH, Germany) equipped with a 0.1 × 0.1-mm² active area Hall probe (Transverse Probe T3-1,4–5.0–70). The 3D field maps were measured by employing compact motorized translation stages (Thorlabs, Inc.) mounted in 3-axis XYZ configuration. NMR field mapping was carried out using a solenoid coil probe with active cylindrical volume 0.6 mm diameter and 0.5 mm long, filled with a copper sulfide water solution (CuSO4 concentration 8 mg/ml; effective T2 ≈ 50 ms).

NMR measurements

were performed by means of commercial electronics (Benchtop MRI unit, Pure Devices GmbH, Germany) and in-house made solenoid coils and impedance matching circuits. The coils of different lengths were wound around glass capillaries using a 32-µm diameter enameled copper wire fixed with a UV light curing glue. The relaxation curves were measured using CPMG pulse sequence with the pulse duration from 10 µm to 40 µm (respectively for the 4-µm and 20-mm long coils) and the echo time between the P180 pulses of 0.2 ms; the sample frequency was 200 kHz. The amplitudes of the P90 and P180 pulses were 1.9 V and 3.8 V, respectively. Different flow rates were supplied using a syringe pump (the Harvard Apparatus PHD 22/2000) and PTFE tubing.

COMSOL® simulations

were performed using AC/DC module, software version 5.6. The static magnetic field distribution was modeled using Magnetic fields, No Currents interface: Remanent flux density model for the permanent magnets (partially accounts for demagnetization and has best agreement with the experiments) and B-H curve model for the soft-magnetic steel inserts and yoke. It is noteworthy that (fixed) Magnetization model used for the permanent magnets gives similar results, whereas Nonlinear permanent magnet model, often used for magnets to account for (shape) demagnetization, gives rather large discrepancy with the performed experiments.

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In NMR of liquids, the nuclear spins need a rather long characteristic time to align along the applied magnetic field – spin-lattice relaxation time, T1, which for example can last for a few seconds for tap water at room temperature. Ideally, the nuclear spins should be well magnetized when entering the NMR probe placed in the magnet center.

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