Hardware and Software for Embedded Compact Broadband Low Field NMR spectrometers (ECBLFNMR)

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Abstract

Numerous compact NMR spectrometers have been designed for an easy measurement of proton NMR spectra. High sensitivity and resolution can be reached even with low field spectrometers (LFNMR) (*i.e.* 60 MHz), thanks to great improvements in electronic hardware, which open up a wide field of analytical quantification and relaxation applications. A specificity of Low field NMR spectrometer is the use of a permanent and cryogen free magnet technology, avoiding the need for weekly and expensive cryogenic services.

Here we present and describe a low field NMR spectrometer fabricated in our laboratory. This spectrometer (ECBLFNMR) operates at basic resonant frequencies ranging from 1 to 60 MHz, with standard sample diameter (5-10 mm). All the embedded hardware is very compact and requires only a 24 V DC power supply, so this spectrometer is portable, easy to install and has a small footprint. This ECBFLNMR is dedicated to education and quantification, and enables low-field NMR research. It may be coupled with scientific experiments not requiring high magnetic fields.

Keywords

Low Field NMR, Mobile NMR, FPGA, ARM microprocessor

1. Introduction

Nuclear magnetic resonance (NMR) is nowadays an analytical technique that is very powerful and unavoidable in many fields. The non destructive nature of the NMR makes it a privileged means to analyze the media in chemistry, biology and medicine on the human body (MRI).

Since the methods of Rabi and Bloch and Purcell, NMR spectrometers have constantly evolved over years, both conceptually and technically. Today, the NMR apparatus has become a very high-tech scientific instrument with the latest advances in electronic and scientific research. Stability, precision, great dynamics and acquisition sensitivity are, among other things, the qualities required for an efficient spectrometer. NMR is a multi-pulse Fourier transform technique, with multi-nucleus and multidimensional experiments. For a detailed understanding of NMR the reader may refer to references [1] to [5] in this article.

In this paper we describe specifically the construction of a very compact NMR spectrometer for low field applications. We call it ECBLFNMR for : Embedded Compact Broadband Low Field NMR spectrometer.

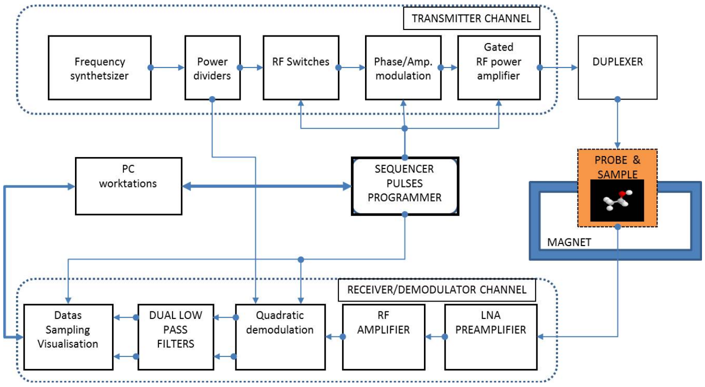


Figure 1– Block diagram of a general NMR spectrometer.
(shims and lock compensation are not shown).

A basic block diagram of a complete Modern Fourier transform NMR spectrometer (FTNMR) is shown in Figure 1. It can be broken down into several major blocks, that we briefly describe, before focusing on their implementation in our embedded electronics.

1.1. Transmitter channel

This is a transmission block whose main function is to excite the spins by means of hard power radio-frequency (RF) pulses. This unit gives the basic reference frequency of the spectrometer. The transmission channel contains a first stage for synthetizing signals whose frequency range extends from MHz to several hundreds of MHz: typically from 1 MHz to 500 MHz for our broadband spectrometer. This reference signal is then divided in order to feed the receiving part synchronously. The radio frequency (RF) pulses are realized by controlling fast switches (fast diodes). A special unit allows the phase and the amplitude of the RF to be modulated. It can be carried out using controlled mixers, or included in the frequency synthesis. Finally a RF power amplifier of the order of ten watts attacks the duplexer placed before the probe.

1.2. Duplexer

The main purpose of the duplexer is to isolate the transmission channel from that of the reception in order to avoid destruction of the input preamplifier. Indeed the NMR probe is the same device for the excitation and the detection of the signal. So we need a duplexer for routing signals between the transmission channel, the receiving channel and the probe.

1.3. Receiver Channel

This reception block detects the signal from the NMR probe and ensures conditioning for subsequent processing. The reception channel comprises a first chain consisting of two stages of preamplifiers in order to optimize the sensitivity. A first low noise preamplifier device with a very low noise factor and a second stage with a general purpose RF amplifier (GPA).

After amplification, the signal undergoes a synchronous demodulation in order to obtain two quadrature components. Each channel is then filtered, to retain only the low frequency part from 0 to some 500 kHz. The signal is finally sampled by a fast ADC converter and stored in memory, for real-time acquisitions. The spectrometer is usually connected to a computer (PC) for data processing.

1.4. Magnet and probe

The structure of the magnet defines the specificities of the spectrometer: low field or high field. For low field we used permanent magnet or inductive device (also called resistive). The detection probe consists of inductive coils and their matching and tuning capacitances networks.

1.5. Pulses sequencer programmer

This element is crucial in NMR spectrometers, because most units of a NMR spectrometer are driven by sophisticated pulse sequences. This sequencer generates the series of pulses of the multi-pulse NMR programs.

2. Hardware description of our compact NMR

The block diagram of the electronics units in our ECBLFNMR spectrometer is displayed in Figure 2, and shows how the functions, previously described in the diagram block of classical NMR spectrometer are implemented.

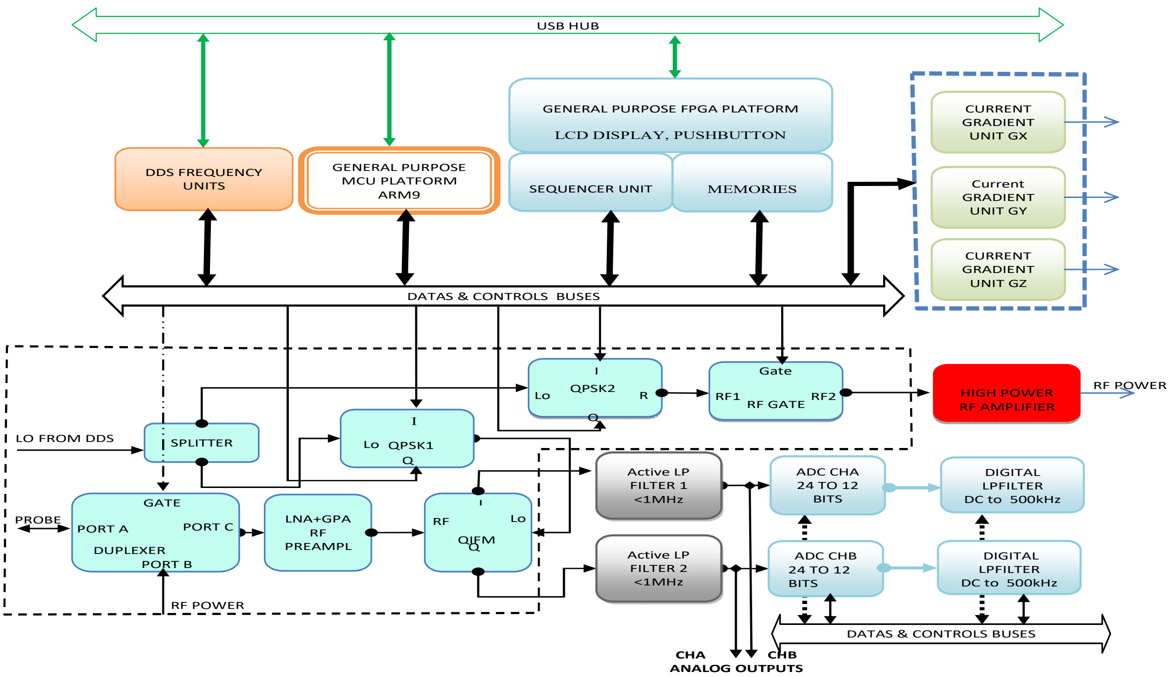


Figure 2 : *Hardware Block diagram of the ECBLFNMR spectrometer.*
It includes Direct Digital Synthesis (DDS) for radio frequency (RF) signal generation. Recent technologies of FPGA, Digital Signal processing and ARM-CPU are used for the control of acquisition and processing of data. Gates and switches are realized with fast diodes. LNA is a low noise amplifier with a noise factor of 2 dB and a gain of 20 dB. GPA is a general purpose RF amplifier with a gain greater than 20 dB; Phase modulation may be done by the two QPSK devices (Quadrature Phase Shift Keyed Modulator QPSK 1 & 2). Demodulation of the signal is achieved with QIFM device (quadrature Intermediate Frequency Modulator).

It includes all the embedded electronic hardware and software either for the transmission or the receiver channel. A Direct Digital signal Synthesis (DDS) is used to generate the frequency. The pulse sequencer is realized with a powerful Field Programmable Gate Area

(FPGA Cyclone II or IV) that operates rapidly with a time base of about twenty nanoseconds (typically 25ns). It generates all the phase, amplitude, frequency and waveform parameters required by the pulses program. The pulse programmer controls the phases of the RF pulses via the two modulators QPSK 1 & 2. A portion of the FPGA is also used to perform digital filtering of data.

Recent ARM microprocessor devices may be used for the general control of the spectrometer and interfacing with computer. A microcontroller platform can accommodate different types of microprocessors for data processing as well as for general interfacing. The ECBLFNMR can work with an ARM NXPLPC1768, an ARDUINO component or a REDPITAYA card.

The technology of the duplexer in our ECBLNMR is based on fast radiofrequency switches devices for broadband frequency use. The duplexer is controlled by the pulse sequencing unit. A four quadrant phase modulation can be done both at excitation or at the reception for phases cycling. This is achieve thanks to the two quadrature phase shift keyed modulateurs QPSK 1 & 2 controlled by the pulse programmer. The quadrature analog components (I and Q) are filtered and available on BNC outputs for oscilloscope display. The analog signals are also digitized by a set of analogs to digitals converters (ADC) offering various resolutions depending of the frequency range required: 24 bits up to 0.5 MHz, 16 bits/0.1 MHz, 14 bits/20 MHz and 12 bits/10 MHz. The radio frequency high power amplifier, for the hard RF pulse, is a unit ZHFL-5W with a power of 5 watts operating in the range from 1 MHz to 500 MHz. The compact spectrometer comprises a current control unit with three outputs (Gx, Gy, Gz) for NMR applications using gradients. This unit is designed to control currents of maximum amplitude 6 A. We perform 1H solenoid probes for low field NMR applications in the laboratory. The embedded hardware of our ECBLFNMR is very compact and requires only a 24 V DC power supply, so this spectrometer is portable, easy to install and has a small footprint. Electronics are mounted on two levels in a space reduced to a 6U frame.

3. Software description

The spectrometer is designed to function autonomously, *i.e.* with a hexadecimal keyboard and a LCD screen. In that case all the code (VHDL for the FPGA and C language for the microcontroler) are compiled and loaded in the onboard eeprom devices. That is the stand alone user mode. The data may be viewed on a simple ocilloscope.

The ECBLFNMR may be connected to a PC via only one USB port for the control and processing of experiments, but also for new developments. As mentioned earlier, the spectrometer includes many programmable elements (FPGA, ARM, DDS, ADC board ...). Each of these elements has a specific development environment interface (IDE) such as Quartus, Eclispe, Mbed, LabView, etc. The processing and analysis of NMR data can also involve special software (MATLAB, MestReNova. ..). It is therefore essential to have easy access to these features. We have developed a general purpose NMR Interface (see Figure 3) that unifies, in a single window, all the software (IDE) needed for NMR experiments and ECBLFNMR developments. Once launched, the GPNMRI interface automatically detects, by recursion, the different programs used to operate the compact spectrometer (ECBLFNMR). It then offers, in a window, programs that can be opened with a single click. The software is developed in Java and contains about 800 lines of code. This gives a jar executable file of reasonable size (1.5MB) and portable on all platforms of operating systems (Windows, Mac OS and Linux). It can easily be configured to integrate programs other than the initial ones.

The interface allows to launch NMR pulses sequences, to visualize and process the data or to develop new sequences (see Figure 3).

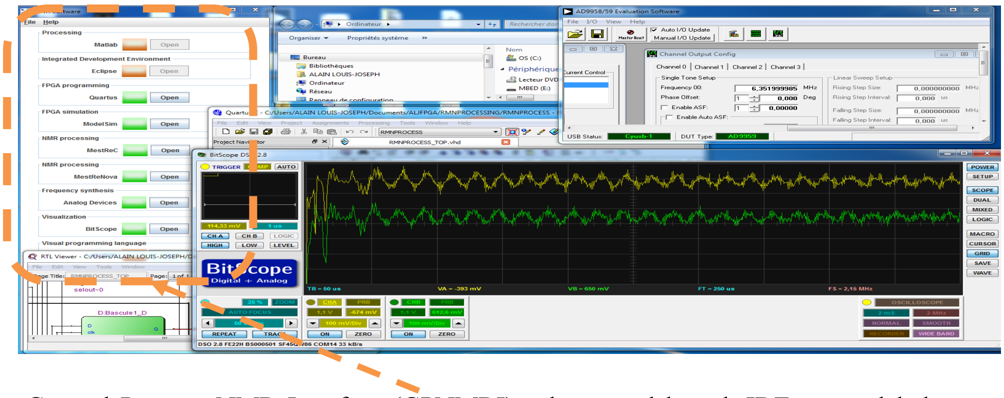
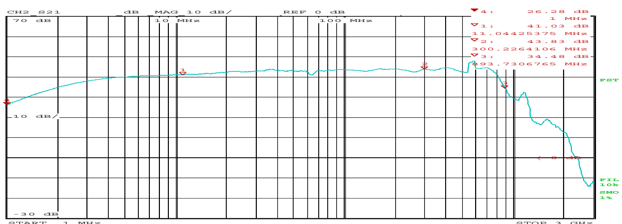


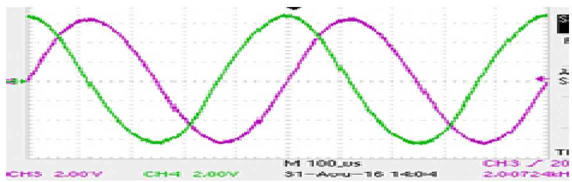
Figure 3 : General Purpose NMR Interface (GPNMRI): detect and launch IDE: green label means available software, red label is for software not detected. Here is an example of the general purpose interfacing tool used to manage the various software required for the use and development of the compact NMR spectrometer: download of standard programs on the ARM microprocessor, control of the basic frequency of the Spectrometer, VHDL code development for FPGA, and real-time visualization of I and Q free induction decay (FID) signals.

4. Tests and results

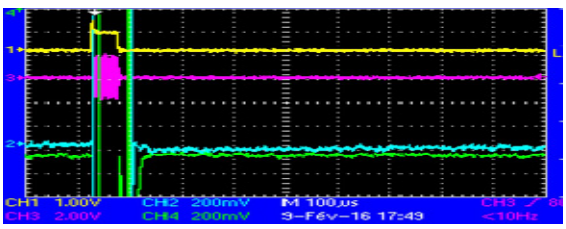
We tested our ECBLFNMR with a proton probe (1H), designed by us, and a permanent magnet just over 1500 Gauss (0.15 T). The resonant frequency is around 6.4 MHz. The detector coil is a solenoid with its network of capacitances for impedance matching and frequency tuning. The test sample is a tube of diameter 10mm, filled with water over a height of about 20mm. The measurement signals are displayed on an oscilloscope and on a network analyzer. The low-noise RF preamplification chain is broadband frequency and offers a gain of about 40 dB at 300 MHz. The frequency response of the two stages RF preamplifier is given in (Figure 4a).



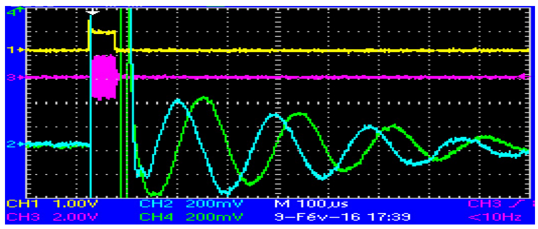
a) Frequency response of the preamplifier chain in the frequency range from 1 MHz to 3GHz.



b) TEST Mode: I&Q components after demodulation for a basic frequency range from 6 to 13 MHz.



c) Pulse acquisition sequence: NO Sample in the magnet. Yellow, TTL pulse from FPGA; Purple, RF excitation pulse; Blue & green, I &Q channels. NO FID.



d) Pulse acquisition sequence, with a water sample in the magnet. Observation of the FID's components.

Figure 4 : Tests and set-up of the NMR spectrometer

An internal test mode for generating frequencies allows to adjust the quadrature of the channels over a range of frequencies predefined by the user. We show an example of quadrature optimization obtained for a basic frequency range from 6 to 13 MHz (Figure 4b).

The I & Q components are demodulated at an offset of 2 KHz. Note that during this test mode we also adjust the gains and voltage offsets of the two channels in quadrature.

A Pulse-Acquisition basic sequence is then performed, and the signals are displayed on an oscilloscope (Figure 4c & d). The radio frequency pulse is of order of 50 µs. Without water sample in the probe, we have only the floor level of noise of the electronic on the two components (I&Q) (Figure 4c). With a water sample in the probe, the components of the NMR free induction decay (FID) are obtained with an excellent signal to noise ratio in a single scan (Figure 4d).

3. Conclusion

We have detailed the construction of a compact, operational and efficient low-field NMR spectrometer (1 to 60 MHz). Among the numerous commercial low-field NMR spectrometers [6] on the market, this is an alternative solution at very low cost and entirely open to users. In the field of existing designs [7], our realization is a new generation of very compact low field versatile laboratory NMR spectrometers, with FPGA, ARMs, LNA and ADCs up to 24bits for high resolution, including also RF power amplifier and gradients units. This ECBLFNMR is dedicated to teaching modern pulses NMR & MRI techniques [8][9][10] and to quantitative applications. The applications of such a mobile device are multiple: two field NMR spectrometer, T2 or T1 relaxation measurements, and low field characterization of solutions. It also enables low-field NMR research [11] as it can be easily coupled with other scientific instrument for dynamic polarization enhancement research. It can be use either with a permanent magnet for low field NMR applications or with superconducting magnets for high field resolution. A multi-platform software solution was developed for interfacing. The GPNMRI is easy to use, allowing access by different IDE (Interface Development Environment) for processing and development (Matlab, Quartus, ModelSim, Mbed, MestReNova, Eclispe,…). The ECBLFNMR is fully user programmable. Users can either design their own pulse programming NMR sequences or load/customize ones.

The ECBLFNMR has a small footprint and holds on a desk. Broadband, the unit can operate with a base frequency (Larmor) ranging from 1 MHz to 60 MHz. It can be powered from a 24v battery for outdoor mobile applications.

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