Development and implementation of the NMR-spectrometer on the basis of the National Instruments technologies

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Abstract. The quality of lubricating oil in mechanical engineering, technology of creation of units, in particular in equipment of transmission gears, is a factor which considerably defines reliability and safety of the whole propulsion system or the greased constructive components. There are many soluble oil additives such as, for example, different additives for extreme compression conditions or additives against wear. Additives are used with mineral oils, products from mineral oils or synthetic oils for lubricant action or chemical properties improvement. The most exact way of definition of the chemical composition of a substance at the moment is the method of nuclear magnetic resonance (NMR). In the first section of this article, a brief and very simplified review of the NMR basic principles using classical physics is provided. The second section is focused on the description of the hardware solutions and the architecture of the NMR spectrometers. The software developments (LabVIEW programs) of the data-acquisition and signal processing techniques are presented in the third section. At the end, results of measurements are provided.

1. Introduction

The quality of lubricating oil in mechanical engineering, technology of creation of units, in particular in the equipment of transmission gears, is a factor which considerably defines reliability and safety of all propulsion systems or the greased constructive components. There are many soluble oil additives such as, for example, different additives for extreme compression conditions or additives against wear. Additives are used with mineral oils, products from mineral oils or synthetic oils for lubricant action or chemical properties improvement. The difference in lubricating oil production technologies is a decisive criterion in competitive fighting. The experience of transmission gears operating shows that even best oils degrade and are subject to change. Thus, there is a gradual transition from planned intervals of oil change to oil change terms depending on oil condition. A criterion is the classical analysis that determine physical and chemical properties of lubricating oil. For example, for wind-power installations, there is a standard instruction according to which it is necessary to take samples of oil and check quality in a laboratory. If parameters of oil worsen, lubricating oil is replaced for safety reasons. A well equipped analytical laboratory and exact sampling time are necessary to determine the corresponding time of oil change.

The most exact way of definition of a substance chemical composition at the moment is the method of nuclear magnetic resonance. Typically, high field NMR systems have many advantages, such as a
high signal-to-noise ratio (SNR), resolution and high image quality. Actually, in many cases for particular purposes, only NMR spectrometers or MR imagers with a subset of some standard commercial features are needed [1]. In addition, using of medium fields (below 0.5 T) is more sufficient in some cases. The cost of the system can then be reduced since these medium fields - with, sometimes, relatively poor performances requirements - could be easily produced. Moreover, the use of medium fields simplifies the design and realization of compact NMR systems which could be especially appreciated for the in situ applications.

A number of groups have worked to develop dedicated NMR systems by using compact MR magnets. For example, a home-built and fully digital system working at 0.1 T (resonance frequency is of about 4.25 MHz) [2]. The system was based on the use of a high-performance Digital Signal Processor (DSP), a direct digital synthesizer (DDS) and a digital receiver. Based on this work, Shen [3] proposed another system working at 0.3 T and allowing larger imaging sizes than in [2]. Another work, carried out in [4], 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 for the specific application of measurement of the polarization for the NMR of hyperpolarized gases (129Xe, 3He etc.). 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 [5]. 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 [5], 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 the sufficient dynamic range to detect the NMR signal of the proton (1H) in such field. In any case, dedicated NMR systems are still far from the experience of most NMR groups. Some groups developed low-field NMR spectrometers that allow detection of the 1H NMR signals at 4.5 mT [6, 7]. These systems are based on National Instruments data-acquisition boards (NI DAQ). These boards are adequate at low frequencies. Moreover, the increased performance and the related software (LabVIEW) are user-friendly and quite easy to use. In these new NMR spectrometers, many analog electronic components were replaced by NI DAQ boards and software. It was shown that the use of advanced data-acquisition and signal processing techniques allow 1H NMR signals detection at 4.5 mT.

The aim of this research is to present advances of standardized National Instruments board and software LabVIEW in the development of medium-field NMR systems. One of the underlying ideas of this research is to make these systems versatile and easy-to-replicate so as to help developers and research groups to realize NMR spectrometers with flexibility, low cost and minimum development time. This includes both hardware design and software developments.

In the first section of this article, a brief and very simplified review of the NMR basic principles is provided using classical physics. The second section is focused on the description of the hardware solutions and the architecture of the NMR spectrometers. This architecture is mainly based on the use of the signal generator and boards from National Instruments. The software developments (LabVIEW programs) of the advanced data-acquisition and signal processing techniques are presented in the third section. The last section will concentrate on results and discussions. The use of the developed system for the measurement will be particularly illustrated.

2. Materials and methods

When a sample, consisting of NMR-sensitive nuclei (1H, 3He, 129Xe; 23Na, etc.), is subjected to uniform static magnetic field \(B_0\), net macroscopic magnetization \(M\) of the sample appears in the same direction of \(B_0\). This magnetization is proportional to this polarizing field \(B_0\) to the density of nuclei within the sample and to characteristic gyro-magnetic ratio \(\gamma\) of the nucleus being studied. In a typical one-pulse experiment, the sample is subjected to a short pulse (called excitation pulse) of radiofrequency magnetic field \(B_1\), applied perpendicularly to \(B_0\) and at characteristic Larmor frequency
For the proton \(^1\text{H}\) nucleus, this frequency is about 42.25 MHz at \(B_0 = 1\) T and about 21.125 MHz at 0.5 T.

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 angle \(\alpha\). This angle is called a “flip angle”. It is proportional to field \(B_1\) and to its duration, \(\tau\), according to the equation: \(\alpha = \gamma \cdot \tau \cdot B_1\).

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 magnetization \(M\) (then to \(B_0\)) and to \(\gamma\), is processed for usage while obtaining the studied nucleus.

The flip angle can be set through the adjustment of the amplitude, \(B_1\), or/and the duration, \(\tau\), of the excitation pulse. For a one-pulse sequence, the maximum NMR signal level is obtained at flip angles of 90° and 180° [8]. One should also know that a variety of parameters contribute to 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 the 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 it depends on the nucleus of interest (through gyro magnetic ratio \(\gamma\)).

3. The implementation of the hardware structure for medium-field NMR systems

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

![Figure 1. The block scheme of the developed spectrometer NMR.](image)

The system is based on industrial computer NI PSle-1078. Static field \(B_0\) of about 0.5 T is produced by a permanent magnet and magnetization coils on the magnet core from electric steel. The excitation pulse (at about 21.125 MHz for \(^1\text{H}\)) is generated by the Digital-to-Analog Converter (arbitrary waveform generator board NI 5442 of National Instruments). This pulse is amplified by a power amplifier and sent to the well-tuned coil (at the working frequency of 21.125 MHz for the \(^1\text{H}\)) which generates 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. The amplified signal is then received by the receiving board (A digitizer board NI 5154 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 5442. 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.

In 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 it 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. However, these magnets can be divided into two categories: permanent magnets and electromagnets. 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 [9, 10]. Another disadvantage, which is the imperfections of the magnetic materials, may increase complexity of implementation. Electromagnets [11] can offer an alternative solution. Typically, the obtained field strength could be as high as 0.5 T. The hybrid system of creation of the magnetic field based on the use of permanent magnets and additional magnetizing coils is offered. Magnetizing coils are applied on the pole core of 10x10 cm. The model of the magnetic system is presented in figure 2.

![Figure 2. The model of the magnetic system.](image)

For the solution of the problem of creation of an adequate model of the magnetic system, the effective approach of natural model trials provided in [12] has been used. The developed strategy of the solution of the return task has allowed solving the problem of identification of the magnetic system, optimization and implementation of its parameters.

4. Development and realization of the software: LabVIEW program
The ‘NMR Spectrometer’ program was developed using LabVIEW and associated instrument drivers (NI-FGEN and NI-SCOPE) of the NI 5442 and the NI 5154 devices. The architecture of the program is open which allows users to build their own modulus if needed. The main panel of the Graphical User Interface (GUI) is shown in Figure 3. The program allows investigating a sample by ‘90° impulses’ and ‘180° impulses’ techniques.
Figure 3. The main panel of the Graphical User Interface.

The algorithm of the program work is shown in figure 4.

The 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. Hardware configurations of the NI 5442 and the NI 5154 are not available in the main front panel, but they could be modified if required in the LabVIEW diagrams.

In figure 5, the results of research of the developed system, where a 180° impulse use allow one to exclude influence of electromagnetic properties of the FID coil, are presented by the example of isopropanol studiyng.
Figure 5. The result of isopropanol spectral measurement: a – received by means of NMR on the basis of superconducting-coil electromagnets of high resolution; b – 0 received by means of the developed NMR device.

The peak of frequency is the share of 20.033 MHz and 20.047 MHz. It should be noted that charts of exemplary FID isopropanol (received by NMR on the basis of superconducting-coil electromagnets of high resolution) and ours are very similar.

5. Conclusion
A National Instruments boards and LabVIEW-based medium field NMR spectrometer working were presented. This spectrometer allows the detection of NMR signals of $^1$H at 0.5 T. The flexibility of the system allows its use for a variety of NMR applications without (or with minor) hardware and software modification.

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