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Designing and building a low-cost portable FT-NMR spectrometer in 2019: A modern challenge

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Résumé: La RMN à transformée de Fourier (RMN FT) et à hauts champs est une technique spectroscopique très performante et incontournable dans de nombreux domaines analytique. Le caractère non destructif de la RMN en fait un moyen privilégié pour analyser les milieux en chimie et en biologie. Les spectromètres RMN à TF compacts, dits aussi de paillasse, sont des alternatives, à bas coût, aux spectromètres classiques hauts champs et haute résolution. Un laboratoire de recherche peut vouloir développer son propre spectromètre RMN compact ("Benchtop Home-Built NMR") à un coût très réduit (~10 k€). Mais pourquoi? Tout d'abord, pour l'utiliser de façon ponctuelle comme canal supplémentaire (noyau X) à un spectromètre haute-résolution, mais aussi pouvoir le coupler à d'autres instruments de Physique, comme par exemple à un microscope optique, pour l'étude de la diffusion de spin dans les semi-conducteurs. Par ailleurs, une RMN « Home-Built » peut être utilisée avec un aimant permanent bas champ pour la quantification d'espèces qui ne nécessite pas nécessairement une haute résolution, évitant le besoin de services cryogéniques hebdomadaires et coûteux. Hors laboratoire de recherche, cette RMN portable peut être utilisée pour l'analyse *in situ* de milieux naturels en extérieur. Enfin, ce spectromètre compact est naturellement dédié à l'enseignement de la spectroscopie RMN, en étant ouvert à l'étude des fonctions électroniques de base constituant l'instrument. La question principale qui se pose alors est: Comment construire aujourd'hui « un spectromètre "maison"? Nous décrivons dans cet article, la réalisation d'un spectromètre RMN basé sur des composants et des cartes électroniques (LNA, ADC, FPGA, ARM, DDS ...) accessibles commercialement, et permettant d'obtenir une RMN de paillasse présentant à la fois une grande dynamique d'acquisition et de bons rapports signal/bruit.

Mots clés: RMN à TF, Benchtop, Bas coût, Electronique. **Erreur ! Référence de lien hypertexte non valide.**

Summary: High-field FT-NMR is a high performance spectroscopic technique that is essential in many analytical fields. The non-destructive nature of NMR makes it a preferred means of analyzing chemical and biological environments. Compact NMR benchtop spectrometers are low-cost alternatives to conventional high field and high resolution spectrometers. A research laboratory may want to develop its own compact FT-NMR spectrometer ("Benchtop Home-Built NMR") with a very low financial cost (~10 k€). But why? First of all, to use it punctually as an additional channel (nucleus X) to a high-resolution spectrometer, but also to be able to couple it with complementary Physics instruments such as an optical microscope to study spin diffusion in semiconductors, for instance. In addition, a "Home-Built" NMR can be used with a low-field permanent magnet for the quantification of species that does not necessarily require high-resolution, avoiding the need for weekly and expensive cryogenic services. Outside the research laboratory, this portable NMR can be used for the *in situ* analysis of outdoor natural environments. Finally, this compact spectrometer is naturally dedicated to the teaching of NMR technique and is open to the study of the basic electronic functions that constitute an NMR spectrometer. The main question then arises: How to build a robust "HomeBuilt" NMR? In this article, we describe the realization of an NMR instrument based on electronic components and boards (LNA, ADC, FPGA, ARM, DDS...) easily commercially available, and allowing to obtain a benchtop NMR instrument presenting both a high acquisition dynamics and a good signal-to-noise ratio.

Keywords: FT-NMR, Home-built, Benchtop, Low-cost, Electronics

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1. Introduction

Conceived and explored in the 1945s in physicists' laboratories [1-5], Nuclear Magnetic Resonance spectroscopy (NMR) is arguably nowadays one of the most essential tool for analyzing the structure of mineral, organic or biological molecular systems. This fantastic achievement was possible with the development of Fourier-Transform NMR (FT-NMR) proposed by R.R. Ernst from [6], that in turn opened the door of first NMR experiments based on sophisticated multi-pulse sequences [7, 8]. Concomitantly, the application of NMR principles/concepts has also offered fundamental opportunities for medical imaging for robust diagnostic purposes. Behind the vast practical aspects of

analysis of matter, NMR basics underlies varied and sophisticated theoretical concepts in physics (data sampling), mathematics (FT principle), electronics (resonating circuit) or quantum mechanics (Hamiltonian, operator density, ...), as nicely described in many theoretical/practical books that are references for any NMR spectroscopists or experienced users [9-16].

As a brief reminder, **Table 1** provides a chronological summary of the main technological/methodological advances from the 1950s to the present days, in conjunction with the magnetic field strength for a continuous enhanced spectral resolution and sensitivity.

Table 1. Historical overview of main instrumental/methodological achievements

NMR methodology	CW-NMR			FT-NMR									
				1D		2D homonuclear		2D heteronuclear gradients		3D, 4D, TROSY ...		FAST NMR	
				Supraconductor magnet			Ultra shielded magnet, cryogenic probe						
Size of analyzable molecules (kDa)	< 10							25	35		50	100	
B ₀ (T)	0.70	1.41	2.11	4.22	6.34	9.39	11.74	14.09	17.61	18.78	21.13	22.32	23.48
¹ H Larmor (MHz)	30	60	90	180	270	400	500	600	750	800	900	950	1000
Microprocessors Intel family name				4004		8080			80490		Pentium 4		Intel Core i7
Number of transistors in microprocessors (Intel family)				2.3 × 10 ³		6 × 10 ³			1.2 × 10 ⁶	9.5 × 10 ⁶	42 × 10 ⁶	291 × 10 ⁶	1.1 × 10 ⁹
Years				1965		1975		1985		1995		2005	
	1950	1960		1970		1980		1990		2000		2010	

Unfortunately, the "Tesla race" has a significant financial cost (purchase) and periodic maintenance (cryofluids), and hence the need for cryofluid-free, FT-NMR spectrometers is becoming acute again. In addition, nowadays, the availability of rather small permanent magnets (soft iron) with a good magnetic homogeneity (up to 10⁻⁴) and stability as well as the significant improvement in electronic performance make it possible to offer very compact, versatile benchtop FT-NMR spectrometers at a reduced cost, making again this new generation of instrument very attractive for various applications in chemistry [17].

In this article, we propose to revisit the development and assembly of a very low-cost (~10 Keuros), portable (benchtop) FT-NMR spectrometer by integrating magnetic and electronic classical elements. For this, the essential units requested will be described, detailing their respective role in the excitation/detection process of signal, for instance. In a memorial approach in particular towards chemists that are generally forgotten the NMR principles, we will start from some (important) basic physical principles of NMR to organize and choose the key block elements necessary for the realization of this "artisanal" spectrometer or home-made, as anglicists would say. Interestingly, such a project is possible with laboratory instrumentation (function generator, oscilloscope, ...) accessible at a lower cost. All requested, specific electronic components/devices with their current nomenclature are listed in Appendix and are available in the market.

Proposing an inexpensive, home-made FT NMR instrument is above all an exciting task of engineering integrated into an educational project, for instance. It obviously cannot be considered as a competition with the new generation of FT NMR benchtop available in the market today from different

manufacturers (Magritek, Oxford, Thermofisher, Nanalysis, ...) which integrates many optional modules (locking, sample spinning, ...) [17]. Moreover, while the overall cost (material/electronic supplies) of the instrument seems attractive at first sight, it does not include the actual human costs (total cost), in particular in view of its pedagogical dimensions in Academic Education. This homebuilt NMR spectrometer can be seen as "alive" NMR support instrument for teaching NMR. Finally, this type of mobile NMR apparatus is well suited for scientific research by coupling NMR and another instrument (such as microscopy).

To have an idea, the time needed by a single engineer to build a one-channel mobile NMR made be estimated to two months, including the tests (and assuming that all electronic hardware components have been already purchased).

2. Contribution of modern electronics

Electronics has developed considerably in recent decades, making it possible to build high-performance systems with high functional integration possible today. To illustrate the evolution of the electronic component power, we have included the number of transistors integrated on the same chip as a function of years in **Table 1**. For example in 1965,

a microprocessor integrated nearly 2000 transistors on the semiconductor chip. The density of the components has increased by a factor of 10^6 in 45 years (1965 to 2010), thereby reducing the size of the electronic circuits. Currently a simple integrated circuit is capable of performing signal acquisition, processing and control functions. The modern smartphone is a blatant illustration of the high-tech concentrate contained in such a small volume. As the density of the components increases, the operating frequency of the hardware circuits has also increased considerably. Hardware can now operate at frequencies of several gigahertz compared to a few hundred kilohertz in the 1970s. These high working frequencies allow complex processes to be carried out in an extremely short time.

Another huge leap in the field of electronic equipment is the memory capacity of components. It is now possible to store and manipulate memory spaces of more than 64 gigabytes. Data storage is no longer a real limitation and it is possible not only to acquire large amounts of data, but also to store the ever more sophisticated programs to control and process this data.

The frequency synthesis is now digital based on DDS cards (Direct Digital Syntheses) over a very wide range of frequencies with very high accuracy. In **Table 2** (nomenclature of components) presented in Appendix, we list the references of a synthesis unit capable of generating frequencies from a few kHz to more than 300 MHz. Analogically, the sizes and costs of low-noise preamplifier components, mixers and other hardware have been reduced. For example, the low noise preamplifiers in the acquisition chain can be made with a few circuits for a hundred euros.

Finally, an important point to keep in mind is the reduction of the energy consumption of modern components, allowing complete battery-powered systems to be powered, making them autonomous and therefore portable, which was unthinkable in the 1980s. In summary, the performance of electronic components has increased while sizes and costs that have been reduced, so the realization of complex units such as a small NMR spectrometer is possible in research laboratories.

3. Some specificities of a Benchtop FT-NMR

Classical or benchtop/mobile FT-NMR spectrometer is a complex instrument, but it can be quite easily divided into separate, well-identified functional blocks [18]. In practice, this instrumentation involves many domains of electronics such as radiofrequency (RF), audio chain, power electronics, low-noise preamplification and transmission lines. In this first decade of the 21st century, FT-NMR spectrometers are fully digital and multi-nucleus. Gradually, they are connected by internet network, and can be therefore remotely controllable, and (unfortunately for experimenters) fully automated. The two operating modes of FT-NMR spectrometers (excitation and detection steps) are performed by fast microprocessors and executed by suitable softwares.

As a matter of principle, the channels (^1H , ^{13}C , X, ...) of spectrometers are similar but independent of each other, and all of them are synchronized by a master clock; a dedicated pulse sequencer controlling the complex multi-channel pulse sequences of the NMR spectrometer. This sequencer takes into account the different transmission delays and hardware dead times (such as the pre-scan delay) in order to produce a reliable and precise pulse timing controlled by the software. The duration of the (hard or soft)

electromagnetic pulses (μs to ms) and the delays between events (μs to s) are those actually achieved by the electronics, without the need to add small compensation delays in the pulse programs. These specificities are made possible thanks to the very high speed and stability of modern electronics.

The assembly of mobile NMR consists of three distinct parts: the magnet, the probe and the data acquisition and processing spectrometer. The magnet and the probe depend on the chosen application: low field or high field. We discuss these aspects later in this article.

The electronics of our mobile NMR spectrometer is designed to acquire very low value signals (less than μV) over a high dynamic range (the noise factor of the detection chain is less than 2 dB, the sampling adapted to the required dynamics (8, 12, 16 or 24 bits). The same applies to frequency synthesis and quadratic demodulation, which can be adapted to a very wide range of frequencies. Note that all the electronic hardware components (RF power amplifier, mixer, preamplifier, ...) have been chosen to work in a wide frequencies range (from 1 MHz to 400 MHz). This allow the portable FT-NMR to work at low field (with static permanent magnet) or at high frequency (with superconducting magnet).

4. Sensitivity and resolution

The homogeneity and strength of the magnetic field of a permanent magnet define the sensitivity and spectral resolution of any NMR experiment. The temporal magnetic stability of a magnet obviously limits the duration of the measurements. Homogeneity and magnetic stability must allow a coherent accumulation of signals in phase.

NMR is often defined as rather insensitive and high-field magnets needs to be used to achieve robust sensitivity and resolution to analyse complex molecules. In high-resolution NMR, chemical shifts must be measured with a resolution of about 0.01 ppm, in order to solve the J couplings of a few Hertz. Magnetic fields are of the order of several tesla and are generated by superconducting magnets cooled by helium and liquid nitrogen. A system of compensation for inhomogeneity (shims) is essential to obtain the required resolution.

Permanent magnets with low field ($< 0.94\text{ T}$ or 40 MHz for ^1H) are called low resolutions because of their low sensitivity and homogeneity. In an inhomogeneous field, the magnetic strength varies in relation of the spatial position in the magnet. Such effect leads to significant broadening or/and overlapping of resonance peaks on spectra, thus preventing exploitable spectral resolution. Nevertheless, the distributions of relaxation times, translational and quantitative diffusion coefficients of molecules are quite accessible at low fields. The characterization of materials (liquid, powder) can be carried out at low resolution with a home-made spectrometer that is much less cumbersome, less expensive in cryogenic maintenance, accessible because it is close to the synthetic bench of molecules. The latter reduces the cost and workflow of any research activity.

The mobile NMR spectrometer we are describing is part of this approach: it is compact and can be used with a low field magnet at Larmor frequencies ranging from 1 to 15 MHz in the set presented.

Although intended for low field, low frequency applications, as mentioned above, the design and the electronic components of our mobile NMR spectrometer can operate at frequencies up to 300 MHz (see the Table in appendix). As a result, this Mobile NMR spectrometer can also be used, without major modifications, at high field using the magnet and the probe part of the industrial spectrometer. In this type of application, the interest is methodological: development of a specific auxiliary channel, magnetization manipulation to control radiation damping, development of new strategies (multiple acquisitions).

5. Back to principles of NMR and practical consequences

The nucleus of atoms (^1H , ^{13}C , ^{15}N , ^{19}F , ^{31}P ...) is characterized by a quantum quantity, the spin which is specific to each nucleus and determines its magnetic properties. The magnetic moment, μ , of a nucleus is directly related to I by its gyromagnetic ratio, γ , which is a constant depending on the nature of the nucleus. The magnetic moment being quantified; the energy states are degenerated.

When this nucleus is immersed inside the static magnetic field, B_0 , the degeneration of energy level is lifted, leading to several energy levels accordingly to the spin value (Zeeman effect). If the nucleus is then subjected to a radiofrequency wave at a specific frequency (Larmor frequency), the atomic nucleus will absorb the energy of this radiation and release it during relaxation mechanisms. This process at the origin of the nucleus magnetic resonance phenomenon depends on the magnetic field and the properties of the molecules. The study and measurement of an isolated atom is not possible due to a lack of sensitivity of NMR, the result is that we are still dealing with a very large set of nuclear spins, the macroscopic result of which we will measure: magnetization. In the presence of a magnetic field B_0 , the resultant of macroscopic magnetization is collinear to B_0 at equilibrium, and proportional to the spin populations (number of nuclei).

Under the action of B_0 , the magnetic moment, μ , of any magnetically active nucleus ($I \neq 0$) is subjected to a force,

$$\vec{C} = \vec{\mu} \wedge \vec{B}_0, \quad (1)$$

which causes a spontaneous precessional movement around the z axis (\parallel to the B_0 axis) at the Larmor frequency (resonance condition) (see **Figure 1**),

$$\nu_{\text{Larmor}} = \nu_0 = -\gamma B_0 / 2\pi \quad (2)$$

where γ is the gyromagnetic ratio of the considered nucleus.

At equilibrium, all nuclei of a sample are subjected to B_0 . The resulting macroscopic magnetization, M_0 , is oriented along the Oz axis.

The first step of any FT-NMR experiment is to excite the spin system with a (generally short) RF pulses in order to remove it from its equilibrium state by tilting the M_0 magnetization in the xy plane (see **Figure 1**). The return of M_0 to equilibrium (denoted FID for Free Induction Decay) is then detected and processed by FT to obtain a frequency spectrum or expressed in part per million (ppm). The

magnetization M_0 observable for a set of spins nuclei I , corresponds to the following law:

$$M_0 = \frac{N\gamma^2 \hbar I(I+1)}{3kT} B_0 \quad (3)$$

where T is the sample temperature, N is the number of nuclei *per* unit volume, I is the spin of the nucleus, K is the Boltzmann constant and B_0 is the static magnetic field.

In practice, the magnetization and therefore the sensitivity of a NMR experiment is directly dependent on the value of B_0 . The exact magnetic-field dependence of the NMR sensitivity (considering the detection coil) can be written as the energy (E in Joule) detected in a coil of volume, V , namely:

$$E = M_0 \cdot B_0 \cdot V \quad (4)$$

where B_0 and M_0 are the static magnetic field and the magnetization per unit of volume, respectively.

A priori, it is therefore preferable to work with high magnetic fields, which are also at the origin of high-resolution NMR in liquids. For instance, at room temperature (300 K) and for a B_0 field of 11.7 T (500 MHz), the detectable magnetization is about 3×10^{-6} (in SI unit), or an energy of of about $10^{-4} \mu\text{J}$ for a sample volume V of 1 cm^3 ($E = M_0 \cdot B_0 \cdot V$). This corresponds to a reception power of about $1 \mu\text{W}$ for an acquisition time of 0.1 ms. As seen, the strength of detected signal is therefore very low. *De facto*, using a so-called low-field NMR, where the B_0 strength varies between 0.15 -1.5 T, makes the experimental NMR detection much more critical.

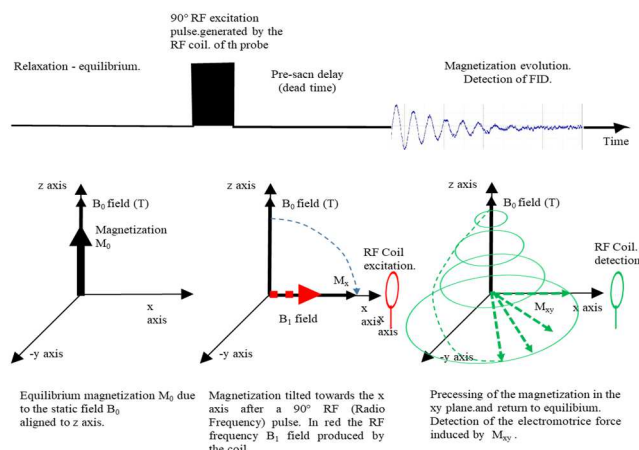


Fig. 1. NMR pulse and acquisition experiment. The macroscopic magnetization is aligned along the B_0 axis (Oz) during a relaxation delay. After an RF excitation pulse, M_0 is flipped in the Oxy plane. The precession of this magnetization give the impulse response (or FID) which is detected.

From electronic viewpoint, the voltage of detected signal (before its amplification) is of low amplitude with an order of microvolt (μV). In practice, the experimental conditions as well as the instrumentation electronics must therefore be optimized to reach acceptable signal-to-noise ratios (S/N). This challenge can be met thanks to the performance of

modern electronic components. In addition, the transmission power required to energize a frequency band of about 10 kHz (for the ^1H nucleus) for a 1 cm^3 volume sample is about 11 W (@500MHz or 11.7 T). For a low-field spectrometer, an excitation power of about 5 W for a pulse duration of about $20\text{ }\mu\text{s}$ is sufficient to generate an instantaneous tilting of 90° of M_0 , i.e. in the xy plane.

6. NMR instrumentation

6.1. The magnet. In the applications of this article, we use a permanent magnet that is sufficiently homogeneous and stable to provide consistent NMR measurements. Considering the analytical purposes of low-field NMR and its transportability, the weight of permanent magnet to be used must be resonable ($< 80\text{ Kg}$), and generate at least a field of 0.15 T (1500 Gauss), leading to a resonant frequency for protons around 6.4 MHz. The axis of the magnet is horizontal and

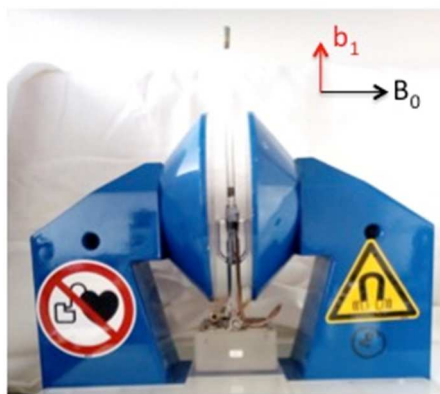


Fig. 2. Permanent horizontal magnet and its probe placed at the center of the magnet gap with a sample tube of 5-mm, inside.

perpendicular to the probe (coil) which consists of a vertical solenoid, and in which the sample is positioned (5-mm tube) (see **Figure 2**).

The NMR probe (the antenna) is carefully placed at the magnetic center of magnet in order to both excite (with a perpendicular field, B_1) and detect the signal along one of the four axes perpendicular to B_0 (x , $-x$, y , $-y$). The probe includes its frequency and impedance system (Tuning and Matching) that is connected to the electronic chain of the equipment. Notet that it is easy to add two external coils on either side of magnet in order to produce a field gradient that can potentially be used for possible monodimensional imaging applications. As the static magnetic field is not homogeneous over all the surface of the magnet, it is therefore necessary to accurately measure the value of B_0 Larmor frequency (ν_0). To this aim, a field map can be made within the permanent magnet in order to determine the exact using a gaussmeter (Teslameter) that is slowly moved into the air gap of the magnet. In our case, to a resonance frequency, ν_0 , of 6.28 MHz for the ^1H . the value measured at the center of the air gap is 1492 Gauss, which corresponds

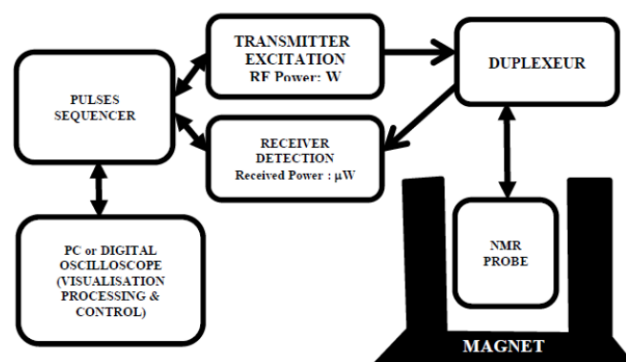


Fig. 3. Simplified "block" diagram of an NMR spectrometer.

6.2 Excitation versus detection. To perform FT-NMR experiments, three steps are necessary: i) create a homogeneous magnetic field B_0 ; ii) excite the spin system to tilt it from its equilibrium state by means of an excitation coil generating a B_1 field perpendicular to B_0 ; iii) detect the FID signal by means of a receiver coil (which is also the same generating the B_1 field excitation), the whole satisfying the Larmor's resonance condition, $\nu_{\text{exc}} = \nu_{\text{dec}} \approx \nu_0$.

The "block" diagram setting up the main parts of an NMR spectrometer is shown in **Figure 3**, which shows the permanent magnet and the NMR probe inside the magnet, an electronic "transmitter" block responsible for exciting the spin system with RF powers of several dozen watts, a second "receiver/detection" block that amplifies the FID signal of very low power (μW) and demodulates the signal in two components of low frequency (separate operations). These two steps can be controlled by a "programmable sequencer" that generates the multiple RF pulses trains and control the delays during the experiment. For very simple experiments (pulse-acquisition), a laboratory pulse generator is sufficient. Finally, data processing and visualization can be performed by a simple oscilloscope, but in a more modern way using a computer (PC) with its software interface. It should be noted that the probe is connected to the excitation and reception blocks via a unit called duplexer. This element can be seen as a signal "switcher" that aims to protect the preamplifiers of the detection channel from high RF excitation power. The duplexer directs all RF power to the probe during the excitation step (by closing access to the receiving channel) first, and then redirects the detected NMR signal to the preamplification/demodulation chain.

For a spectrometer operating at frequencies of the order of 15 MHz and with relatively low powers, a simple structure based on a pi (quarter-wave filter) can be used to perform this function. It is a design strategy for a very low-cost mobile NMR. First, the pi filter requires only a few electronic components at very low cost (self and capacities), capable of withstanding high powers (several Watts). In addition, it works automatically and there is no need to control it during acquisition, simplifying any pulse programs. The realization of the circuit is simple and fast. We will describe how to make a quarter-wave duplexer in Section 8 of this article for a frequency around 6.4MHz. Since the PI filter (or quarter-wave filter) is a circuit tuned to the Larmor frequency of the nucleus under consideration, its use is therefore limited to a small frequency band.

For wide band applications (*i.e.* frequencies from MHz to few hundred MHz), a duplexer based on fast RF electronics switches with fast diodes is mandatory. This type of duplexer is much more complex to implement because a pulse program within a microprocessor must control it. The description of such a circuit is out of scope for the version of the mobile NMR spectrometer we present in this article. We are targeting a low-cost mobile laboratory NMR with laboratory instrumentation, duplexer pi filter and low-field magnet operating at 0.15 T (or 6.4 MHz for ^1H).

6.3 The lock system. An electronic locking system may be easily add to makes the mobile NMR device capable to continuously control the stability of the magnet, in order to

avoid a slow shift of the magnetic field over time [see for instance, pp. 44 to 50 of ref. 19]. The lack of lock control can be particularly troublesome when work at high magnetic fields, and/or when recording long-term acquisition NMR experiments. Considering the aims (pedagogical aspects) and the application domains (1D NMR, detection of abundant nuclei, of such a spectrometer, no locking system has been integrated into the instrument presented in this article. *De facto*, but also to reduce the cost of this homemade instrument, no locking system has been integrated.

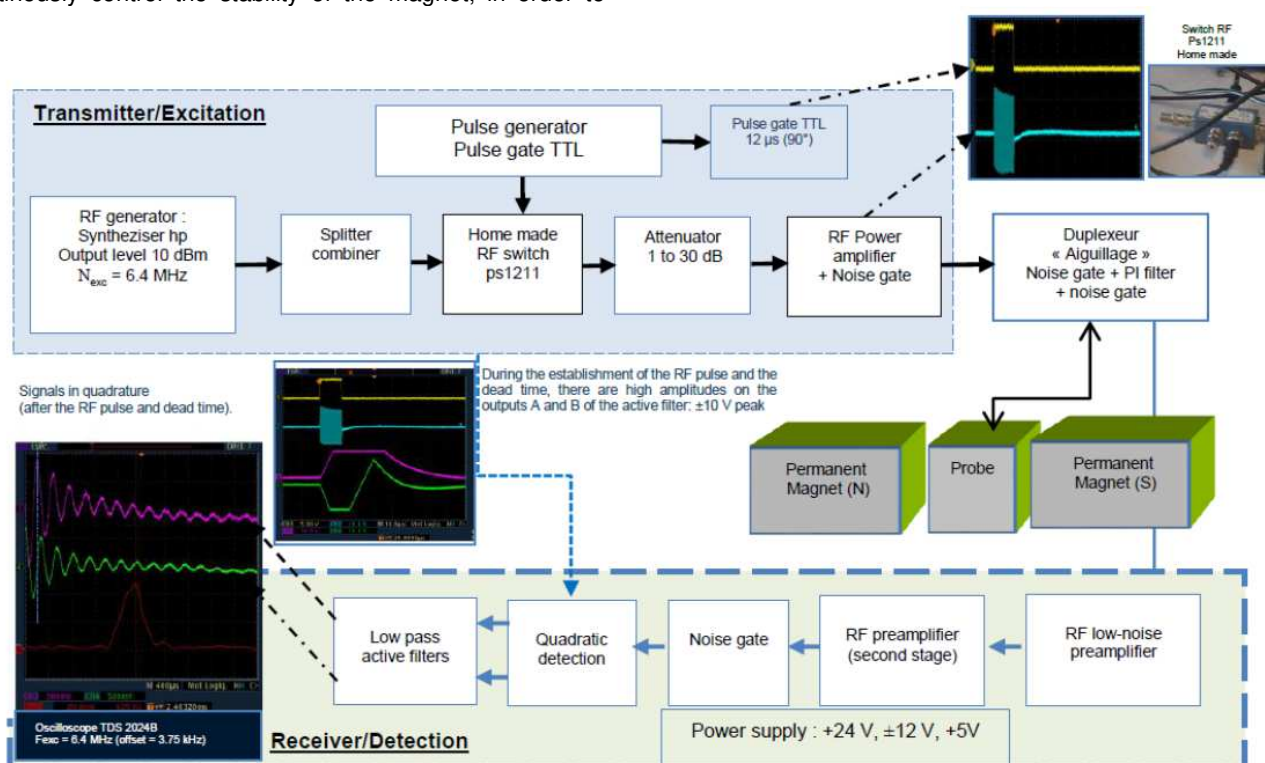


Fig. 4. Schematic description of the NMR spectrometer blocks along with the visualization of some of key signals obtained. Note the presence of the duplexer.

7. NMR device synoptic

For the sake of clarity, the general electronic diagram of a NMR spectrometer is schematically proposed in **Figure 4**. As seen, the attenuators and tuning/matching circuits are present at the input and output of the power amplifier to avoid excess power that can be harmful to the detection chain. The excitation step generates one or more RF pulses at the Larmor frequency. These pulses are directed *via* the duplexer to the NMR probe to tilt M_0 perpendicularly to the z axis. The high-frequency resonance Free induction Decay signal (HFFID) is then routed *via* the duplexer to the detection circuit. The detection circuit includes at least two very low-noise RF preamplifiers to amplify this HFFID signal. The signal is then demodulated with an intermediate frequency, in quadrature (quadratic detection leading to a

90° phase shift of the signal) in order to generate a real (R) and an imaginary (I) component accordingly to **Eq. 5**,

$$S(t) = A \times \{\cos(2\pi\nu t + \phi) + i \sin(2\pi\nu t + \phi)\} \quad (5)$$

where A is the amplitude and ϕ is the phase of the detected signal. This signal is then filtered to eliminate unwanted frequency components. At the output of the active "low-pass" filter, the signal is lowered to the ten kHz range (often wrongly called « audio filter »). The R and I components of signal can then be observed and checked on an oscilloscope or digitized *via* an analogue-digital converter (ADC) for further processing.

Although the spectrometer is optimized to work around the 6.4 MHz frequency, it can easily operate efficiently over a wider band. All the electronic components required for the construction of low-cost NMR spectrometer are listed in **Table 2**, with the adequate associated nomenclature. The

choice of the bandwidths of the various elements present in the nomenclature makes it possible to work up to frequencies around 500 MHz. The assembly of the various blocks can be made entirely on printed circuit board or more simply by connexion components (BNC or SMA outputs).

8. Key electronic elements of the spectrometer

Let's now review the key elements of the equipment (probe, switch, RF preamplifier, ...) in the order of the synoptic block diagram presented in **Figure 4**.

8.1 RF pulse generator. The first important function of an NMR spectrometer is the generation of the basic frequencies (BF) necessary for excitation/reception of signal. The RF excitation pulse is realized by means of a switch connected to a frequency synthesizer and a pulse generator. However, to perform a synchronous detection (resonance), part of this frequency must be directed to the detection circuit.

The generation of RF pulses can be performed in different ways. A first one consists to use a standard frequency and pulse generator present in any physicist laboratory. In order to ensure sufficient stability and accuracy for NMR, the resolution of the function generator must be at least 10^{-6} . Typically, an arbitrary function generator of type HP33120A

or Agilent 33220A operating up to 15 and 20 MHz respectively with a power of 10 dBm is perfectly suited for a low field NMR spectrometer. For pulses, the pulse generator can also be used to produce square-shaped pulses. For pulses of the order of ten microseconds (10 μ s) a pulse generator with a resolution of less than 100 ns is sufficient (e.g. Keysight 33220A). The time and frequency parameters are adjusted from the control panels of these laboratory instruments. In this case, only very simple single-pulse sequences are possible: 90° pulse - acquisition; 180° inversion pulse - acquisition. The primary advantage of laboratory equipment is their availability at a lower cost, their speed and simplicity of implementation (no software development for pulse programs). The second way to generate pulse sequences is to use a board based on an Advanced Reduced instruction set computing Machines (ARM) microcontroller, a Field Programmable Gate Area (FPGA) unit and a Direct Digital Synthesis AD9959 (DDS) which allow digital frequencies up to 300 MHz to be synthesised. The advantage in this case is to have a very compact and powerful unit to generate the basic pulses and frequencies. It can be developed, for example, from an NXPLPC1768 ARM, an Altera Cyclone II FPGA [20, 21] and a DDS. This board is capable of sequencing four fully independent channels. The FPGA allows the most complex pulse sequences to be performed, the ARM component then manages both the system and data processing. This option allows stand-alone battery operation for outdoor (off-laboratory) use.

8.2 The Switch. The role of this circuit is to switch the RF signal for a controlled pulse duration. This element can be realized by means of four fast diodes (type 1N4148) mounted in head to tail or with a PS1211 mini-circuit

component (switch), which offers fast switching times (< 500 ns) and "unbalanced" very weak between channels (see **Figure 5**).

8.3 The RF power amplifier. As our mobile NMR is aimed to work from low to high field, it is necessary to have broadband RF excitation pulse. The excitation provided to the spin system must be sufficiently high, with relatively short RF pulses (10 to 100 μ s) to excite a bandwidth covering the spectral width of an observed nucleus. The selected power amplifier must be able to operate in pulse mode but also in continuous wave (CW) if nuclear decoupling or any other type of low power excitation needs to be achieved. It is imperative that the amplifier has a good linearity in order to adjust the RF pulse accurately. A power amplifier generates a high noise level at the output even at rest. It is therefore necessary not activated. We used for our prototype a ZHL-5W-1 unit operating in a frequency range from 1 to 500 MHz.

8.4 The duplexer. As already mentioned above, the duplexer has a very important role, in particular to protect, during reception, the low noise preamplifier chain from the high RF power emitted during excitation. It is a three-port system A, B and C (see **Figure 6** where the first port (A) is connected to the power amplifier, the port (B) is connected to the probe and the output (C) is directed to the low noise detection preamplifier chain. For broadband systems, it is preferable to use a switch network electronically controlled by the pulse sequencer. In low-frequency (low-field) NMR applications, a structure based on diode arrays and a quarter-wave filter can be used; the diagram is shown in **Figure 6**. A first network of four

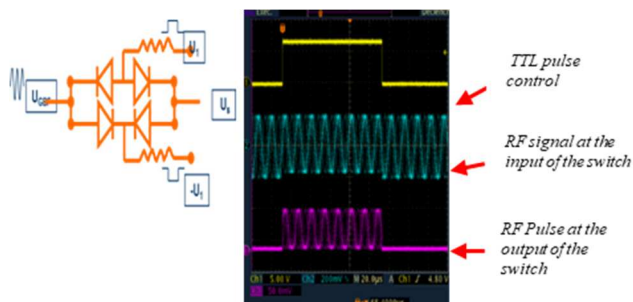


Fig. 5. (On left) Structure of an RF switch based on fast diodes. (On right) Example of input and output signals from a RF switch. Here, the RF pulse has a length of 80 μ s.

head-to-tail diodes (noise gate 1) acts as an automatic switch: these diodes are blocked (open circuit) for small amplitudes, thus preventing any return of the signal from the probe to the power amplifier. The quarter-wave system, referred to as PI filter, has an input (B) / output (C) phase shift of 90° . It is called a quarter wave because it has a phase shift optimized at 90° around a given frequency, in our case, around 6.28 MHz. This filter consists of a choke and two capacitors. The filter input is connected to the probe (port B) and the output (port C) to the low noise preamplifier chain. For a specific frequency, a quarter-wave

filter has a minimum amplitude at its output (C) when its input (B) is maximum. In order to reinforce the protection of the preamplifiers, a second network of head-to-tail diodes can be incorporated to protect the detection circuit by short-circuiting (at the threshold of the diodes) any residual voltages. The filter setting must combine input and output impedance matching, as well as phase shift and attenuation at the resonance frequency. The characteristics of the quarter-wave filter were adjusted to the network analyzer with the Smith diagrams of the input (S11) and output (S22) impedances as a function of frequency. The input (S11) and output (S22) impedances have been adjusted to have an actual value as close as possible to $50\ \Omega$ (in practice $44.5\ \Omega$ at the resonance frequency of 6.28 MHz), while maintaining a 90° phase shift between input and output.

8.5 The NMR probe. Different probe body shapes (solenoid, saddle coil) can be produced and used depending on the adaptability to the magnet. The most simple detection antenna consists of a solenoid coil. A probe body made entirely of glass (borosilicate) can be used (see **Figure 7a**). For a 10-mm probe, the solenoid is wound on the glass tube

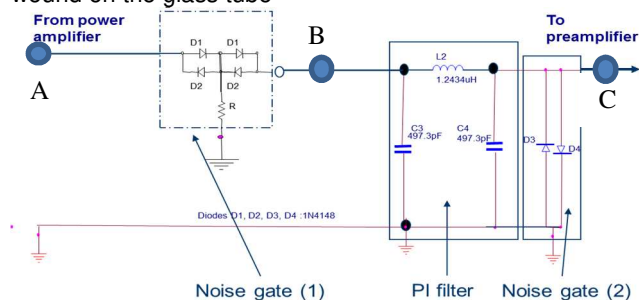


Fig. 6. Electrical diagram of the duplexer, including two diode arrays and the PI filter. The PI filter has a minimum RF signal at the receiver (port C) when there is a maximum on the probe (port B). The "noise gate 1" isolates any signal feedback from the probe to the power amplifier in order to minimize losses during detection. The "noise gate 2" protects the detection chain from residual voltages eliminated by the PI filter.

with an inner diameter of 10 mm, (thus allowing the detection of a large amount of analyte), but small diameter (5 mm) could be used. The capacitive network is integrated into the probe. The solenoid is made of rigid copper wire with a diameter of 1 to 1.5 mm and includes about 20 turns with a diameter of 10 mm over a length of 20 mm; the better the quality of the winding, the smaller the artefacts will be. The elementary structure of the frequency tuning and impedance matching system is described in **Figure 7b**. A network of capacitors adjustable from 5 to 500 pF allows frequency tuning (C_t) and impedance matching (C_m) for a coil of the order of $2.2\ \mu\text{H}$. The tuning allows the probe to be adjusted to the resonance frequency ($\nu_0 \approx \nu_1 = 1/2\pi\sqrt{LC}$). Impedance matching is performed at this frequency to adjust the probe circuit to $50\ \Omega$ and minimize the energy losses.

Frequency tuning and impedance matching are performed using a network analyzer to accurately position the probe's attenuation peak at the desired resonance frequency while

observing the impedance evolution on the Smith chart (see **Figure 8**). Impedance matching consists of refining and lowering the resonance peak as low as possible in order to absorb all the energy in the coil. In our case, the resonance is adjusted with the network analyzer at 6.284 MHz, the attenuation obtained is -50.816 dB with a very good impedance matching ($49.72\ \Omega$), and consequently a good detection sensitivity for a low-field spectrometer. The half-height width obtained at this frequency is 35 kHz, giving a quality coefficient Q of 179, a very acceptable value.

Once the frequency tuning has been achieved, a second important parameter to be estimated is the dead time (or response time) of the probe (see **Figure 9**). The dead time is the time required for the probe to dissipate the energy stored during excitation. Some of the excitation energy is sent to the probe via the duplexer, but once the RF pulse has disappeared, significant energy remains in the probe. The higher the quality factor Q of the probe, the higher the dead time is. This is a disadvantage for detection since it will be necessary to delay the measurement of this time in order: i) not to destroy the low-noise preamplifiers; ii) not to superimpose the low detection signal on the high amplitude due to the probe signal.

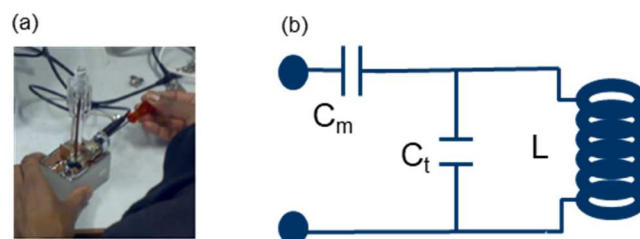


Fig. 7. (a) Example of a probe made of glass with its integrated capacitive devices C_m and C_t . (b) NMR probe wiring diagram: C_m and C_t are adjustable capacitances from 5 pF to 500 pF, inductance coil, $L = 2$ to $3\ \mu\text{H}$.

In the model presented in this article, the dead time is about $250\ \mu\text{s}$ before the two quadrature detection channels return to zero (see **Figure 9**). The detection of the useful signal needs to be delayed by this delay and the detection circuit is reinforced, by a network of diodes, in order to support the peak amplitudes of several volts for a few hundred microseconds.

8.6 The receiving chain: Radio frequency preamplifier.

The signal from the probe is too weak and cannot be used directly, so it must be amplified. The amplification chain consists of two stages with radiofrequency preamplifiers in series. The noise factor of a preamplifier cascade is almost equal to that of the first stage. A "BA01500" preamplifier with

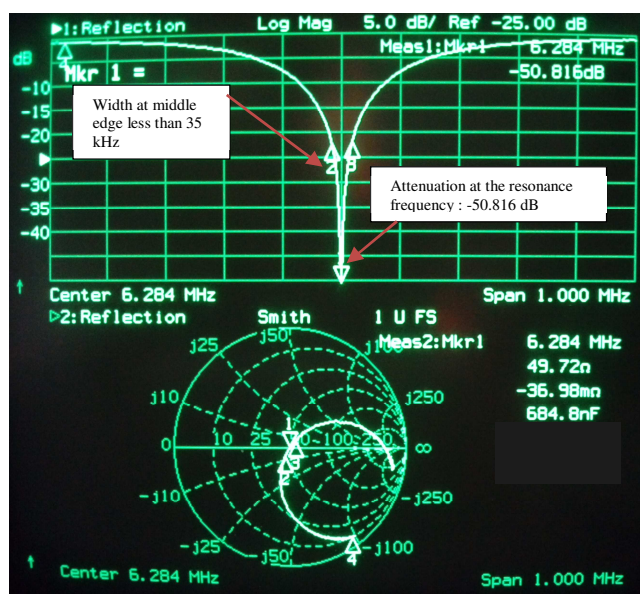


Fig. 8. Picture showing the measurement of the frequency tuning and impedance matching curve (top) and on the Smith chart (bottom) on a network analyser.

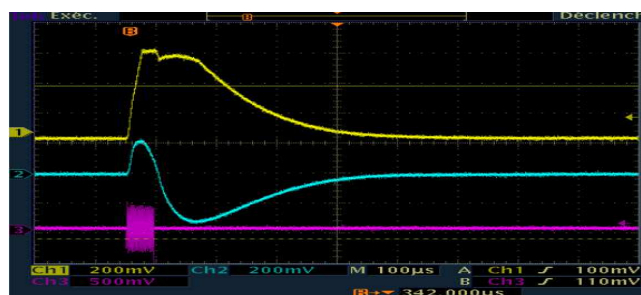


Fig. 9. Observation of the probe's dead time: about 250 μs. At the top and in the middle are displayed the two quadrature detection channels (yellow and blue). At the bottom is displayed the rectangular (purple) RF excitation pulse (50 μs of length).

the lowest factor is placed first, followed by a RF2046 home-made broadband preamp unit. The Low Noise Amplifier: LN BA01500-35 has a frequency range varying from 0.1 to 500 MHz with a gain of 35 dB. The intermodulation product power at the output is +12 dBm (output IP3), and the undistorted input power is -35 dBm (1 dB compression point). Thus, the maximum permissible input power is therefore -35 dBm.

Our home-made preamplifier is made with three RF2046 components in series: the overall gain of the channel measured at the network analyzer (without distortion) is 35 dB at 6 MHz and 31 dB at 1 MHz and the operating frequency range is from DC continuous to 1 GHz. The output intermodulation product (IP3) is +23 dBm and the noise factor (NF) is equal to 3.8 dB. This configuration of the elements as well as their performances allows to ensure the detection of signals on a large dynamic range without distortion: typically from some 100 μV to 14 mV.

8.7 Quadrature demodulation. Quadratic demodulation is based on the principle of synchronous detection to obtain real and imaginary components of the NMR signal. Its structure is based on a Quadrature Intermediate Frequency Mixer (QIFM) demodulator whose schema is given (see Figure 10). It consists of a 90° hybrid splitter, two mixers and a 0° combiner (summing). Each of these elements is listed in the Table of components (see Table 2).

At the input of the 90° hybrid divider, we have the base frequency of the Local Oscillator (LO) input spectrometer this is actually the Larmor frequency of the spectrometer. At the input of the 0° divider, we have the FID: this is the high frequency response RF. At the output of the quadratic demodulator on channels I (imaginary) and Q (quadratic) -the signals are of the form: i) $\cos[2\pi(v_{rf} + v_0)t] + \cos[2\pi(v_{rf} - v_0)t]$; ii) $\sin[2\pi(v_{rf} + v_0)t] - \sin[2\pi(v_{rf} - v_0)t]$. We therefore obtain two quadrature components with all the "sum" and "difference" frequencies of the demodulated signals: $2\pi(v_{rf} + v_0)$ and $\pi(v_{rf} - v_0)$, plus harmonics. Only low-frequency signals in the ten-kHz range are interesting, and the signal must be filtered to retain only the lowest frequencies using a low-pass filter.

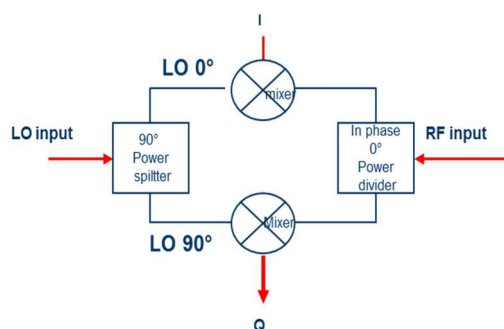


Fig. 10. Structure of a Quadrature Intermediate Frequency Mixer demodulator (QIFM). Mixers: X2L-06-414 from Pulsar Microwave.

8.8 Double actif audio low-pass filter. An active double low-pass filter is required to keep only the low frequencies (see Figure 11). Each filter is designed to charge the demodulators on 50 Ω and consists of two stages allowing an overall gain of about 30 dB in voltage. As around the resonance, the measured frequency differences are about ten kilohertz, the filter bandwidths are adjustable over a range from 100 Hz to 500 KHz.

The two filter channels must be adjusted to have the same frequency and phase responses throughout the useful bandwidth. This is to avoid unbalancing between the real and imaginary components of the signal, which would degrade the spectrum in frequency (quadrature artifact, zero frequency, ...). Precise gain and phase adjustments in the bandwidth are made with a network analyzer, the only true and effective way to visualize the response of both channels as a function of frequency.

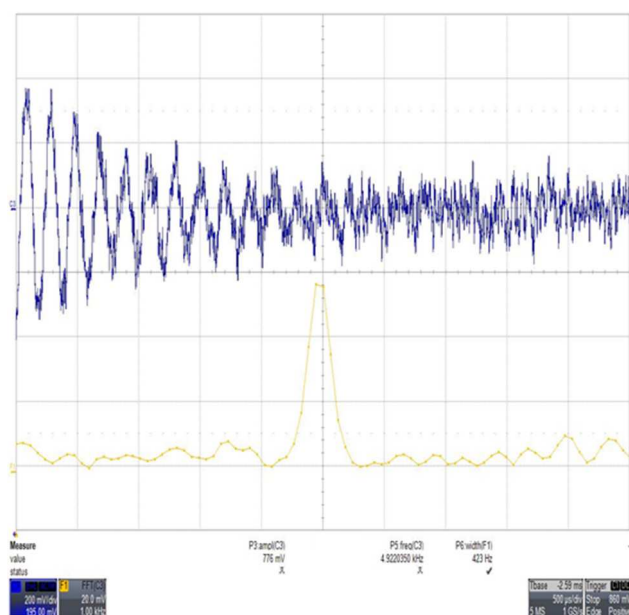


Fig. 11. ^1H signal (one component) of a water sample (5 mm) obtained (one SCAN : NS = 1) after the demodulation stage and low pass filtering, observed on a digital oscilloscope with FFT. Top : FID component. Bottom : Spectrum : presence of the peaks at 4.92 KHz. Note the linewidth of 423 Hz. The Larmor frequency was $\nu_0 = 6.4$ MHz. The RF excitation pulse length is 50 μs . See appendix experimental setup for details.

9. Setting of spectrometer

9.1 Probe control. The tuning of the probe to the resonance frequency has been described above.

9.2 Cancellation of the DC offsets. Any DC voltage offset in the detection circuit generates a zero frequency component that will affect the quality of the spectra. To reduce this unwanted effect, it is important to carefully cancel the offset voltages of the active double low-pass audio filter in order to have a signal so close to zero when the input signal is then zero.

9.3 Signals at the output of the audio amplifier without sample. At first, we work without putting a sample in the magnet in order to minimize the imperfections of the assembly: adjustment of the LO level (10 dBm); cancellation of continuous offsets. In the absence of a sample, the voltage offsets are less than mV. In addition, note that the probe's deadtime after the RF pulse is 400 μs (see **Figure 12a**).

9.4 Signals at the output of an audio amplifier with sample. The imperfections of the assembly having been minimized, we introduce our sample (a 5-mm tube of water), and we visualize the precession signals in quadrature mode (see **Figure 12b**). Note the very low magnitude of the signals.

9.5 Optimization of the 90° flip angle. The NMR signal obtained at this step, although observable, is relatively small

in amplitude. To maximize the amplitude of the FID, it is now necessary to adjust the strength excitation pulse for a tilting of M_0 equal to 90° . For this reason, the duration of the pulse is varied until a magnetization is obtained whose intensity in the xy plane is maximum. The amplitude variation of the magnetization vector is observed as a function of the excitation pulse duration. In our case, the pulse duration is between 13 to 14 μs . Another approach is to determine the pulse duration corresponding to an angle of 180° , for which the macroscopic magnetization is then null (tilted to the axe -Oz).

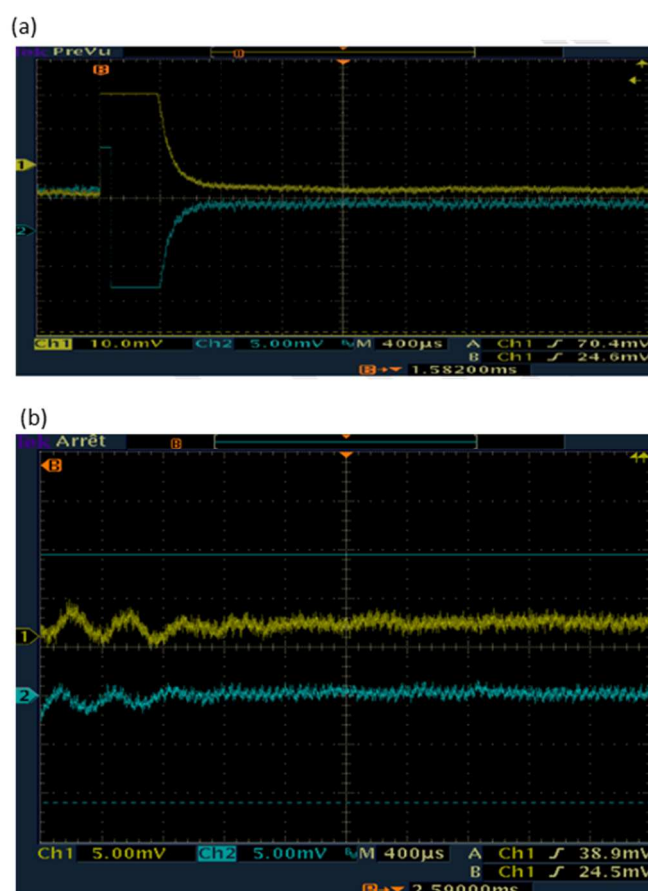


Fig. 12. (a) No NMR tube: Real (high, channel 1) and imaginary (low, channel 2) components at the output of the audio frequency amplifier after an RF excitation pulse length of 50 μs . (b) Example of one-scan quadrature detection of a low magnitude ^1H signal (5-mm water tube). See appendix for the experimental set up.

9.6 Increase in the S/N ratio. The signal-to-noise ratio (SNR) of an NMR experiment involve numerous factors such as: i) the magnetization M_0 , ii) the filling factor and the quality factor of the probe, iii) noise factor of preamplifier and so on [22]. After optimization of all the electronic hardware and parameters, we can also increase the SNR during the acquisition. The SNR can be improved by averaging the FID signals over a large number of acquisitions (NS scans). Indeed, on an average of NS acquisitions, the noise will be averaged towards zero due to

its random origin, while the useful signal will increase on average. Thus, the SNR is improved at the root of NS as displayed in **Figure 14**.

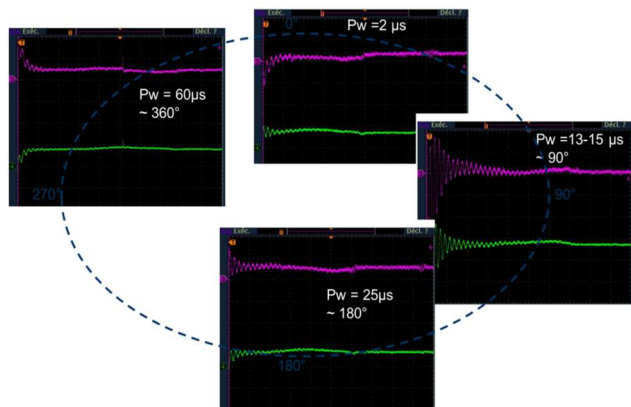


Fig. 13. Adjustment of the RF excitation pulse duration to obtain a maximized signal corresponding to a 90° pulse.

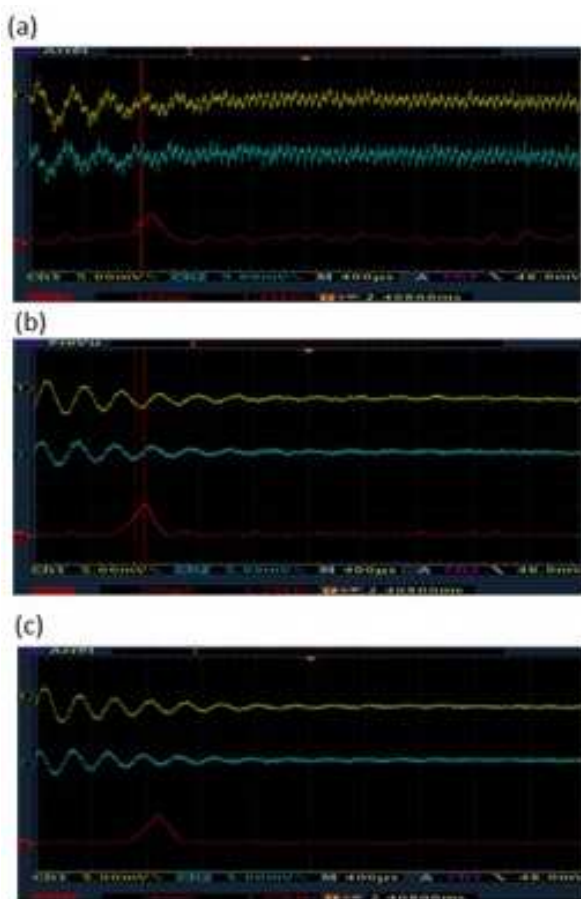


Fig. 14. Effect of the averaging on the SNR. Comparison of the FIDs obtained by adding successively : (a) 2 scans, (b) 32, (c) 64 scans. (5-mm water tube). On (c), note the vertical line setting at 3.45 MHz. See the Appendix for the experimental set up.

10. Possible applications

Benchtop FT-NMR allows many applications in chemistry, such as the analysis or characterization of small molecules, or the monitoring of chemical reactions. The teaching of NMR methodologies (effects of excitation/inversion pulses, pulse calibration, optimisation of acquisition parameters, ..., as well as the description of some experiments as the measurement of relaxation parameters, spin echoes, ...) is also another aspect of their use. The portable low-field NMR is particularly suitable for nuclei with high natural sensitivity, such as ^1H , ^{19}F , ^{31}P , ^{29}Si . However, other « exotic » nuclei with high gyromagnetic ratios such as ^7Li , ^{11}B , ^{23}Na can also be detected [27,28].

A (home-built) mobile NMR spectrometer can be also used to develop projects that aim to couple NMR and optical microscopy in a semiconductor [23] to locally polarize nuclear spins (dynamic polarization) and to measure by optically detected NMR the spatial profile of nuclear magnetization, with good spatial resolution. This fundamental study of nuclear spin diffusion does not require a high magnetic field (less than 0.15 T). NMR functionalities are reduced to signal excitation and detection for parameter optimization, without the need for frequency resolution.

11. Conclusion

The low-field FT-NMR spectrometer presented in this article is fully operational. This revisited instrument can be used to carry out simple NMR measurements such as the determination of chemical shifts (δ), the spin echoes, the measurement of T_2 (^1H) or T_1 relaxation times, etc. On the other hand, it provides a pedagogical approach to the teaching of NMR to students at all levels, from the physical concept to the electronic realization through the many possible analysis applications [24, 25, 26]. It is also an excellent demonstration element at scientific events for a more general public. Finally, it can be inserted in real experiments of physics requiring NMR to be coupled with another measurement technique such as optics for example.

Noteworthy technical evolution of the instrument is possible. Thus if the permanent magnet is equipped with an additional pair of coils with currents in the range of 1 to 3 A, it can then be injected to create additional field gradients to B_0 . The spectrometer is then able to perform some simple 1D imaging experiments, or to experimentally measure molecular diffusion coefficients.

Although sensitivity is reduced at low-field strength, quantification and measurement of relaxation and diffusion parameters of neat samples or in solutions are possible [27, 28]. This kind of portable NMR spectrometer could appear then the ideal tool for outdoor NMR analysis of ecosystems.

12. Acknowledgements

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14. Conflicts of interest

The authors declare no conflicts of interest.

15. Appendix

15.1 Definitions of acronyms and notations used

ARM : Advanced Reduced instruction set computing Machines

ADC : Analogue to Digital Convertor

BNC : Bayonet Neill-Concelman (connector)

B₀ : Static magnetic field

BF : Basic Frequency

B₁ : RF coil. The term B₁ is used to characterize the field, in the plane, orthogonal to the static B₀ field. In the terminology frequently encountered in the fields of NMR, the B₁ coil is a resonant "LCR" circuit tuned to

the Larmor frequency. This coil is used for the detection and the excitation of the nuclei in FT-NMR experiments. L is inductance, C capacitance and R resistance.

CW : Continuous Wave

dB : Decibel

dBm : Power in Decibel : $0 \text{ dBm} = 1 \text{ mW} / 50 \Omega$

DDS : Direct Digital Synthesis

FFT : Fast Fourier Transformation

FID : Free Induction Decay

FT : Fourier Transformation

FPGA : Field Programmable Gate Area

LO : Local Oscillator

NF : Noise Factor

Q : Quality factor

QIFM : Quadrature Intermediate Frequency Mixer

RF : Radiofrequency

NMR : Nuclear Magnetic Resonance

SMA : SubMiniature version A (connector)

SNR : Signal-to-Noise Ratio

TTL : Transistors Transistors Logics

T₂ : Spin-spin relaxation Time

T₁ : Spin-lattice relaxation Time

ν_0 : Larmor Frequency (MHz)

15.2 Experimental set-up (except otherwise in the text).

All NMR and test experiments have been made with the electronic setup outlined in **Figure 4**. A permanent magnet with a field of 1500 G (6.4 MHz) is used for the static magnetic B_0 field (see Figure 2). A home-made solenoid-type probe device and a 5-mm water sample tube were used for the tests. The frequency and the RF pulse duration have been set with the frequency generator and the pulse generator mentioned in the text. Data have been acquired and processed with some digital oscilloscopes with a sensitivities of 1 or 2 mV/div.

15.3 Characteristics and nomenclature of the electronic components. **Table 2** provides the characteristics and nomenclature of the electronic components used.

Table 2. Characteristics and nomenclature of electronic components

Characteristics	Designation	Number of devices	Frequency range (MHz)
1500 G permanent magnet, 40-mm air gap, NMR homogeneity on a useful diameter of 20 mm, divergent polar pieces on a diameter of 200 mm, height 400 mm.	TE2M C4-1367 1500 G	1	6.2 - 6.8 MHz
Directional coupler 30 dB, 3W, SMA (pulsar)	C5-08-411	2	5-1000
SMA connector (pulsar)		20	0.1 - 1000
Mixer level 7 dBm SMA (pulsar)	X2L-06-414	2	(LO/RF) 1-1000 : (IF) DC-1000
Power divider/combiner, 2 way (0°), phase balance: 2°, amplitude balance: 0.3 dB	P2-09-411	3	5 - 1000
Insertion loss 0.8 dB, SMA (pulsar)			
90° hybride power divider	QE-01-412	1	2 - 10
Phase balance 2°, amplitude balance 0.4 dB, insertion loss 0.5 dB, SMA (pulsar)			
Low noise RF preamplifier NF = 2, gain: 35 dB, RF output power: 5 dBm	BA01500-35	1	0.1 - 500
Absolute maximum rating input			
RF power: -35 dBm (Elhyte)			
Low noise RF preamplifier NF = 2.9, gain: 25 dB, output RF power: 5 dBm (mini circuit)	ZFL1000LN+	1	0.1 - 1000
Low noise RF preamplifier RF; NF = 3.8, gain: 23 dB, RF output power: 5 dBm (home made)	RF2046	3	DC -1000
Power amplifier	ZHL 5W -1	1	
Operational amplifier	OP27	1	DC - 8
Operational amplifier	OP37	1	DC - 60
Resistor	1 kΩ, 10 kΩ	10	
Variable capacitor	5 pF to 500 pF	4	
Probe (home made)		1	6.1 - 6.8
Sequencer board with ARM + DDS (home made)	NXPLPC1767+	1	0.01 - 301
Or Frequency generator and laboratory pulse generator (see text).	DDS 9959	1	
Analog or digital oscilloscope with FFT		1	
AC/DC adapter 24 V / 6.4 A	AHM150PS24	1	
Total estimated cost			~ 10 k€