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Low-field and benchtop NMR

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ABSTRACT

NMR started at low field. Important discoveries like the first observation of NMR in condensed matter, the spin echo, NMR for chemical analysis, Fourier NMR spectroscopy, 2D NMR spectroscopy and magnetic resonance imaging happened at field strengths considered low today. With time the footprint of the NMR instruments at these field strengths shrunk from the laboratory floor to the tabletop. The first commercial tabletop NMR instruments were compact relaxometers for food analysis followed by mobile relaxometers for materials testing and oil-well exploration culminating in tabletop spectrometers for chemical analysis, capable of performing nearly the whole methodical portfolio of today's high-field instruments. The increasing sensitivity afforded by the lower noise of modern electronics and the unfolding richness of hyperpolarization scenarios along with detection schemes alternative to nuclear induction enable NMR at ultra-low field strengths down to zero applied field, where spin-spin couplings in local fields dominate the residual Zeeman interaction. Miniaturization and cost-reduction of NMR instruments outline current development goals along with the development of smart-phone-like apps to conduct standard NMR analyses.

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1. Early NMR instruments

The first magnets employed to detect NMR in condensed matter were electromagnets [1–3]. Following the discovery of the chemical shift [4,5], spectrometers with more stable permanent magnets were constructed [6]. ^{1}H NMR reference spectra were first collected at 20 MHz [7]. Eventually the ^{1}H resonance frequency could be pushed to 90 MHz with water-cooled electromagnets. Such magnets were a natural choice, because NMR spectra were measured routinely by forced oscillations in field-sweep mode at a fixed transmitter and receiver frequency. Their delicate water cooling could be avoided with permanent magnets fitted with frequency sweep coils. The Varian line of 60 MHz spectrometers, the A-60 (Fig. 1a) [8] and the EM-390 with permanent magnets, introduced in 1961 and 1975, respectively, were the chemist's workhorse from the sixties to the eighties [9,10]. Pulsed NMR was developed with low-field instruments, i.e. the inversion recovery experiment in 1949 [11] and the NMR echo in 1949 [12] by Erwin Hahn as well as Fourier NMR spectroscopy in 1966 by Richard Ernst and Weston Anderson [13]. Even the first 2D NMR spectra were measured at 60 MHz on a modified Varian spectrometer with a permanent magnet [14]. Still in 1980, IBM launched a series of 80 MHz Fourier NMR spectrometers with table-size permanent magnets [9]. In fact, in the seventies, NMR at 90 MHz was conventional NMR and not low-field NMR. While NMR with superconducting magnets in those past days was called high-field NMR, today high-field NMR is the standard, and NMR below 100 MHz is low-field NMR.

High-field superconducting magnets were first developed in the 1960ties by Varian, but their commercial success only started in the early 1970ties with the introduction of Fourier spectrometers by Bruker beginning at field strengths as low as 100 MHz for protons [9]. Following the advances in superconducting magnet technology the field strength further increased and with it the sensitivity and chemical-shift dispersion of NMR spectroscopy. In parallel, NMR spectrometers with permanent magnets and electromagnets became obsolete, because their field strength could not be pushed far beyond 90 MHz ^{1}H frequency. Eventually NMR at this and lower field strength became known as low-field NMR. Nevertheless, research and applications did not become obsolete but rather gained momentum in the recent past through the development of commercial mobile and tabletop NMR instruments as well as through the research-driven access to NMR signals from ultra-low fields by hyperpolarization and advanced detection methods [15–21].

2. Benchtop relaxometry

As spectroscopy conquered higher and higher fields and permanent magnets and electromagnets became excluded from

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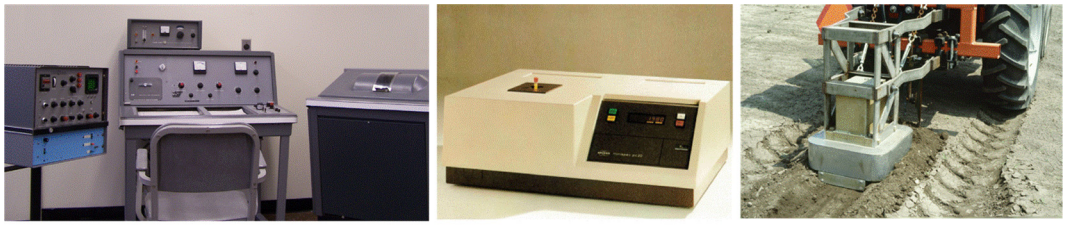

a) b) c)

Fig. 1. Historic low-field NMR instruments. (a) The Varian A-60 CW spectrometer introduced in 1961 [8]. (b) The Bruker minispec pc20 from 1980 replaced the p20i relaxometer from 1973 and the minispec p20 from 1972 [33]. The old minispec shown has much improved over the years since then. (c) Stray-field relaxometer for measurement of soil moisture [63].

main-stream NMR, the innovation in commercial low-field NMR went into making the magnet and the spectrometer electronics smaller to arrive at more compact NMR instruments [22–32]. Compared to water-cooled electromagnets, permanent magnets are maintenance free. Also, their energy density is higher so that permanent magnets can be made smaller than electromagnets. But with the magnet size decreasing at constant sample size, it becomes increasingly more difficult to produce magnetic fields sufficiently homogeneous to resolve the chemical shift. Consequently, the first compact NMR instruments employed permanent magnets with moderately homogeneous fields good enough to excite the sample volume non-selectively but insufficient for high-resolution liquid-state ^{1}H spectroscopy. They served to measure relaxation and diffusion but not chemical shift.

For a long time, relaxation and diffusion NMR signals were exclusively analyzed in the time domain by fitting model functions to experimental signal-decay and build-up curves in order to extract the relaxation times and self-diffusion coefficients. Consequently, this type of NMR became known as time-domain NMR. In 1972 Bruker started marketing the Minispec® p20, a compact tabletop relaxometer (Fig. 1b) [33], and not much afterwards, Oxford Instruments followed with a related permanent-magnet based tabletop instrument, the Newport Analyzer, after acquiring Newport Instruments in 1975 [29,34]. This instrument was subsequently succeeded by the MQC series of relaxometers. Tabletop relaxometers are still an important item in the portfolio of both instrument manufacturers and can be acquired with pulsed field gradients to enable flow and diffusion studies. The Minispec was specifically developed to determine the solid-fat content, and the droplet-size distributions in food products [35]. Subsequently many other industrial applications relying on relaxation, translational diffusion, and multi-quantum filtering were developed which do not require spectroscopic resolution for signal detection [28]. They are applied not only in the context of food and agriculture [36,37], but also to understand the morphology and ripening of cement and concrete [38,39] the properties and morphology of polymer materials [40–45], the wettability and aging of rock and tight sand [46–48], and even the physico-chemical properties of molecules in mixtures forming complex fluids [49–51]. Moreover, the rheological properties of fluids can be studied, and flow rates be measured [52–54]. A unique niche application of compact relaxometers is the quantification of biomarker molecules by means of the relaxation properties of functionalized nanoparticles, which specifically bind to the disease markers [55–57]. Combined with microfluidic technology these relaxometers are pioneering bedside NMR and point-of-care NMR [30,31,58]. The most straight forward application of relaxometers is for detection of isotope concentrations, for example, to determine contaminations in marine fuel oil [59] and animal slurries [60]. By stepwise change of the resonance frequency, even wideline spectra of solids can be acquired in this very simple and robust way [61].

3. Mobile NMR

In the 1970ties NMR sensors with electromagnets and with permanent magnets were being explored at Southwest Research in San Antonio, Texas, for specific monitoring tasks. Applications ranged from detecting explosives in mail, landmines in the ground, the curing state of resins, and the moisture content in bridge decks, soil and buildings. A particularly noteworthy development was the construction of single-sided NMR devices, sometimes also referred to as one-sided access NMR instruments. Different versions were developed with electromagnets that had to be lifted with a crane [62], with permanent magnets that were pulled across a field with a tractor (Fig. 1c) [63], or were handheld looking like a cloth iron for materials, tissue and food analyses [64–68]. These instruments started the era of mobile NMR with permanent magnets.

Mobile relaxometers that operate in the earth's magnetic field were considered by Russell Varian already in 1952 to be of interest to the oil industry just a few years after the discovery of NMR in condensed matter [69]. Prototype well-logging earth-field relaxometers were built in the 1960ties following the observation that the ^{1}H T_1 relaxation times correlate with the viscosity of crude oil [70]. But well-logging instruments were commercialized only in 1995 once permanent magnets became part of their design [71]. Stray-field magnets in well-logging NMR instruments raise the thermal polarization above that of the earth's field, and their gradient enables placing the detection volume into the borehole wall by the principle of slice selection known from magnetic resonance imaging (MRI), so that information about the rock formation and the type of fluid residing in the rock pores can be obtained from inside the rock formation. The magnet and the electronics are packed into a tube that is inserted into the borehole to measure diffusion-weighted relaxation signals from the borehole wall outside the instrument. Because the magnet is inserted into the object and not the sample into the magnet, well-logging NMR is also called inside-out NMR. While the original wireline logging tools that were pulled up the borehole have advanced to logging-while-drilling tools, a new generation of tools capable of radial imaging is currently being developed [72–75], and concepts for down-hole fluid analysis by NMR relaxometry are being investigated [76] based on multi-dimensional Laplace NMR correlation maps which can rapidly be acquired with sparse-sampling strategies [77].

Well-logging NMR along with porous media studies [78–81] are the main drivers advancing the methodology of time-domain NMR. Porous media are, in fact, studied best at low magnetic field, because differences in magnetic susceptibility between pore matrix and pore space lead to distortions of the magnetic field on the pore scale. In the literature these field distortions are summarily addressed by the notion of internal gradients although the resultant local field variations are not necessarily linear. Well logging instruments usually operate at ^{1}H NMR frequencies of 2 MHz

and below where the impact of internal gradients on the decay of the transverse magnetization from fluids diffusing in the pores can be suppressed at short echo times. Many laboratory studies of fluids and gases in rock cores are conducted at this frequency as well [73,82,83]. Particular noteworthy is the growing use of inversion algorithms reminiscent of the inverse Laplace transformation [84] to convert time-domain signal-buildup and -decay curves to distributions of exponential-relaxation times and diffusion coefficients, so that time-domain NMR is nowadays often referred to as Laplace NMR in harmony with Fourier NMR describing the processing of spectroscopic and imaging NMR data by Fourier transformation of the response to pulsed excitation [81]. A particularly powerful outcome of this widening understanding of standard time-domain NMR is two- and multi-dimensional Laplace NMR [81,85], which generates correlation maps of distributions of relaxation times and diffusion coefficients in close analogy to multi-dimensional Fourier spectroscopy [14] albeit with only up to five lines per dimension. Yet these types of experiments provide a wealth of insights into heterogeneous multiphase objects [81].

Various designs of compact stray-field sensors have been developed [68,86–93] and new methodologies and technical applications continue to be explored [89,94–99]. The NMR-MOUSE is a compact stray-field NMR sensor, which other than its predecessors [64] and most other NMR sensors was originally designed without attention to a low gradient of the stray field, resulting in a smaller size and thinner signal-bearing volume [100]. This and similar sensors find applications for nondestructive testing of various proton-rich materials and products like polymer products, food, biological tissue, and objects of cultural heritage [27,68,92,101–104]. In the course of time the sensitive volume of the NMR-MOUSE was flattened into a slice with the area of a 10 Euro-cent coin down to less than 10 μm thick [105]. A related C-shaped permanent magnet with slightly more confined access is the GARFIELD magnet [106]. When the NMR-MOUSE is mounted on a precision lift, high-resolution depth profiles corresponding to 1D images can be acquired with it pixel by pixel by changing the distance between the sensor and the object step by step and acquiring CPMG-like multi-echo trains at each position. From the acquired multi-echo signals the profile amplitude is derived in terms of different contrast parameters. Moreover, the sensitive slice can be positioned inside thin strata like paint layers to study their properties, for example, the solvent uptake when cleaning master paintings with solvents [101,107,108].

By increasing the number of magnet elements and displacing them in a controlled manner, the stray-field could be shimmed to a uniform gradient in a limited volume distant from the sensor surface, so that the Fourier transform of an echo provides a frequency encoded depth profile in a single shot [109]. Further refinement allowed to shim the stray-field locally even to a degree of homogeneity sufficient to resolve the ^{1}H chemical shift from a selected volume within a liquid placed in a beaker on top of the sensor [110]. Playing with the stray-field of the NMR-MOUSE in this way provided the skills to shim Halbach magnets [111,112] to better than 10^{-8} homogeneity across the diameter of a conventional NMR sample tube by displacing individual magnet elements [113,114].

4. Field cycling

Filed-cycling NMR experiments aim at understanding the molecular dynamics as a function of the NMR frequency [115]. The primary NMR parameter of interest for that is the longitudinal relaxation time T_1. Different motional mechanisms are probed by matching the resonance frequency to the relaxation rate. This requires tuning the evolution field during T_1 relaxation to the chosen Larmor frequency of the observe nucleus. But to achieve optimum detection sensitivity, the strength of the detection field should be high. Both demands can be met by conducting the measurements in a two-dimensional fashion either by shuttling the sample between different fields or by switching field strength during the experiment [116]. Slow motions are particularly interesting in order to understand the dynamics of macromolecules in the melt and complex liquids in the confinement of pores such as hydrocarbons in shale [117–120]. Longitudinal relaxation at low magnetic fields is measured in field cycling experiments employing electromagnets with which the field strength can be changed rapidly within times much shorter than the relaxation time [121]. The sample is typically polarized in a preparation period at high field, the polarization relaxes during a variable evolution period at low field, and the remaining polarization is recorded in terms of transverse magnetization at high field during the detection period as a function of the evolution period. In field cycling experiments 'high field' means a field strength of about 1 T, which is considered low for most NMR spectroscopists as well as in the context of this review. With appropriate shielding, relaxation can be probed also at magnetic fields lower than the earth's field [122]. Moreover, the detection sensitivity can be enhanced with hyperpolarization techniques such as DNP to unmask otherwise hidden components [123].

5. Magnetic resonance imaging

For magnetic resonance imaging (MRI) the requirements for field homogeneity are less stringent than for high-resolution spectroscopy [28]. MRI used to be the most important application of low-field NMR, because the standard field strength was 0.5 T, where thermal polarization is sufficient to provide good-quality images [124,125]. Even today, many clinical MRI instruments in use would be considered low-field instruments from an NMR spectroscopist's point of view although they employ superconducting magnets, because their ^{1}H NMR frequencies are below 100 MHz. For narrow-bore MRI magnets, field strengths up to 2 T can well be generated with compact permanent magnets [23,24] suitable for field applications [126] to inspect plants, roots, and agricultural products [127–131], to study transport phenomena and reactions in chemical engineering [132], phenomena encountered in enhanced oil recovery [133,134], and even to scan the human head in an emergency vehicle or in remote regions of human civilization [135]. Open, openable and dedicated MRI magnets have been developed to study trees and plants in the green house and in the field [136–138]. Image acquisition times and information content are optimized following strategies of undersampling and compressed sensing which take advantage of prior information and statistical principles [139,140].

6. Benchtop spectroscopy

As opposed to C-shaped permanent magnets, Halbach magnets are cylindrical with a field direction transverse to the cylinder axis and ideally zero stray-field outside [24,26,28,111,112,141]. Their geometry favors the cylindrical sample tubes common to most NMR spectroscopy experiments. These permanent NMR magnets are assembled from several magnet blocks with dimensions, magnitude, direction and distribution of their polarization varying in the percent range. This is why permanent NMR magnets are difficult to design on a computer and to shim to homogeneity better than 10^{-8} across the sample volume as needed to resolve the ^{1}H chemical shift and J multiplets from liquids. Moreover, they are sensitive to temperature variation. The brute-force approach is to produce magnets with a large ratio of magnet volume over sample

volume, whereby the volume of a robust permanent NMR magnet includes the thermal insulation. This approach was used in the past leading to table-size permanent magnets for NMR spectroscopy.

The first small permanent magnets suitable for high-resolution NMR spectroscopy were scaled-down versions of large magnets at similar ratios of magnet volume over sample volume, whereby, like in the early days of NMR spectroscopy, the position of the small sample had to be identified experimentally as the region of highest field homogeneity [142]. Subsequently different strategies to expand the homogeneous field region in permanent magnets were found by incorporating adjustable elements and pole shoes in the magnet assembly [24,113,141,143–146] as well as novel principles of active shimming [147,148], so that compact NMR spectrometers providing chemical shift resolution became commercially available as of about 2010. Prototypes have been tested before and evaluated in particular for reaction monitoring and process control applications to replace floor-size low-field NMR spectrometers for process control [149,150].

Today low-field NMR spectrometers are sometimes referred to also as low-resolution spectrometers. Then 'low resolution' refers to the frequency range of the chemical shift, which scales with field strength, because in terms of linewidth high-end benchtop NMR spectrometers can well compete with high-resolution high-field NMR spectrometers even though the sample is not spun to average out the residual field inhomogeneity of the compact magnet (Fig. 2a) [151]. This along with an external lock and maintenance-free operation makes compact NMR spectrometers attractive not only for rapid-access chemical analysis of liquids including hazardous solutions on the workbench and in the fume hood of the chemistry laboratory but also for high throughput scenarios encountered in monitoring applications to flow chemistry, studies of reaction mechanisms, process control, and chemical discovery [15,16,28,29,152,153]. Although their field strengths are lower than those of the high-field spectrometers common today, benchtop NMR spectrometers are better suited for chemical analysis than their bulky predecessors from 40 years ago, which operated at similar field strengths, thanks to superior electronics and the advanced methodology. In fact, their sensitivity is sufficient to analyze metabolites in body fluids [154] and to serve as a chemical detector in size-exclusion chromatography [155]. When employed for detection of hyperpolarized substances [156,157], their low field strength may become an issue negligible to the cost difference, as spectra of complex mixtures are simplified by the selectivity of the hyperpolarization process and the sensitivity is more and more determined by the degree of hyperpolarization.

7. NMR at ultra-low fields

NMR spectroscopy is also of interest at ultra-low field and even zero field [158–160]. Half a century ago, when to NMR at 1 T to 2 T field strength was considered to be high-field NMR, low-field NMR was NMR at the earth's magnetic field [161–163]. The earth's field was first explored in NMR by relaxometers probing crude oil in well logging applications [69,70] and by magnetometers [164] determining the strength and orientation of the earth's field, for example in search for buried cultural artifacts (Fig. 2b) [165]. Early on, the signal was enhanced by prepolarizing the sample at higher field or by Overhauser DNP [166]. DNP enhancement was also employed to produce the first earth-field 2D NMR spectra [167]. Further studies concerned the analysis of Antarctic sea ice [19,168] and imaging of the human body [169]. A particular intriguing application is the localization and the contamination of groundwater by earth-field NMR with surface coils up to 100 m in diameter [170], and more recently the identification of oil spills below the sea-ice cover by an NMR apparatus suspended from a helicopter [171].

For a long time, the notion persisted, that the chemical shift is too small to be detected in the earth's magnetic field, and the only chemical information obtainable was the hetero-nuclear *J*-coupling. However, not only can large chemical shifts like those of Xenon be detected [172] but also homonuclear *J*-couplings if the magnetic equivalence of two protons, for example, is broken by the coupling to a heteronucleus like ^{13}C, so that chemical structures can be analyzed by earth-field spectroscopy [173,174].

From the point of spectroscopy, ultra-low fields begin in the region, where the indirect coupling starts to be comparable to and stronger than the Zeeman interaction [174]. This field strength can be below the strength of the earth's magnetic field. At zero applied field, the local magnetic fields arising from the electrons orbiting the nuclei in the molecules determine the appearance of the NMR spectrum. Moreover, the anisotropy of the spin interactions encountered in solids vanishes so that narrow lines can be obtained for chemical analysis of solids by high-resolution NMR spectroscopy [20,175–177]. The information from the Zeeman

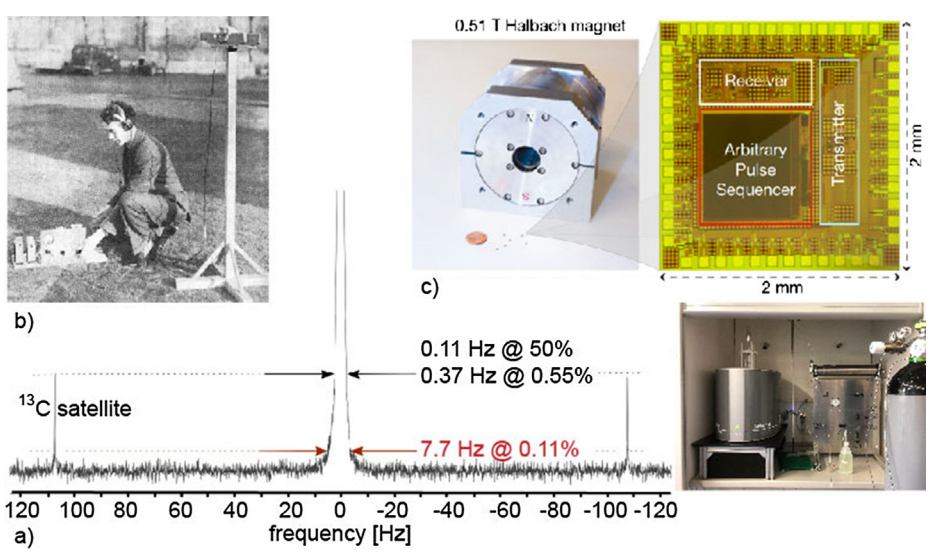

Fig. 2. Compact NMR instruments. (a) ^{1}H NMR line-shape of a state-of-the-art tabletop NMR spectrometer (left) [151] and spectrometer inside a fume hood set up for high-pressure gas NMR spectroscopy (right). (b) Earth-field magnetometer operated by "Mr. P. Mansfield". The signal is collected from a water bottle (top right; from [165], Fig. 7; copyright J.G. Powles). (c) Single-chip prototype NMR spectrometer (reprinted with permission by PNAS [32], Fig. 1).

interaction about the identity of the nuclear isotope is lost but chemical information from isotropic spin-spin couplings is preserved, so that the chemistry of fluids in pores can still be identified by spectroscopy at zero field where internal gradients are absent [178].

Without the need for superconducting magnets, the instrumentation for zero and ultra-low-field NMR becomes less expensive than high-field instrumentation. But other challenges need to be met [179]. These are in particular the vanishing thermal polarization and the impact of the earth magnetic field and stray magnetic fields from various sources, which need to be well shielded at high homogeneity of the residual field. Moreover, at ultra-low polarization, nuclear induction fails for signal detection, and field cycling for pre-polarization, hyperpolarization [21], and alternative detection methods, most importantly, optical detection, need to be employed [159,160,180,181]. From a scientific point of view, low and ultra-low fields are interesting, because long-lived homo- and hetero-nuclear singlet states can be populated at ease [182], and hyper-polarization methods from singlet hydrogen molecules (p-H_2) work best in that regime [183–186]. Also, NMR ultra-low-fields opens a new window to scientific discovery [154,187]. From a technical point of view, ultra-low fields are interesting, because the magnet weight and size are reduced to the weight and size of the shield, so that NMR instruments become smaller and mobile. An application where this may become commercially viable is mobile MRI with hyperpolarized chemical marker molecules as thermally polarized MRI already produces useful brain images at 6.5 mT [188]. These features make ultra-low-field NMR a field of growing interest to scientific research.

8. Miniaturization and outlook

The largest component in an NMR spectrometer today is the magnet followed by the rf amplifier. All other components including the probe can be made small and have already been miniaturized to a single chip smaller than a thumb nail [189,190]. Prototype miniature NMR spectrometers have been built and demonstrated to be capable of producing 2D spectra from liquid samples trapped in capillaries (Fig. 2c) [32]. The principle difference to miniature relaxometers [191,192] is not the electronics but the field homogeneity of the compact magnet. Current efforts, therefore, focus on improving the passive and active shim strategies of permanent magnets [26,141]. Moreover, low-power broad-band excitation can eliminate the need for bulky radio-frequency amplifiers [193–196]. Because the footprint of NMR spectrometers has been reduced from the laboratory floor to the tabletop in the last decade, it does not take much to envision that the future will bring even smaller NMR spectrometers, perhaps smaller than a coffee cup, with field strengths up to 1 or 2 T [25,29,197]. If these become a commercial success will depend on the need for them. Given that small spectrometers will be less expensive than large ones, they need to become a commodity produced in large numbers. A potentially widespread application of miniature NMR spectrometers can be in the life sciences, for example for fingerprinting the metabolites in body fluids like blood at a doctor's office or of urine and saliva at home to track the performance of the human body in terms of a chemical reactor converting food to energy [29,153]. Their operation should be guided by apps from tablet PCs or smart phones, and the raw data could be transmitted in wireless networks for analysis and communication of results. Acquisition times of 10 min are reasonable, and metabolites can be detected at 60 MHz in urine [140]. If metabolite fingerprinting at 60 MHz proves good enough compared to high-field NMR spectroscopy to detect developing disease at an early stage, the regular use of personal miniature NMR spectrometers at home for metabolite fingerprinting could enable the turning of medicine from curative to preventive by moving the analytical laboratory from a central facility into the homes of the users.

A more imminent field of application is the use of compact NMR spectrometers as sensors. The information content of the different types of NMR spectra is far richer than that which competing analytical methods like mass spectrometry, gas chromatography and infrared spectroscopy can deliver, even if their mass sensitivity is higher. Already today tabletop NMR spectrometers are explored for reaction monitoring, process control and as detectors in robots for chemical discovery [153]. Decreasing their size further will cultivate a widening range of new applications, where miniaturized NMR spectrometers serve as dedicated chemical sensors in industrial processes driven by the need for increased precision and automation [32]. In this regard, also ultra-low field NMR bears intriguing potential for further miniaturization of chemical sensors in combination with sensitivity enhancement by hyperpolarization and optical detection [159,181].

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