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Low-Field NMR/MRI Systems Using LabVIEW and Advanced Data-Acquisition Techniques

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1. Introduction

Nuclear Magnetic Resonance (NMR) and Magnetic Resonance Imaging (MRI) have become powerful non-invasive analytical tools for a large palette of applications, ranging from solid state physics, to all the branches of chemistry, biology, medical research and medical diagnosis (Ernst et al., 1989). Nowadays, we are also witnessing new areas of applications for a variety of non-invasion measurements such as the quality control of food products (Asfour et al., 2004, Raoof et al., 2002), etc.

To meet the needs for expanding research projects and applications, powerful and expensive spectrometers or imagers are commercially available. Although, these, usually high or medium field, NMR/MRI systems have many advantages, such as a high signal-to-noise ratio (SNR), resolution and high image quality, their use in some specific applications could be prohibitively expensive. Actually, in many cases, for particular purposes, one may only need NMR spectrometers or MR imagers having a subset of the features of a standard commercial one (Gengying et al., 2002). In addition, the use of low- and very-low fields (below 100 mT) could be sufficient in some cases. The cost of the system can then be dramatically reduced since these low- and very-low fields – with, sometimes, relatively poor performances requirements- could be easily produced. Moreover, the use of low fields simplifies the design and realization of compact and portable NMR systems which could be especially appreciated for the in situ applications.

Nevertheless, NMR spectrometers using low fields and so low frequencies (up to several 100 kHz) are not commercially available. A number of groups have worked to develop dedicated MRI/NMR systems by using compact low-field MR magnets. For example, we have proposed in a previous work a home-built and fully digital MRI system working at 0.1 T (resonance frequency of about 4.25 MHz) (Raoof et al., 2002). This system was based on the use of a high-performance Digital Signal Processor (DSP), a direct digital synthesizer (DDS) and a digital receiver. These very advanced hardware and signal processing techniques were typically employed in the base-stations of mobile phone. Based on this work, Shen (Shen et al., 2005) proposed another system working at 0.3 T and allowing larger imaging sizes than in (Raoof et al., 2002). Another work, carried out in (Michal et al., 2002) was focused on the realization of a wideband receiver for a home-built NMR spectrometer working at 55.84 MHz (high field).

Some groups have NMR systems working at low field for the specific application of measurement of the *polarization*¹ for the NMR of hyperpolarized gases (^{129}Xe , ^3He ...). Most of these systems were actually developed by modifying high frequency and high cost commercial spectrometers. One research group has, however, developed its own NMR system (Saam & Conradi, 1998). This system was used for monitoring the *polarization* of hyperpolarized helium (^3He) at 3 mT. It was a fully analog system where authors performed a phase-sensitive detection of the NMR signal. They used then an oscilloscope for signal visualization. Despite the great merit of the original and elegant electronic solutions developed in (Saam & Conradi, 1998), the detection of hyperpolarized ^3He signals was relatively not a hard task since their levels were quite high (at least 10 mV). Actually, this spectrometer did not allow sufficient dynamic range to detect the NMR signal of the proton (^1H) at such field.

In any case, dedicated low-field NMR systems are still far from the experience of most NMR groups. Recently, we developed very low-field NMR spectrometers that allow detection of the ^1H NMR signals at 4.5 mT (Asfour, 2006, 2008, 2010). These developments were initially motivated by their application in the measurement of the *absolute polarization* of hyperpolarized xenon (^{129}Xe).

These systems were based on the use of data-acquisition boards (DAQ). These boards are adequate at low frequencies. Moreover, they have increased in performances and the related software (LabVIEW) made their use quite straightforward. In these new NMR spectrometers, we replaced as much analog electronics as possible with DAQ boards and software. We show that the use of advanced data-acquisition and signal processing techniques allow detection of the ^1H NMR signals at 4.5 mT.

The aim of this chapter is to present these advances in the development of low-field NMR systems. One of the underlying ideas of this chapter is to make these systems versatile and easy-to-replicate so as to help developers and research groups in realizing NMR spectrometers with flexibility, low cost and minimum development time. This is why we describe in some details the variety of the practical aspects of realization. This includes both hardware design and software developments.

For a reader who could be not familiarized with the NMR technique, we present, in a first section of this chapter, a brief and very simplified review of the NMR basic principles using classical physics. The second section is focused on the description of the hardware solutions and architecture of the NMR spectrometers. This architecture is mainly based on the use of signal generator and data-acquisition boards from National Instruments. The software developments (LabVIEW programs) and the advanced data-acquisition and signal processing techniques are presented in the third section. The last section will concentrate on applications and discussions. The use of the developed system for the measurement of the nuclear polarization of hyperpolarized gases will be particularly illustrated.

In addition to these new advances, general principles of the NMR instrumentation are sometimes illustrated. While these aspects could seem basic for confirmed NMR developers, we believe that they may be of great value for beginners, students and for education purposes. Actually, while many publications (academic courses, books, journals...) illustrate

¹ See section 5.1 and references (Asfour, 2010) and (Saam & Conradi, 1998) for the definition of the *absolute polarizations*.

the principles of the NMR, these publications usually concentrate on NMR physics rather than NMR instrumentation. We hope that this chapter could be of help for those who desire to learn about this exciting area. This is why schematics of the developed hardware, software as well as any detail about the design could be obtained by simply writing to the author.

2. NMR basic principles

As it is well known, when a sample, consisting of NMR-sensitive nuclei (^1H , ^3He , ^{129}Xe ; ^{23}Na , etc.), is subjected to a uniform static magnetic field B_0 , a net macroscopic magnetization M of the sample appears in the same direction of B_0 . This magnetization is proportional, roughly speaking, to this polarizing field B_0 , to the density of nuclei within the sample and to the characteristic gyro-magnetic ratio γ of the nucleus being studied.

In a typical one-pulse experiment, the sample is subjected to a short pulse (called excitation pulse) of a radiofrequency (RF) magnetic field B_1 , applied perpendicularly to B_0 and at the characteristic Larmor frequency f_0 . This frequency depends on the nucleus and of the static magnetic field according to the equation (1):

$$f_0 = \frac{\gamma}{2\pi} B_0 \quad (1)$$

For the proton (^1H nucleus), this frequency is about 42.25 MHz at $B_0 = 1$ T and about 190 kHz at 4.5 mT, while it is about 52 kHz for the xenon-129 (^{129}Xe) at this last field.

The effect of the excitation pulse is that the magnetization, M , is “tipped” or rotated from its initial direction (or from its thermal equilibrium state) by an angle α . This angle is called “flip angle”. It is proportional to field B_1 and to its duration, τ , according to the equation (2):

$$\alpha = \gamma \cdot \tau \cdot B_1 \quad (2)$$

At the end of the excitation pulse, the NMR signal- called also the Free Induction Decay (FID) - is received at the same frequency f_0 . This signal, which is proportional to the magnetization M (then to B_0) and to γ , is processed to be used for obtaining a “fingerprint” of the environment of the nucleus being studied.

The flip angle can be set through the adjustment of the amplitude, B_1 , or/and the duration, τ , of the excitation pulse. For a one-pulse sequence, the maximum NMR signal level is obtained at a flip angle of 90° . However, the choice of the optimum value of this flip angle in more advanced NMR/MRI pulse sequences depends on many considerations which are out of the scope of this chapter.

One should also know that a variety of parameters contribute in the signal-to-noise ratio (SNR). Firstly, and roughly speaking, the SNR is proportional to the square of the static magnetic strength. The SNR, the image quality and spectral resolution are enhanced at high field. This is one of the main reasons for which NMR experiments are usually performed at high fields. Secondly, the SNR is proportional density of the nuclei within the sample being studied and its depends on the nucleus of interest (through the gyro magnetic ratio γ). Finally, for a given nucleus, a given B_0 and a given volume, the SNR depends strongly on the characteristics of the detection coil.

3. A hardware structure for low-field NMR systems

Based on these simplified principles, Fig. 1 illustrates the general hardware architecture of the developed low-field NMR systems.

Fig. 1. The hardware architecture of the low -field NMR systems.

The static field B_0 of about 4.5 mT is produced by a pair of Helmholtz coils. The excitation pulse (at about 190 kHz for ^1H) is generated by the transmitter (arbitrary waveform generator board NI 5411 from National Instruments). This pulse is amplified by a power amplifier and sent, via the duplexer, to the well-tuned coil (at the working frequency of 190 kHz for the ^1H) which generates the excitation field B_1 .

At the end of the excitation pulse, this same tuned coil detects the weak NMR signal. This signal is transmitted to a low-noise preamplifier via the same duplexer. The amplified signal is then received by the receiving board (A digitizer board NI 5911 from National Instruments) for digitalization and processing.

A monostable-based circuit generates TTL control and synchronization signals from a single and very short (about 10 ns) TTL pulse ("Marker") that could be generated from the NI 5411. At least, two signals are necessary. Since the same coil is used for both transmitting and receiving (i.e. a transmit-receive coil), a "blanking signal" is required to control the duplexer. This signal "blanks" the preamplifier input during the excitation pulse and it isolates the transmitting section from the receiving one during the NMR signal detection. Another control signal (trigger signal) is necessary for triggering the signal acquisition with the receiving board.

An example of the timing diagram of a one-pulse NMR experiment realized by the developed spectrometer is shown in Fig. 2.

Fig. 2. The timing-diagram of a typical one-pulse NMR experiment.

The main elements of this hardware are developed in the next sections. Details about the hardware design are given to enable developer to easily replicate the system.

3.1 Magnetic field production: The Helmholtz coils for low and very-low fields

In low-field and very-low-field NMR systems, the static magnetic fields, B_0 , could be produced using a variety of magnet categories and structures. The choice of a category and a structure is strongly related to the application, and its depends on many considerations such as the value of the magnet field, the desired performances (field stability, spatial homogeneity...), the cost and complexity of realization as well as the ease-of-use. These magnets can however be divided into two categories²: permanent magnets and electro-magnets. Permanent magnet of 0.08 T has been used in the development of an MR imager for education purposes (Wright et al., 2010). Permanent magnets with a typical field of 0.1 T have also potential industrial applications (quality control of food products) (Asfour et al. 2004). Some dedicated MR imagers using permanent magnets are commercially available for medical applications. Original and elegant structures of permanent magnets of 0.1 T have been proposed in a portable system for potential application for the high resolution NMR in inhomogeneous field (LeBec et al., 2006).

The main advantage of permanent magnets is that they do not use any power supply. However, these magnets could not offer a good stability of the field because of the temperature-dependence of their magnetization. Another disadvantage is the imperfections of the magnetic materials and may be the complexity of realization.

Electro- magnets can offer an alternative solution. Typically, the obtained field strength could be as high as 0.5 T. A water-cooled electro-magnet of 0.1 T was used in (Raouf et al., 2002) for a dedicated MRI system for both medical and industrial applications.

² High fields are generally created by superconducting magnets

In the systems developed in (Asfour, 2006, 2008, 2010), a pair of Helmholtz coils was used as illustrated by Fig. 3 (only one Helmholtz coil is shown). More homogeneous magnetic field could be obtained using four coils.

Fig. 3. Helmholtz coils producing a 4.5 mT magnetic field. A stabilized supply current of about 5A is used. A metallic shield serves for shielding the NMR coil when it is positioned inside the magnet

These coils produce a magnetic field of 4.5 mT when they are supplied by a DC current of about 5 A. The design of Helmholtz coils is well-known. It will not be developed here. It is however important to know that it is crucial to use a stabilized power supplier to maintain a constant value of the produced field and hence a constant resonance frequency. This is fundamental for the NMR, especially at low field where NMR signal averaging is still necessary.

3.2 The transmitter: Arbitrary waveform generator NI 5411

The excitation pulse at the working frequency is digitally synthesized using the PCI board NI 5411 from National Instruments. This device is an arbitrary-waveform generator (AWG) which has been chosen for its interesting features for the NMR, especially its high flexibility for pulse sequences programming and generation. The related NI-FGEN instrument driver is used to program and control it using LabVIEW.

The device can operate in two waveform-generation modes: Arbitrary mode and DDS (Direct Digital Synthesis) mode. This flexibility allows its use for a large palette of applications, and it is specially appreciated for the NMR. The paragraph 4.2 and the user-manual of the device give more description of these modes and their use.

In both modes, the digitally synthesized waveform is interpolated by a half-band digital filter and then fed to a high-speed 12-bit DAC (Analog-to-Digital Converter). The DAC output is optionally applied to an output amplifier and/or an analog filter to generate the final analog output signal.

Digital outputs are also available. One digital output ("Marker") is a TTL compatible signal that can be set up at any point of the analog waveform being generated. This signal is used as a trigger pulse for the generation of TTL synchronization and control signals (see Fig. 1 and Fig. 2).

Full details and description of the board architecture and features could be found in the user manual of the NI 5411.

3.3 Power amplifier and generation of the TTL synchronization and control signals

The generated pulse from the AWG is amplified by a power amplifier stage. This stage was based on the use of the operational amplifier (op-amp) AD711. This op-amp was chosen for its high output slew rate (Saam & Conradi, 1998), (Asfour 2006, 2008, 2010). The amplifier was realized on the same board together with the circuit that generates the synchronization and control signals. Fig. 4 shows the schematic of the main parts of the board.

Fig. 4. Schematic of the power amplifier and the circuit for the generation of synchronization and control signals.

The stage allows more than 25 V peak-to-peak output measured on a high impedance oscilloscope. Since the amplifier is loaded by the duplexer and the NMR coil, the voltage across the coil should be lower and it depends on how well the coil is tuned during the transmitting period.

The design of op-amp-based power amplifiers for the NMR has to take into account the oscillations that could appear when the amplifier is loaded by the capacitive and inductive coil. The design in Fig. 4 employs a 100 Ω resistor which enables the amplifier to drive large capacitive loads. The resistor effectively isolates the high frequency feedback from the load and stabilizes the circuit.

The synchronization and control TTL signals are generated using a monostable-based circuit (74123) triggered by the "Marker" from the AWG NI 5411. The circuit produces two complementary signals for blanking the preamplifier and for triggering the acquisition using the receiving board. The duration of these signals could be easily adjusted by external capacitors and resistors.

3.4 The NMR coil, duplexer and low-noise preamplifier

The well-tuned coil is one of the key elements for a successful detection of the weak NMR signals. Regardless the geometry of the coil, the equivalent electrical circuit of an NMR coil is an inductance which is tuned by one or more capacitors to form a parallel resonant circuit

at the working frequency. The geometry and the electrical structure of the coil are generally chosen to optimize the spatial homogeneity of the excitation field within the sample and/or the sensitivity of detection. For a given application, the coil structure depends strongly on these desired performances as well as on the working frequency.

A large number of geometries, electrical structures and coil configurations have been studied, published and use. The interested reader may refer to (Mispelter et al., 2006) for more information about the NMR and MRI coils.

However, coils for low and very-low frequencies have actually not been widely investigated. A simple sensitive coil is proposed here. It is a transmit-receive surface coil of about 400 turns of a Litz wire with an average diameter of 2 cm and a height of 0.5 cm (Fig.5) The developed inductance is about 1.3 mH measured at 190 kHz. Fig. 5 shows the coil as well as its position, together with the sample, inside the magnet and the shield.

Fig. 5. The transmit-receive coil (left figure). On the right figure, the coil could be seen inside the Helmholtz coils and the shield. The coil is loaded by a sample consisting of Pyrex (a type of glass) cylinder filled with pure water.

Calculations have showed that a quality factor, Q , of the resonant circuit of at least 120 (at 190 kHz) is necessary for the detection of ^1H NMR signals. Even at this relatively low frequency, the use of a Litz wire was important to minimize the skin effect and to achieve a high quality factor Q . The measured quality factor of the final coil was 220 at 190 kHz and 130 at 52 kHz, about two times and a half greater than the one that could be achieved with solid wire of the same gauge and geometry.

Tuning the coil was achieved using fixed capacitors and variable ones. A software modulus allows displaying the resonance curve in real time for fine tuning (see paragraph 4.3). A same coil could be easily used for different frequencies. The only modifications are the tuning capacitors. To facilitate these modifications, tuning components could, if desired, plug-in on pin DIP component carriers. The coil is connected to the duplexer by ordinary coaxial cable; there are no tuning elements in close proximity of the coil.

A duplexer is necessary when the NMR coil is used for both transmitting and receiving. During the transmitting period, the duplexer must “blank” the preamplifier avoiding its overloading and its possible destruction by the high power excitation pulses. This same duplexer isolates the transmitter from the receiver during the receiving period. This avoids electrical noises from the transmit section.

Duplexers are usually built using quarter-wavelength lines. However, at low frequency, the length of such lines is very important and their use is not advised, at least from practical point-of-view. Moreover, for such line lengths, the signal attenuation could dramatically decrease the SNR and shielding requirements become more stringent to avoid external interferences.

Duplexers at low frequency have actually not been widely investigated. Here, a structure of duplexer is presented. This structure was inspired from the work in (Saam & Conradi, 1998). Fig. 6 shows the whole electrical circuit of the duplexer associated to the NMR coil and the first stage of the low-noise preamplifier.

The NMR coil inductance L is tuned to the working frequency using parallel fixed capacitor C_T and variable one C_{T-var} . The duplexer is based of the use of a Field-effect Transistor (FET) switch J108.

During the transmitting period, the blanking TTL signal is in high level. The “*Command of the switch*” (based on the bipolar transistor 2N3906) sets the gate voltage of the J108 to 0 V. The FET switch is then in its on-state, putting the preamplifier input to the ground and avoiding its overload. In the case of an eventual dysfunction of the FET switch, an additional protection of the preamplifier is achieved by the limiting crossed diodes D5-D6.

Notice that the capacitor C2 avoids the short circuit of the NMR coil when the switch is in its on-state. Also, during transmitting, crossed diodes D3-D4 conduct, putting C2 in parallel with C_T and C_{T-var} , and the NMR coil would not be well -tuned if an additional inductor L1 is not used. Indeed, this inductor offsets the increased capacitance. The value of L1 should be chosen according to the value of C2 so as the final resonance frequency of the parallel circuit-formed by L , L2, C_T and C_{T-var} and C2- to be closed to the working frequency.

During the receiving period, the “*Command of the switch*” sets the gate voltage of the FET switch to -15 V by charging the capacitor C3. The switch is now in its off-state. Diodes D3 and D4 are blocked. They isolate so the transmit section. On the other hand, they disconnect the additional inductor L2 from C2. This capacitor becomes now just a coupling capacitor to the preamplifier.

Fig. 6. Schematic of the NMR coil, duplexer and the first stage of the preamplifier.

The preamplifier is another key element for NMR signal receiving at very-low field. It is high gain, high dynamic range and low-noise. Several stages, based on the use of the OP37 (or OP27) low-noise op-amp, were associated. In Fig. 6, one can see the first stage which was realized, together with duplexer on the same printed circuit board. Crossed diodes D7-D8 in the feedback network prevent the overload of the following stage. In addition, the high frequency response is rolled off by a 22 pF capacitor (C4).

Optional analog filters could be used in adequate locations between the different stages of the preamplifier to optimize the SNR and/or the dynamic range. The total gain is programmable between 2 and 2000.

The design of low-noise and high gain preamplifiers is relatively complicated and requires some know-how. For developers who could not be familiarized with such development, the author could advise commercial low-noise preamplifiers. For example, the low-noise preamplifier SR560 from Stanford Research Systems is adequate for frequencies up to few 100 kHz.

3.5 The receiving board

The receiving board (NI 5911 from National Instruments) is the last receiving key element. This device is a high-speed digitizer with a flexible-resolution ADC (Analog-to-Digital Converter) and it ensures high sensitivity and high dynamic range. These features were the main crucial criteria in choosing the device for the NMR.

The analog input of the board -that could be AC or DC coupled- is equipped with a differential programmable gain input amplifier (PGIA). This PGIA accurately interfaces to and scales the input signal to match the full input range of the ADC so as to optimize accuracy and resolution. The ADC is 8-bits and is clocked at 100 MHz sampling frequency like a desktop oscilloscope. However, flexible resolution can dramatically enhance the final effective resolution of the ADC.

Full description of the board features could be found in the user manual of the NI 5911.

4. Software development: LabVIEW programs

4.1 Overall view of the developed NMR spectrometer program

The “*Low-field NMR Spectrometer*” program was developed using LabVIEW and associated instrument drivers (NI-FGEN and NI-SCOPE) of the NI 5411 and the NI 5911 devices. The architecture of the program is open which lets users build their own modulus if wanted. The main panel of the Graphical User Interface (GUI) is shown in Fig. 7.

User could choose the frequency, amplitude, and duration of the excitation pulse as well as the repetition time (TR) for a one-pulse NMR sequence. The gain of the low-noise preamplifier should be given when quantitative measurements on the NMR signal have to be performed. Other hardware configurations of the NI 5411 and the NI 5911 are not available in the main front panel, but they could be modified if required in the LabVIEW diagrams.

When the pulse sequence is defined, user can start the NMR experience using the “*NMR Signal Acquisition*” panel. The program allows NMR experiences at any excitation frequency up to 20 MHz. Currently, NMR signal acquisition and measurements are performed on two nuclei: proton (^1H) and xenon (^{129}Xe). The resonance frequencies are of about 190 kHz and 52 kHz, respectively.

Fig. 7. The main front panel of the low-field NMR spectrometer

The developed program contains also two other main functionalities: the “Coil Tuning” and “The Calibration of the Flip Angle”. These functionalities are required for an NMR spectrometer. In fact, each NMR or MRI experiment follows, at least, three fundamental steps in chronological order: coil tuning, calibration of the flip angle calibration of the excitation pulse and NMR signal acquisition and processing

Fig. 8. The general structure of the LabVIEW diagram for NMR spectrometer

In the global program, these functionalities are organized into three *main sub-routines* (*Virtual Instruments VI*) and the whole program is based on LabVIEW *events structure*. A simplified view of the LabVIEW diagram is illustrated by Fig. 8. The three *main sub-routines* are also developed using sub-routines (VI).

Neither *all* of these VI, nor the general LabVIEW programming methods and functions will be presented in this chapter. Indeed, the next sections will be only concentrated on some specific main parts, ideas, advanced data-acquisition and signal processing techniques for the NMR using LabVIEW.

4.2 NMR signal acquisition

4.2.1 Programming sequences for excitation

The arbitrary waveform generator (AWG) NI 5411 is used to generate the excitation. As it was mentioned in paragraph 3.2, two operation modes are available.

A DDS mode uses a reference clock frequency to produce a digital waveform with programmable frequency, amplitude and phase. The DDS produces high frequency accuracy and resolution, temperature stability, rapid and phase-continuous frequency switching as well as low phase-noise. These features are of great value for the NMR. This is why DDS technique has been used in our previous works (Raoof et al., 2002), and it is actually now employed in modern commercial NMR spectrometers and MR imagers. However, the *arbitrary mode*, available with the device, is more flexible. Indeed, the DDS mode is well suited for applications that require continuous generation of standard waveforms that are repetitive in nature such as sine, square and triangular waveforms, etc.

Fig. 9. The concept for creating a one-pulse NMR sequence using the *arbitrary mode*. The lengths of the buffers are programmable and they depend on the sampling frequency.

In a typical NMR/MRI sequence, the excitation pulse(s) must be generated during limited duration(s) within the repetition time TR. Unless the use of additional hardware (like a fast analog switch) in the excitation path, the DDS mode could not be suited since it generates continuous waveform. Arbitrary mode has more features and it is therefore preferred for the generation of the NMR pulse sequences. This mode allows defining waveforms as multiple buffers of samples. These buffers can then be downloaded to the memory of the AWG, linked and looped in any order to form a single sequence. In a one-pulse NMR sequence, at least two buffers are necessary: a sine waveform buffer of limited duration (τ) and a buffer

of zero values. This zero- values buffer could then be repeated to obtain the final repetition time TR between two sine pulses. This feature is particularly useful for the implementation of more advanced NMR /MRI sequences (spin-echo or gradient-echo) where two or more pulses are required. Fig. 9 illustrates the concepts of waveform samples, buffers, linking and looping for a one-pulse sequence.

In addition to buffer 1 (sine waveform) and buffer 2 (zero values), and for the flexibility of implementation, the buffer 0 is used to position the TTL “Marker” signal which will be used to trigger the generation of the TTL synchronization and control signals. This “Marker” can be set at any position (time) of a waveform buffer. This position is specified by giving an offset count (in number of samples) from the start of a waveform buffer.

The creation of samples of each buffer is easily done and straightforward using general LabVIEW functions. These buffers (an array of samples) are then downloaded to the AWG’s memory using the driver functions NI-FGEN of the device. Fig. 10 shows a part of the corresponding LabVIEW code.

Fig. 10. The LabVIEW code for downloading the created buffers in the AWG’s memory.

The waveform linking, looping and the sequence creation are then implemented according to the Fig. 11 of LabVIEW code. When creating the sequence, developer has to define the number of buffers (*sequence length*), the number of *repetitions (loops)* for each buffer. It is also important to enable the “Marker” signal and to route it to a digital output. The *position of the marker* in number of sample of buffer 0 must also be defined.

Other instrument configurations must be defined. These configurations include the *sampling rate* (up to 40 MHz) of the DAC, the use of the *digital filter* before the DAC, the *gain* of the output amplifier and the use of the output *analog filter*. The generation of the analog waveform could then be *enabled*.

Notice that other hardware configurations of the NI 5411 could also be defined (not shown in Fig. 11). The user-manual of the AWG NI 5411 gives you full description of the large number of functionalities offered by the device.

Fig. 11. A part of the LabVIEW code for waveform linking, looping and the sequence creation.

4.2.2 NMR signal detection and processing

After the end on the excitation pulse, the NMR signal from the preamplifier is applied to the analog input of the receiving board. The signal is scaled by the input amplifier (PGIA) of the NI 5911 and then sampled at 100 MHz with a resolution of 8-bits. Our previous development experience showed- and one could verify- that this resolution is far to be sufficient for NMR detection, especially at such low field.

The flexible-resolution of the ADC can dramatically enhance the ADC effective resolution. In this flexible-resolution mode, the ADC is sourced through a noise shaping circuit that moves quantization noise on the output of the ADC from lower frequencies to higher frequencies. A digital lowpass filter applied to the data removes all but a fraction of original shaped quantization noise. The signal is then resampled at lower sampling frequency. In this way, the effective resolution is enhanced. A resolution of 11 bits is obtained at 12.5 MHz of sampling frequency. This same resolution can be as high as 21 bits for a sampling frequency of 10 kHz. In our experiments, a final ADC effective resolution of 14 bits at 5 MHz of sampling frequency was typically used.

The implementation of the NNR signal acquisition and of the flexible resolution is done using the advanced instrument driver functions (NI-SCOPE) of the NI 5911. Fig. 12 illustrates the main concept of the LabVIEW code for the configuration of the NMR signal acquisition with flexible resolution. Acquisition is hardware-triggered by the trigger signal applied to the PFI1 digital input of the device.

Fig. 12. A part of LabVIEW code for the configuration of the NI-5911 in flexible-resolution acquisition mode.

After acquisition with flexible resolution, data is read from the device memory and additional digital signal processing is performed by software. This processing is still necessary despite the flexible-resolution acquisition. This is why the digital data is fed to bandpass digital filter (Butterworth filter) with programmable bandwidth. The use of this filter is optional but it could be of great importance in noisy environments. In our experiments, the usefulness of this filter was still limited. After bandpass filtering, a quadratic demodulation or digital-down-conversion (DDC) is performed as it is shown in Fig. 13.

The DDC is achieved by multiplying the digital NMR signal (with N bits of effective resolution) by digital sine and cosine waveforms at the working frequency. Digital lowpass filters (Butterworth) -with programmable cutoff frequency and order- are applied. The quadratic base-band signals I/Q (In-phase and Quadratic signals) could then be obtained. However, since the useful bandwidth of these base-band signals should be no more than few hundreds Hz (depending mainly on the homogeneity of the static magnetic field), decimation technique with programmable decimating factors was implemented to improve the final effective resolution, dynamic range and the signal-to-noise ratio (SNR). In fact, the SNR is inversely proportional the total bandwidth. In digital systems, the useful bandwidth is limited by the final sampling frequency. Decimation decreases the sampling frequency

and, when combined with *dedicated lowpass filtering*, improves the final SNR and dynamic range.

Fig. 13. Down-sampling and Digital Down-Conversion (DDC) of the NMR signals.

The simplified principle for implementing the DDC and decimation using LabVIEW is described in Fig. 14.

Fig. 14. Simplified Labview diagram for reading data from the NI 5911 memory and performing the digital down conversion (DDC) and decimation.

In Fig. 15, the “NMR Signal acquisition” Panel is shown. The user could choose the acquisition parameters such as the sampling rate for the flexible-resolution acquisition (The obtained effective ADC resolution is displayed for information), the cutoff frequencies and orders for the filters, the decimation factor, the trigger source, the scaling gain of the PIAG of the receiver, and the number of sample to be acquired, etc. Starting the NMR pulse sequence is achieved by the event “send pulse” and data is acquired and displayed according to the defined parameters.

Fig. 15. The panel “NMR signal Acquisition”.

Fig. 16. The baseband I/Q parts of the ¹H NMR signal obtained at 4.5 mT with 10 signal averages.

In addition to all these advanced signal procession techniques (flexible-resolution, DDC with decimation), NMR signal averaging (i.e. multiple acquisitions of a same signal) is still necessary to obtain a signal with a measurement quality. Fig. 16 shows the I and Q parts of the ^1H NMR signal of a sample of pure water (about 100 milliliter). About 10 averages were used. These baseband signals are acquired with 500 Hz of off-resonance (difference between the Larmor frequency and the frequency of the excitation pulse). A 90° pulse of 0.8 ms was used. The repetition time was of 2500 ms and the gain of preamplifier was set to 1000. Data were acquired with a resolution of 14 bits (5 MHz of sampling frequency before the DDC). For decimation, a decimating factor of 500 was used. The final sampling frequency was about 10 kHz, and the positive useful bandwidth was of 5 kHz.

Additional signal analysis such as Fast Fourier Transform (FFT) as well as different measurements could then be performed in real time. For example, Fig. 17 shows the magnitude of the FFT of the NMR signal of Fig. 16.

Users could easily add more modulus in the program to perform other kinds of measurements on the signal or on its spectrum. Such measurements could be performed using general LabVIEW functions.

Fig. 17. Magnitude of the FFT of an-off resonance the NMR signal in the baseband.

4.3 Tuning the NMR coil

For a given tuning capacitor value, the resonance frequency of the NMR coil is generally shifted when the coil is placed inside the magnet and loaded by the sample. One might argue that this shift, resulting from capacitive coupling between the coil and the sample, could be small at low frequency. However, even at frequencies such as 190 kHz or 52 kHz, where the SNR of the detected NMR signal is inherently very low, we believe that it is still important to optimize the tune the coil for each sample and inside the magnet. The shift in the resonance frequency could dramatically cause losses in the SNR. Therefore, tuning the

coil must still the first fundamental step in each NMR/MRI experiment. The NMR spectrometer must then allow tuning the coil in real experimental conditions. A simple method for tuning the coil is illustrated in Fig. 18. The NMR coil is excited by a continuous sine wave through the mutual coupling with coil 1. The response of the resonant circuit is measured by the pickup coil 2. It is important that excitation and pick-up coils be placed sufficiently far from the resonant circuit to avoid any modification of the resonance frequency which could be due to the mutual coupling between the coils.

Fig. 18. A method for tuning the NMR coil inside the magnet.

The AWG NI 5411 was used in its DDS mode to generate sine waves of different frequencies. Each frequency is generated during a given duration. During this duration the receiver NI 59 11 was used to acquire the coil response.

Fig. 19 shows the “Coil Tuning” front panel where one can see an example of the resonance curve of a coil. User can choose and modify in real time some parameters like the frequency interval or resolution. The current resonance frequency, the bandwidth as well as and the quality factor of the coil are displayed in real time.

Fig. 19. “Coil Tuning” front Panel

The implementation of this procedure is accomplished using the instrument drivers of the NI 5411 and the NI 5911 and general LabVIEW functions. The use of the DDS mode was useful and quiet adequate for this purpose. The related driver functions could be easily used.

4.4 Calibration of the Flip angle

The third required functionality an NMR spectrometer is the “*Flip angle Calibration*”. For a given sample, it is crucial to know, for a given pulse duration and amplitude, the resulting flip angle. In a one-pulse sequence, the maximum NMR signal is obtained at a flip-angle of 90°. More sophisticated NMR/MRI sequences require knowledge and adjustment of an optimum flip angle.

A LabVIEW modulus was developed to perform this calibration. Fig. 20 shows the front panel of this modulus.

Fig. 20. The front panel for the “*Calibration of the Flip Angle*”

For a given pulse duration, a one-pulse NMR sequence is repeated for various pulse amplitudes. The NMR signal is measured for each excitation pulse. The maximum of the obtained curve corresponds to the 90° flip angle. For flip -angles greater than 90°, the NMR signal will decrease (not shown on the Fig. 20).

5. Examples of applications and discussions

5.1 Polarization measurements of hyperpolarized gases

The field of applications of this spectrometer is wide. A first application consists in measuring the *polarization*, P , of hyperpolarized gases. For a basic understanding of this parameter, one can say that the NMR signal, S , is related to the magnetization M and to the polarization by the equation (3), (Saam & Conradi, 1998, Asfour, 2010):

$$S \propto M \propto N.P \quad (3)$$

where N is the number of nuclei within the sample.

During the last decade, the MRI of hyperpolarized gases, such as ^3He and ^{129}Xe , had become widespread for a large palette of applications, especially medical research and clinical diagnosis. For example, the proprieties of xenon in biological environment make it a promising MRI probe for brain physiology and functional studies (Asfour, 2010).

Hyperpolarization is a technique which allows compensating the intrinsic low levels of NMR signal of such gases (when compared with the ^1H signal). Indeed, at equilibrium, and for a same magnetic field B_0 , the NMR signal of a ^{129}Xe population is about 10000 lower than the one that could be obtained from the same volume of protons. This is because of the intrinsic lower gyro-magnetic ratio and lower density of the xenon.

The Hyperpolarization process can dramatically increase the polarization level of the gas before using it for the NMR or MRI in-vivo experiments. Consequently, the magnetization and the NMR signal levels are typically enhanced by a factor 100000. For a same volume, the NMR signal of the hyperpolarized gas becomes more important than the proton signal.

In our laboratory, the ^{129}Xe is hyperpolarized by spin-exchange with Rb optically pumped by laser at 795 nm. About 0.1 g of Rb is introduced in a 100-ml Pyrex container (cell), which has subsequently filled with a mixture of helium, nitrogen and ^{129}Xe at 5 bars at room temperature. The cell is heated to about 120°C , set in a 4.5 mT magnetic field produced by *Helmholtz coils*, and exposed to a circularly polarized laser. In few minutes, about 20 ml of hyperpolarized xenon can be obtained to be used for in vivo MRI experiments (generally at high field). Monitoring the available *polarization* of the gas during the optical pumping and at the end this process is critical. In fact, one must be able to quantify the effects of changing temperature, pressure, gas mixture, laser power, etc. The goal is to guarantee a maximum polarization or to diagnose eventual problems. The quantitative measurement of the polarization, during and at the end of the pumping process can actually be performed by NMR, since the gas being polarized is subjected to the 4.5 mT static magnetic field. The Larmor frequency f_0 for this field is about 52 kHz.

The measurement method of the *polarization* requires comparing the levels of hyperpolarized ^{129}Xe NMR signal to another reference signal such as a ^1H signal, (Saam & Conradi, 1998, Asfour, 2010).

The dynamic range of our systems allowed the detection of both the ^1H and hyperpolarized ^{129}Xe signals. Fig. 21 shows the quadratic base-band NMR signals of hyperpolarized acquired at 4.5 mT with the developed spectrometer the hyperbolizing process.

The sample (pyrex container or cell) has the same shape and the same volume that the one which was used to acquire the ^1H signals of Fig. 16. These signals were collected by the same coil that was used for collecting the ^1H signals and which was retuned to 52 kHz by simply modifying the tuning capacitors. The gain of the preamplifier was set to 2 and no signal averaging was used. The excitation pulse duration was of the 800 μs with a repetition time of 2 seconds. The gain of the low-noise preamplifier was set to 2. The sampling frequency of the received signal before the DDC was of 5 MHz so as the effective ADC resolution was of 14 bits. The final sampling frequency in the base-band was of 10 kHz.

Comparing the NMR signals of ^1H and hyperplorized ^{129}Xe allow determination of the polarization P of the gas (Asfour, 2010). The system described here has been in use in our lab for some years and gives a reliable measurement of the polarization.

Fig. 21. The baseband I/Q parts of the hyperpolarized ^{129}Xe NMR signal obtained at 4.5 mT. No signal averaging was used.

5.2 Other applications

In addition to the measurement of the polarization, there are other potential applications of this spectrometer. These applications may especially concern NMR non-invasive measurements. A first application could be the measurement of the quantity of water contained in a given sample since the NMR signal is proportional to the density of water within the sample. One could then quantify the degree of humidity of the sample. This application could be generalized to concern the quality control of food product, etc.

A second application could concern the measurement of both transversal T_2 and longitudinal T_1 relaxation times of the sample. This could find application for temperature measurement of biological samples. Actually, NMR is a very important technique for measuring temperature of a sample in millikelvin and below through the temperature dependence of the spin-lattice relaxation time T_1 or through the measurement of magnetic susceptibility (Pobell, 1991). These measurements should be performed at frequencies below 1 MHz to minimize power dissipation in the sample. Pulse sequences for such measurements could be easily implemented on our system without hardware modifications.

Another potential application of this non exhaustive palette of applications may concern educational purposes in the several areas: electronics, signal processing and biomedical engineering, etc. This is allowed thanks to the flexible and open structure of both hardware and software of the spectrometer. We are currently building another spectrometer in the Department of Physics Measurements at the Grenoble University. Practical courses and projects in electronics, signal processing, instrumentation and measurements, NMR physics could be take place during and after the development of the spectrometer. Students could simulate and build the Helmholtz coils, the power amplifier, the duplexer, the coil and the preamplifier. They could also develop their own applications for the control of the NMR sequence and the data acquisition using LabVIEW.

Notice finally that this low frequency NMR spectrometer could also be dedicated for other applications. It is low cost and easily transportable (for in situ experiments).

6. Conclusion

A DAQ- and LabVIEW-based NMR spectrometer working at low field was presented. This spectrometer allowed detection of the NMR signals of both ^1H and ^{129}Xe at 4.5 mT. The flexibility of the system allows its use for a palette of NMR applications without (or with minor) hardware and software modification. It is also easy-to-replicate.

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