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# Mobile NMR for geophysical analysis and materials testing

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**Abstract:** Initiated by well logging NMR, portable NMR instruments are being developed for a variety of novel applications in materials testing, process analysis and control, which provides new opportunities for geophysical investigations. Small-diameter cylindrical sensors can probe short distances into the walls of slim-line logging holes, and single-sided sensors enable non-destructive testing of large objects. Both sensors are characterized by small sensitive volumes. Barrel-shaped magnets that accommodate the sample in their center have higher sensitivity due to a larger sensitive volume but can accommodate only samples like drill cores, which fit in size to the diameter of the magnet bore. Both types of magnets can be scaled down to the size of a coffee mug to arrive at sub-compact NMR equipment. Portable NMR magnets are reviewed in the context of applications related to geophysics.

**Key words:** Mobile NMR, portable NMR, relaxation, imaging, spectroscopy

## 1 Introduction

The development of NMR methods, hardware, and applications has been in constant evolution since the first experiments on condensed matter in 1945. Currently, sub-compact NMR machines, that is, mobile or portable NMR instruments smaller than desktop NMR machines are being developed following the trend in miniaturization encountered in many areas of technology, such as in cell-phone technology. In fact, apart from the magnet, the cell phone is an NMR console. To arrive at a small NMR device the most bulky component, the magnet, needs to be made small. A prominent example of a small NMR magnet is the NMR-MOUSE (Nuclear Magnetic Resonance-Mobile Universal Surface Explorer) (Eidmann et al, 1996), a stray-field NMR sensor similar to a well logging sensor but with a more inhomogeneous field, which is suitable for many applications in non-destructive testing of large objects (Blümich et al, 2008a). Different efforts are under way to arrive at small, sub-compact NMR machines with a variety of magnets (Blümich et al, 2008a; Demas and Prado, 2008) dedicated to specific purposes, such as the analysis of geophysical drill cores (Anferova et al, 2007), soil moisture, and the detection of particular analysis targeted by functionalized super-paramagnetic nano-particles in biological samples (Sillerud et al, 2006; Lee et al, 2008). Most systems are void of spectroscopic resolution, and find application in relaxation analysis and imaging. With the progress in shimming the magnetic field, the ratio of signal-bearing volume to magnet size increases, and NMR spectroscopy becomes feasible with small permanent magnets.

Mobile NMR started with the development of NMR sensors for use in well logging (Brown et al, 2001; Coates et al, 1999; Dunn et al, 2002). Subsequently small and compact desktop spectrometers with permanent magnets were developed, which received little attention compared to the high-field spectrometers for chemical analysis and medical imagers but find important use in different fields like polymer and food analysis. With the electronics and the magnet becoming smaller, the compact desktop instruments shrink to sub-compact instruments that can be carried along to the site of the object (Blümich et al, 2008a).

In the course of miniaturizing the magnet, the development of the NMR-MOUSE is a prominent milestone (Eidmann et al, 1996). Several variants of this unilateral NMR sensor with different magnetic field profiles and degrees of homogeneity have been developed (Blümich et al, 2008a; Demas and Prado, 2008). One is the NMR endoscope (Haken et al, 1998; Mauler, 2006; Zur, 2004). This is a device similar to a well logging sensor but with a diameter of 2 cm and less. In fact for analysis of arterial plaques, an endovascular NMR endoscope with an outer diameter of just 1.7 mm has been developed (Zur, 2004). Such devices can be scaled to fit slim-line boreholes for measurement of soil moisture and moisture transport in the bore-hole walls. Yet with decreasing diameter and size, the accessible depth shrinks. For example, the useful measurement depth for a 20 mm diameter endoscope is less than 5 mm. Access to larger depths requires larger sensors. For this purpose the Profile NMR-MOUSE (Perlo et al, 2005) has been scaled up to reach depth of 25 mm. The resultant device is a compact, unilateral NMR sensor with

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dimensions 20 cm × 20 cm × 10 cm and a flat sensitive slice located 25 mm above the sensor surface. This sensitive slice can also be shaped convex or concave to collect signal from a slice fitting the surface of a cylinder. Such slices can be used to measure depth profiles, that unravel the stratigraphy of layered structures in terms of NMR signal amplitudes, relaxation and diffusion parameters. The strong gradient of the order of 10 T/m gives rise to a thin sensitive slice. This is good for resolving thin layers, but complicates the sampling of representative properties of soil and rock, which are often characterized by a hierarchy of voids and pore structures ranging from sub-micrometers to centimeters.

Average parameters are obtained more reliably from larger volumes. This requires more homogeneous fields for measurements, so that magnets are preferred, which surround the region of interest of the object. This is why the object shape is preferably cylindrical, and such magnets can be used for studying drill cores and soil columns. For this purpose, open-ended, tube-shaped magnets have been constructed from permanent magnet material following the ideas worked out by Halbach (Halbach, 1980; Raich and Blümler, 2004). Yet the inevitable variation in shape and magnetic properties of the individual magnet blocks used to compose such a Halbach magnet lead to variations in the magnetic field that so far have been minimized at the expense of increasing the magnet size or reducing the sample volume. But recently, novel shim technologies to homogenize the magnetic field generated by arrays of blocks from permanent magnets have been designed, that lead to a significant increase of the ratio of sensitive volume to magnet size (Perlo, 2007). As a consequence, Halbach magnets can now be used for desktop imaging and for miniature NMR spectrometers to perform chemical analysis in environments under extreme conditions of pressure and temperature or in dangerous areas.

## 2 Slim-line logging

For studies of groundwater and moisture in the vadose zone, narrow logging holes become increasingly popular, and it is of interest to develop slim-line NMR logging tools that fit such narrow holes. While commercial tools are about 10 cm in diameter, the endovascular NMR endoscope has an outer diameter of only 1.7 mm (Zur, 2004). As the diameter of these inside-out NMR devices becomes smaller, also does the maximum depth of the sensitive volume. We found that a 20 mm outer diameter endoscope (Mauler, 2006) constructed from permanent magnets similar to the endovascular endoscope is suitable for studying water content and drying characteristics of soil columns (Fig. 1(a) and (b)). At 5 mm from the surface of the cylindrical magnets from NdFeB, the magnetic flux density is 0.16 T and the gradient is 23 T/m. In such inhomogeneous magnetic fields, NMR measurements are conducted with multi-echo trains for detection. The amplitude at time zero of such an echo train scales with the number of protons in the sensitive volume, which is with the moisture content in geophysical applications. The shape of the echo envelope decay may be analyzed to extract transverse NMR relaxation times $T_{2\text{eff}}$ and their distributions, which may provide information on pore-size distributions for fully water saturated porous rocks and sand (Blümich et al, 2008a; Brown et al, 2001; Coates et al, 1999).

Drying curves of an initially water saturated sand column were measured over 15 days with the slim-line logging tool moving up and down over a distance of 36 cm inside a plastic pipe embedded in the sand (Fig. 1(c)). A general problem with such measurements in unsaturated soil is environmental noise that contaminates the measured data. A particular issue with such narrow tools is the close distance of the sensitive volume to the plastic pipe that accommodates the tool. When shifting the tool to different positions, lateral tool motion may lead to overlap of the sensitive volume with the pipe wall. Furthermore, the sensitive slice is thin so that the sensitive volume and with it the signal-to-noise ratio are small. To achieve a distant and large sensitive volume, the diameter of the magnets should be as large as possible.

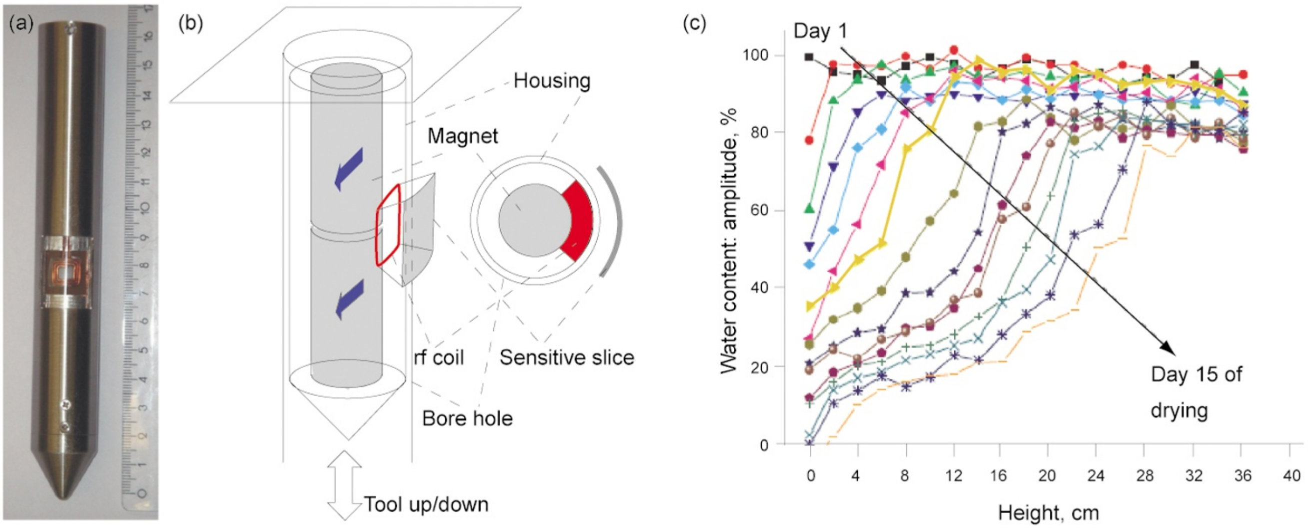

**Fig. 1** Slim-line logging tool with an outer diameter of 20 cm

(a) Photo; (b) Sketch of the tool showing its construction from two cylindrical magnets with transverse polarization, the radio-frequency (rf) coil, and the sensitive slice; (c) Drying curves of an initially fully water saturated sand column in terms of moisture content versus height for 15 days of

## 3 Depth profiling

The curvature of the sensitive slice of the slim-line logging tool is largely determined by the polarization and geometrical arrangement of the magnets. Depending on the gap between the two magnets (Fig. 1(b)), the sensitive slice can be curved either way along the axis of the tool. At one particular gap width between the magnets, this curvature vanishes and the slice is parallel to the cylinder surface. This principle of shaping the stray field of a magnet arrangement has been made use of in the construction of the Profile NMR-MOUSE (Perlo et al, 2005). This stray-field NMR sensor consists of four magnet blocks arranged with gaps on an iron plate that serves as a magnetic yoke (Fig. 2(a)). The magnet gaps are adjusted in such a way, that the magnetic stray field is homogeneous in a slice a given distance in the center above the sensor and parallel to its surface. By varying the distance between the sensor and the object, the sensitive slice is shifted within the object, and profiles of NMR parameters versus depth into the object can be measured.

The distance variation is achieved by means of a computer driven precision positioning device, which moves the NMR-MOUSE in fixed small increments between scans (Fig. 2(a)). The sensor is connected to a portable spectrometer, which in turn is connected to a computer (Fig. 2(b)). With this device a depth profile through the mortar cover of a painted stone wall was measured after spraying the wall with water (Fig. 2 (c)). As the moisture content is low and the sensitive slice is thin, the measurement times are long. During this time, the water spreads inside the mortar by diffusion and dries from the outside. These effects determine the moisture profile in addition to the moisturizing properties of the different layers of the wall. At about 3 mm depth into the wall, a step was found in the profile which points at an interface between two mortar layers. These mortar layers were subsequently identified when drilling a hole into the wall after the measurement (Fig. 2(d)).

For materials testing, the Profile NMR-MOUSE is a most useful tool (Blümich et al, 2008a). The structure of layered materials is readily revealed and often can be correlated to its function, most notably for barrier layers against the transgression of moisture in containers like gasoline tanks but also human skin. Sensors with sensitive slices 3 mm, 5 mm, 10 mm, and 25 mm away from the surface have

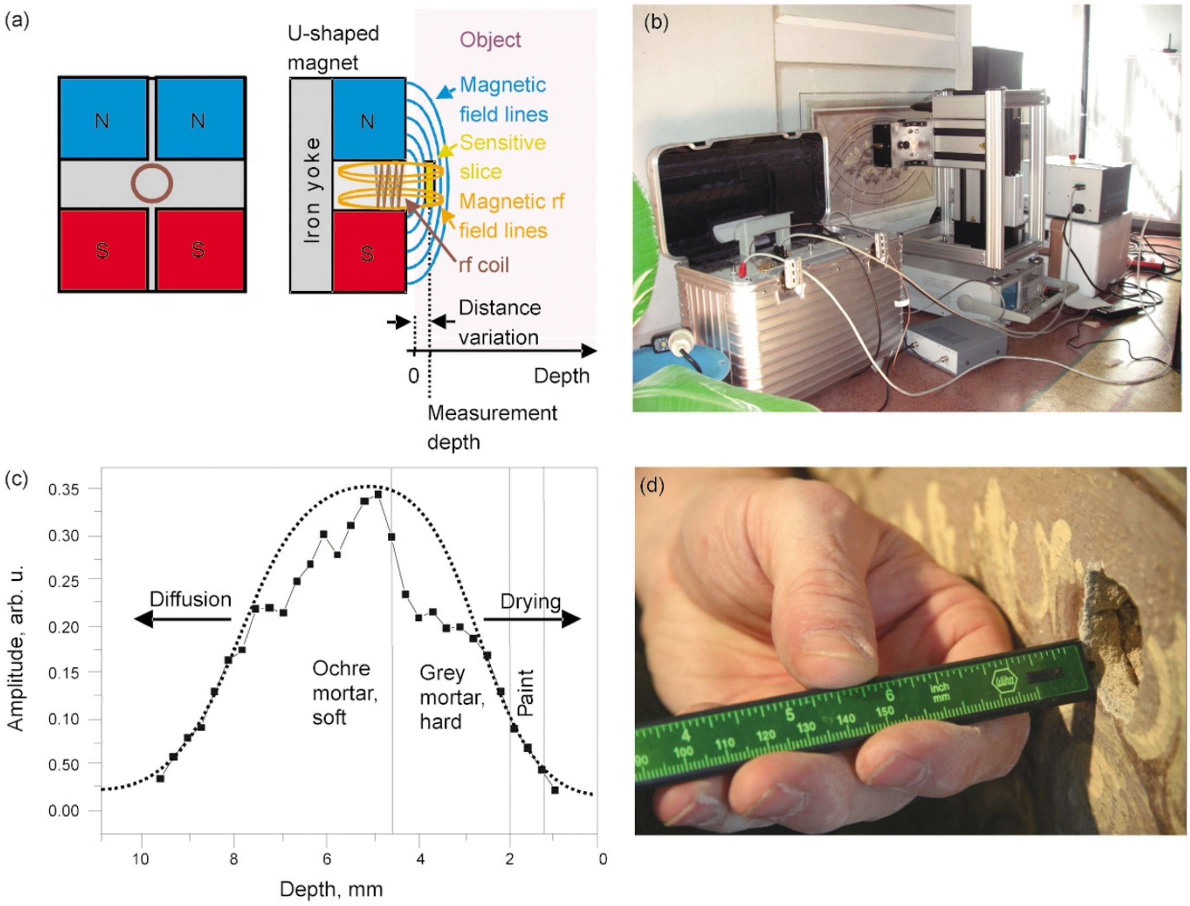

**Fig. 2** (a) Construction and principle of use of the Profile NMR-MOUSE. The flat sensitive slice is located at a fixed distance above the sensor surface. By varying the distance between the sensor and the object, the sensitive slice is shifted within the object; (b) Set-up for the Profile NMR-MOUSE to measure a depth profile into a wall. The NMR-MOUSE is mounted on a computer-driven positioning device that adjusts the distance between the wall and the sensor in fixed small increments between scans. The NMR spectrometer is mounted in the aluminum case in front; (c) Depth profile across 10 mm of the wall section depicted in (b) measured during 12 h after spraying the wall with water. The profile shape is influenced by water diffusion into the wall and drying out of the wall. A step is found at about 3 mm depths; (d) This step marks the interface between two mortar layers, which were discovered after the measurement when opening up the wall

been built and tested. The closer the sensitive slice to the radio frequency coil, the better the sensitivity and the depth resolution of the sensor. The best depth resolution achieved so far is 2.3 μm (Perlo et al, 2005). Less than that is sufficient to study paint layers in old master paintings (Presciutti et al, 2008). Curved sensitive slices to measure cable insulations or pipe walls from the inside or the outside can also be realized by different gap widths in the magnet construction. Distance information in soft matter can be obtained with an accuracy of better than 10 μm in time intervals of less than 1 s, when resolving position across the slice thickness by Fourier imaging making use of the strong gradient perpendicular to the plane of the slice for space encoding. The same gradient can conveniently be used for diffusion measurements (Rata et al, 2006).

## 4 NMR relaxation: drill cores and processes

The sensitive volumes of stray-field NMR sensors are typically small or the frequency is low, as large sensitive volumes at high field strength are hard to achieve outside the magnet. For this reason, measurements in a homogeneous magnetic field inside a magnet should be preferred over stray-field NMR measurements outside a magnet whenever possible, as closed magnets can provide large sensitive volumes at high field strengths. Magnets built from permanent magnet material are smaller than electromagnets, so that small, sub-compact magnets are preferably built from FeNdB or SmCo magnets (Demas and Prado, 2008). An ingenious way to build hollow cylinder magnets is due to Halbach (Halbach, 1980; Raich and Blümler, 2004). Such a Halbach magnet is composed of individual permanent magnet blocks suitably arranged, so that the magnetic field inside is transverse to the cylinder axis and ideally zero outside. Due to the granular structure of the magnet material and inaccuracies in shape, position, and magnetization direction of the magnet blocks, the magnetic field inside a Halbach magnet is not good enough for chemical shift resolved spectroscopy but often good enough for relaxation measurements.

NMR relaxation measurements are elementary to geophysical well logging and core analysis (Coates et al, 1999; Dunn et al, 2002). A drill-core scanner has been built to map the properties of fluid saturated drill cores in parallel transverse slices along their length (Fig. 3(a)) (Anferova et al, 2007). Between acquisitions, the magnet is shifted in steps along the length of the drill core under computer control. At each position of the drill core (Fig. 3(b)), a multi-echo decay can be measured, for example, and the distribution of relaxation times (Fig. 3(c)) calculated from it by inverse Laplace transformation. From these distributions, different properties of the porous medium can be derived (Coates et al, 1999; Dunn et al, 2002), such as porosity and fluid permeability. Porosity is proportional to the integral over the distribution (Fig. 3(d)), and permeability is estimated with the help of models by partial integration.

Such simple relaxation measurements are fast and can be conducted in a few seconds. A more detailed characterization of fluids in porous media in the weakly inhomogeneous fields of Halbach magnets is possible by multi-dimensional Laplace NMR (Song, 2006). A 2D relaxation time distribution, for example, requires the acquisition of 32 or so relaxation decays. As an example, Fig. 4(b) shows the $T_2$-$T_2$ exchange distribution of water saturating aluminum oxide powder. It has been measured with the Halbach magnet shown in Fig. 3(a). Three environments can be identified for the water molecules which are understood as the free bulk water and the water molecules in the two layers of the Helmholtz double layer covering the $Al_2O_3$ particles (Blümich et al, 2008b). The existence of cross-peaks indicates exchange of the water molecules between all three environments within the exchange time $t_{mix}$ of 80 ms.

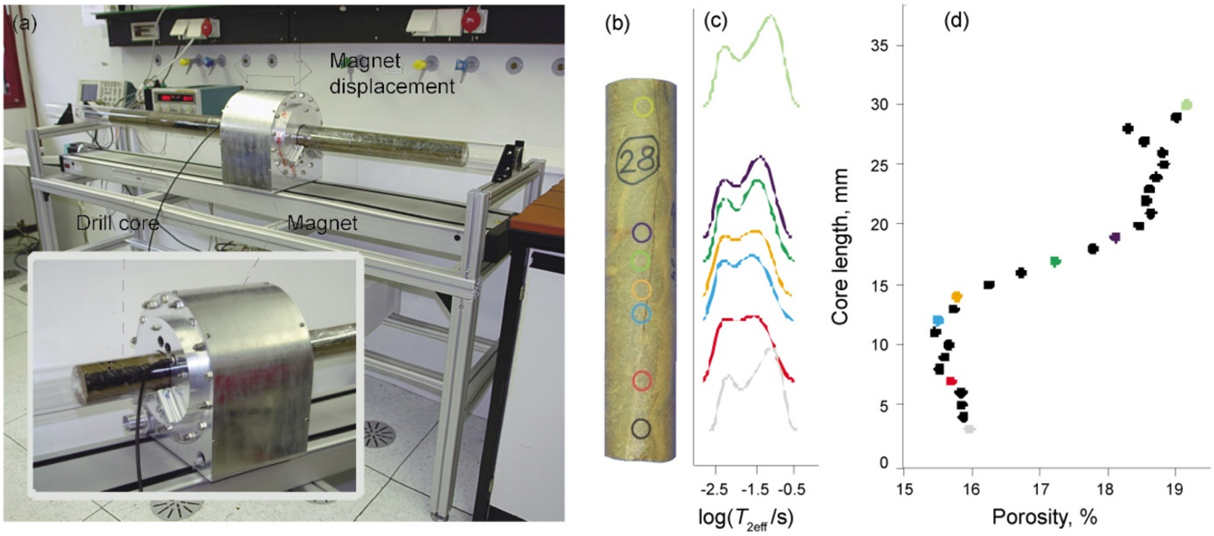

**Fig. 3** Core scanner for analysis of geophysical drill cores (a) The drill core rests in a plastic pipe which passes through a Halbach magnet. Different positions are addressed by moving the magnet under computer control along the core before executing the measurement; (b) Drill core from sandstone with different positions marked; (c) Distributions of transverse relaxation times for different positions; (d) Porosity for different positions along the drill core

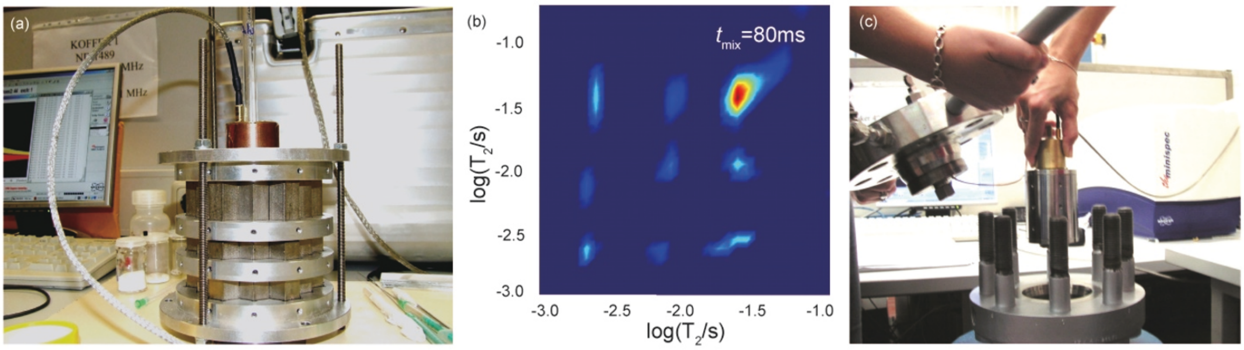

**Fig. 4** (a) Halbach magnet consisting of 12 hexagonal bar magnets that accommodates a sample tube for relaxation experiments; (b) 2D $T_2$-$T_2$ exchange distribution of water saturating aluminum oxide powder measured with the Halbach magnet shown in (a). The water molecules exchange between three different water environments; (c) Sub-compact Halbach magnet that fits into an autoclave for pressure-dependent measurements

As small magnets can readily be moved, they can also be carried to hostile environments, such as those characterized by high temperature or high pressure. In a way this is known from well logging NMR, where measurements are executed at temperatures up to 150° C and pressures up to 1400 bar. A high-temperature Profile NMR-MOUSE has been built from SmCo magnet blocks to measure the temperature dependence of NMR parameters of different materials up to 140° C (Blümich et al, 2008b). Furthermore, a small Halbach magnet has been built from six identical bar magnets with hexagonal cross-sections, which fits into an autoclave for pressure-dependent measurements (Fig. 4(c)). This demonstrates that sub-compact NMR magnets are small enough to fit into commercially available equipment for measurements at varying temperature and pressure, eliminating the need to acquire dedicated probes that create such environments inside the bore of superconducting magnets.

## 5 Imaging

For imaging the demands on field uniformity are higher than for relaxometry. The highest demands on field homogeneity are for spectroscopy. Slice-selective 2D images have been obtained from a stray-field magnet similar to the Profile NMR-MOUSE but fitted with coils to produce a magnetic field parallel to the main field with orthogonal gradients in the plane of the sensitive slice (Perlo et al, 2004). Magnets with inhomogeneities, such as those encountered in Halbach magnets and even more so with stray-field magnets like the NMR-MOUSE should be shimmed by incorporating additional small permanent magnets, as electric shims would require excessive currents (Perlo et al, 2007). Preferably the shim magnets are arranged with the same symmetry as the main magnet, but with opposite polarity. By adjusting the positions of the magnets, the field inhomogeneities can be eliminated to different orders of accuracy, depending on the number of shim magnets. This concept has been applied to the NMR-MOUSE to arrive at a volume external to the magnet with a homogeneity sufficient to achieve high resolution in $^1$H NMR spectra (see below) (Perlo et al, 2007) and to homogenize the field distribution within a Halbach magnet for imaging objects with diameters up to 30 mm (Fig. 5) (Danieli et al, submitted). The resulting magnet has a figure of merit of $Q \approx 10^{-3}$ defined as the sensitive volume for imaging (10 ppm homogeneity) over the total volume of the magnet. This compares to $Q \approx 10^{-3}$ (4 ppm homogeneity) for medical imagers and $Q \approx 10^{-6}$ (1 ppm homogeneity) for high-field NMR spectroscopy magnets. To increase $Q$ is a central issue in the design of sub-compact NMR magnets for small NMR machines (Blümich et al, 2008a).

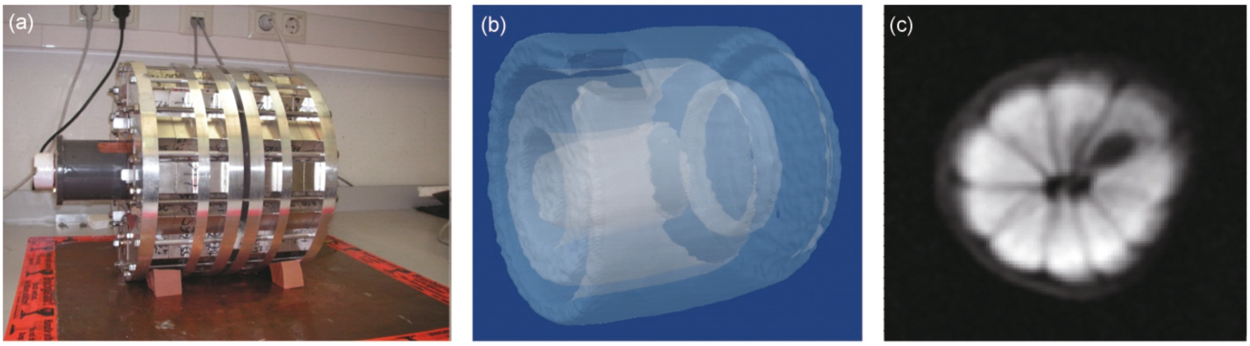

**Fig. 5** (a) Halbach magnet with shims that generate a volume homogeneous within 2 ppm field variation in a 30 cm$^3$ volume for imaging; (b) 3D surface-rendered image of water filled beakers stacked inside each other. An air bubble is visible in the outer water shell. The image was reconstructed from ten 2D slices. The dimensions of the outer water shell are 28 mm diameter and 50 mm length. The total acquisition time was 20 seconds; (c) 2D slice-selective image through a small lemon consisting of 64 × 64 pixels. The acquisition time was two minutes

# 6 Spectroscopy

Magnets for NMR spectroscopy place the highest demands on field homogeneity and field stability. The concept of shimming by adjusting the positions of permanent magnet blocks within the array of magnet blocks that generates the main magnetic field has been elaborated by example of stray-field NMR spectroscopy (Fig. 6(a)) (Perlo et al, 2007). Here the container with the fluid to be measured is placed on top of the magnet, so that the sensitive volume is located within the fluid (Fig. 6(b)). This device has a high figure of merit *Q* of $10^{-4}$ at 0.25 ppm homogeneity and thus high sensitivity. As the field is homogeneous, the NMR spectrum (Fig. 6(c)) is measured in the conventional way by simple pulse excitation with the exception, that the excitation pulse is applied in the presence of a gradient field to selectively excite the region of homogeneous field. While this measurement provides proof of principle that contrary to common perception, NMR spectra can be measured in a homogeneous part of the stray-field of magnets, temperature-induced field drifts need to be addressed before routine use of such a magnet. A way to achieve this is by constructing the overall magnet from individual magnet blocks with different temperature coefficients and arranging the blocks in such a way, that the net field drift is sufficiently suppressed, while the field homogeneity is maintained.

A technologically more relevant use of this concept is in the design of sub-compact magnets for NMR spectroscopy. Batteries of such magnets can be employed for high-throughout analysis by placing the analyte into the sensitive volume by means of capillaries using micro-coils for excitation and detection (Sillerud et al, 2006). This is a more powerful approach than relaxation analysis to identify cells marked by ferromagnetic nano-particles (Lee et al, 2008) as the chemical shift provides higher resolution for sample identification than the relaxation scale. By using capillaries, the demands on magnet perfection are reduced as the sensitive volume is minimized and small figures *Q* of merit suffice for spectroscopy with magnets the size of a coffee cup or even smaller.

When applying the concept of shimming with permanent magnets to coffee-cup-size Halbach magnets, *Q* can be increased, so that standard 5 mm sample tubes can eventually be employed. An encouraging result on the way towards this goal is documented in Fig. 7 (Blümich et al, 2008b). The NMR spectrum of toluene depicted in Fig. 7(b) has been

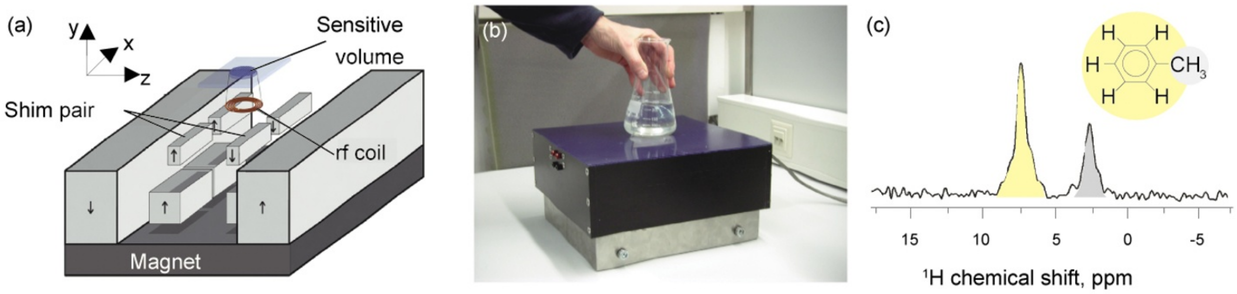

**Fig. 6** (Perlo et al, 2007) (a) Stray-field NMR magnet with shims for ex situ NMR spectroscopy from a sensitive volume external to the magnet; (b) Photo of the set-up for measuring the NMR-spectrum of toluene contained in a flask positioned on top of the magnet; (c) $^1$H NMR spectrum of toluene at 0.25 ppm resolution acquired from a 12 mm$^3$ sensitive volume at 8.33 MHz within 60 s

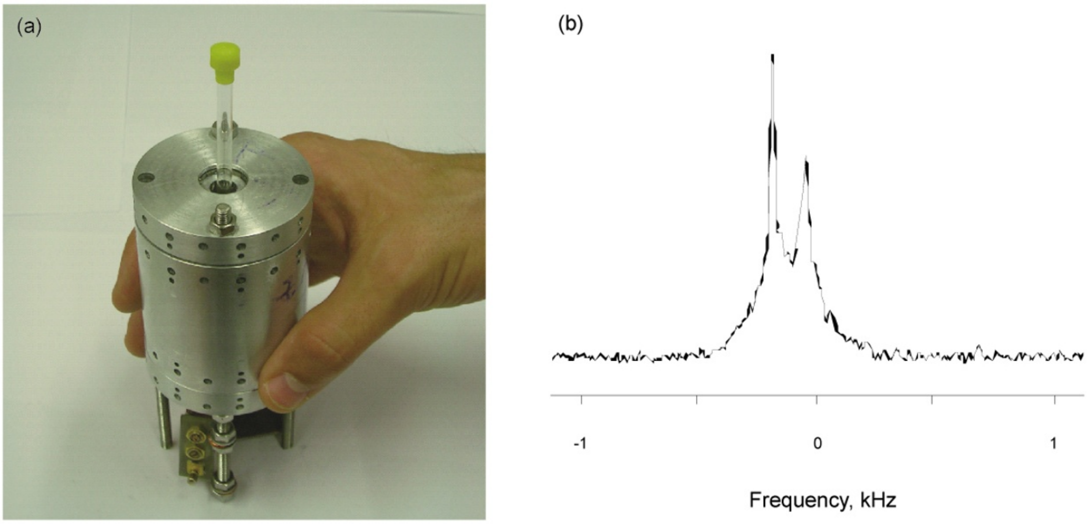

**Fig. 7** (Blümich et al, 2009) (a) Sub-compact Halbach magnet with shims from small permanent magnets designed to operate with a conventional 5 mm sample tube; (b) 30 MHz $^1$H NMR spectrum of toluene measured in this magnet with a 2 mm diameter sample tube. The current spectroscopic resolution is 1 ppm and a figure of merit of $Q \approx 10^{-4}$

measured at 30 MHz with the sub-compact magnet shown in Fig. 7(a) using a sample tube of 2 mm diameter. The field homogeneity needs to be improved by further iterations of the shim magnet displacements. To this end, the precision with which the shim magnets can be displaced is currently being increased. Such magnets could be used for example for chemical analysis of oil and drilling fluid down-hole inside a well logging tool.

## 7 Summary

Mobile NMR requires small magnets to be carried along to the site of the investigation. Open stray-field magnets are traditionally used in well logging. Smaller versions lead to slim-line logging tools that can be employed in narrow bore holes to study wetting and drying processes in the unsaturated soils and rock formations of the vadose zone. By appropriately shaping the sensitive volume external to the sensor into flat or curved slices, stray-field NMR sensors can be used for materials analysis. For example, the Profile NMR-MOUSE exhibits a thin and flat sensitive volume suitable for high-resolution depth profiling. Stray-field NMR sensors pose no limitations on the object size. But closed magnets, such as Halbach magnets produce a homogeneous field with a volume larger than that of a stray-field sensor and at higher field than a stray-field sensor. Consequently, closed magnets are preferred for the construction of small, sub-compact NMR machines applicable to samples small enough to fit inside the opening of the magnet. Either magnet type, open or closed can be shimmed to arrive at a homogeneity sufficient to measure $^1$H NMR spectra, for example, of oil down-hole in the well.

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