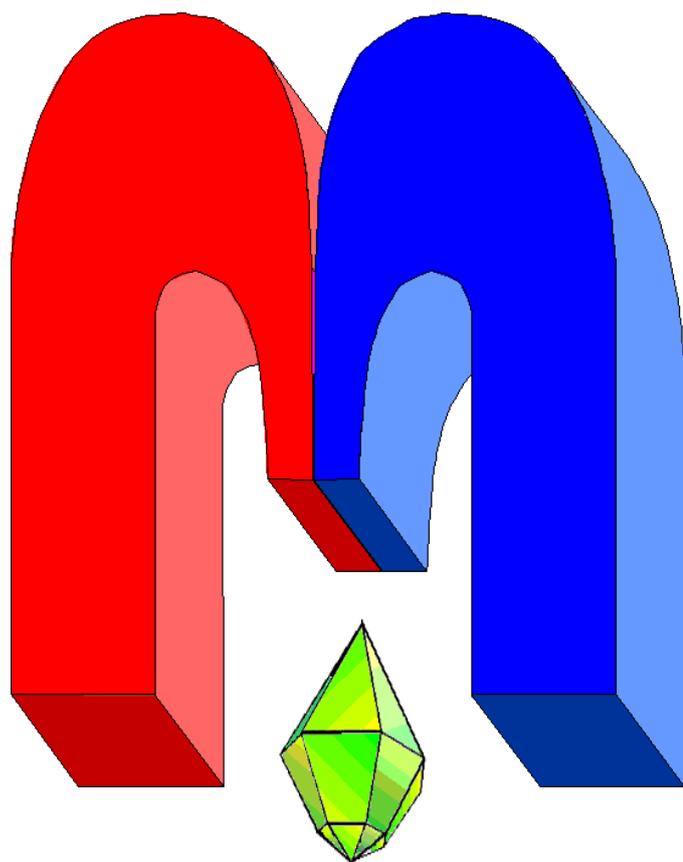


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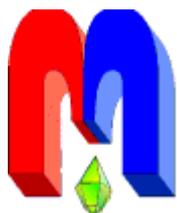
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In Kazan University the Electron Paramagnetic Resonance (EPR) was discovered by Zavoisky E.K. in 1944.

# The home-built pulse nuclear magnetic resonance spectrometer with digital quadrature detection for $^3\text{He}$ research at low temperatures

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The home-built pulse nuclear magnetic resonance spectrometer for  $^3\text{He}$  research is described. The temperature range is 1.5 – 4.2 K, the frequency range is 3 – 20 MHz, the dead time is 10  $\mu\text{s}$  at 10 MHz. The spectrometer software is based on programming language LabVIEW. The advantages of digital technique are described; particularly the implementation of digital quadrature detector.

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**Keywords:**  $^3\text{He}$ , nuclear magnetic resonance, spectrometer, low temperature, digital detection

## 1. Introduction

The nuclear magnetic resonance (NMR) of  $^3\text{He}$  in contact with porous media could give an unique information about structure, porosity and pore-size distribution of the media [1-4]. It was found [5] that the magnetic relaxation of liquid  $^3\text{He}$  in confined geometry acquires new features compared with the relaxation in bulk liquid, and a new mechanism of magnetic relaxation has been proposed. Thus, the longitudinal relaxation time, the transverse relaxation time and the spectral line width strongly depend on the size of the volume filled by liquid  $^3\text{He}$ .

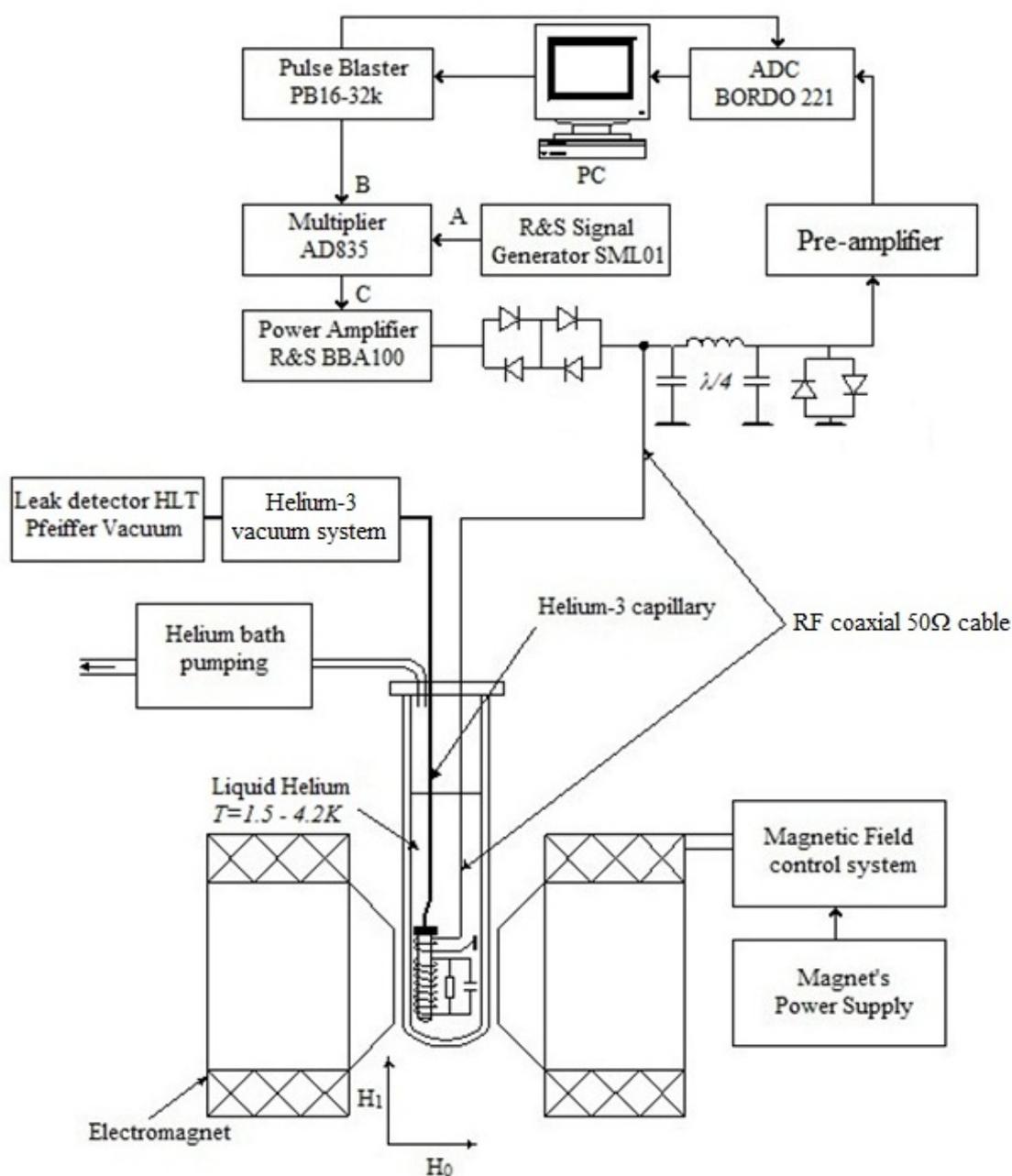
As it is well known, there are two types of the NMR spectrometers for condensed matter: the devices for liquids and ones for solids. In liquids due to molecular motion there is effective averaging of the local magnetic fields produced by neighbor nuclei, that is why NMR lines are narrow. This type of resonance is also known as High Resolution NMR (HR NMR) [6]. As a rule, components of the spectrum have narrow width and their form is not interesting for researchers. In HR NMR the spin-spin relaxation time  $T_2$  is comparable with the spin-lattice relaxation time  $T_1$  and is about seconds. It is much longer than the dead time of the standard NMR spectrometer ( $\sim 10 \mu\text{s}$ ). In solids nuclear moments are localized in the crystal structure. For nuclei with spin  $I = 1/2$  the line width of the NMR spectrum is due to dipole-dipole interaction with neighbor electron and nuclear spins. The NMR line width can vary from few Gauss (local field produced by nearest nucleus) to thousands of Gauss ( $^{165}\text{Ho}$  NMR,  $I = 7/2$ , in  $\text{Rb}_2\text{NaHoF}_6$  [7]). In Wide Line NMR (WL NMR) the spin-spin relaxation time  $T_2$  is shorter than the spin-lattice relaxation time  $T_1$  and can be even shorter than the dead time of the Pulse NMR spectrometer.

$^3\text{He}$  monatomic molecules at low temperatures in porous media can be found in three different aggregate states: solid (adsorbed layer), liquid and gaseous. The structure of the porous samples leads to the transformation of the diffusion motion spectrum and, as a result,  $^3\text{He}$  NMR takes the intermediate position between HR NMR and WL NMR. So it requires the specific equipment. Firstly, the dead time should be the same order of magnitude as in the standard HR NMR spectrometer. Secondly, the homogeneity of the magnetic field should be higher than in the standard WL NMR spectrometer. Thirdly, the magnetic field stability should be quite good. Such investigations require high time resolution of Analog-to-Digital Converter (ADC) because the  $^3\text{He}$  spin echo signal in porous media can be quite sharp (about 5  $\mu\text{s}$ ) in time dimension. The implementation of digital technique gives the following advantages: a simple realization of various pulse sequences, phase inversion, absence of additional noise in digital schemes using of digital filters with sharp characteristics, flexible realization of the spectrometer operation schemes.

In recent years several implementations of digital techniques for NMR spectrometers have been developed [8-10]. The major differences of the described NMR spectrometer are: LabVIEW platform usage, software realization of quadrature detector and filters. The usage of LabVIEW platform makes the modifications of spectrometer's abilities easy and flexible, including pulse sequences and signal processing algorithms. In particular, the software realization of quadrature detector avoids problems with synchronization [9].

## 2. Spectrometer block diagram

It was suggested to use the BORDO-221 ADC (Auris Inc) with time resolution down to 10 ns. For synchronization of the spectrometer digital part and for the pulse sequences generation the Pulse Blaster PB16-32k, SpinCore Technologies Inc., was used. The spectrometer digital part is controlled by LabVIEW software.



**Figure 1.** Block diagram of the pulse NMR spectrometer.

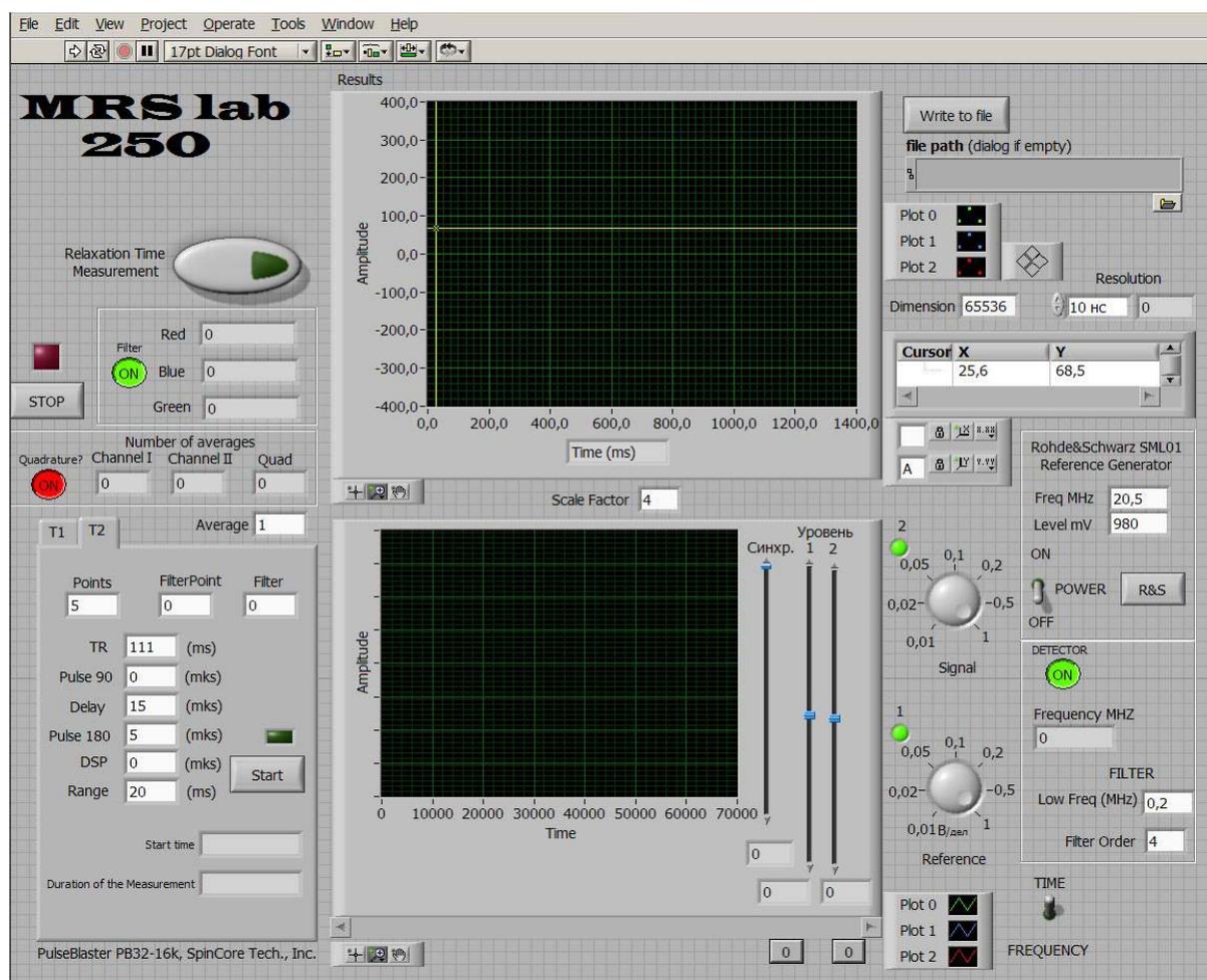
The block diagram of the spectrometer is shown in fig. 1. Reference radiofrequency (RF) signal from the Rohde&Schwarz SML01 generator is applied to the input A of the multiplier AD835. Programmed on LabVIEW, the Pulse Blaster is connected to the input B. As a result, at the output C of the multiplier, we obtain the RF pulses with the frequency equal to the Rohde&Schwarz SML01 generator frequency. After the Rohde&Schwarz BBA100-C125 amplifier, the video pulses are applied to the NMR coil and excite a spin system. The response of a spin system after pre-amplifier proceeds to ADC.

The vacuum system for  $^3\text{He}$  research in contact with porous samples was used. To control the volume of  $^3\text{He}$  which is condensed in the sample the calibration volume ( $8\text{ cm}^3$ ) was designed. The amount of  $^3\text{He}$  is controlled by pressure gauge Piza 111. The helium leak detector Pfeiffer Vacuum Inc has been used for system leakage testing. The temperature of the sample  $1.5 - 4.2\text{ K}$  is achieved by  $^4\text{He}$  bath pumping and measured by calibrated Allen-Bradley carbon resistor.

### 3. Analog-to-digital converter and pulse generator

The digital part of the NMR spectrometer consists of the pulse generator (Pulse Blaster PB16-32k, SpinCore Technologies Inc.) and the analog-to-digital converter (ADC BORDO 221, Auris Inc.).

PB and ADC, made as a PCI board, are connected to PCI bus. The LabVIEW program was written for controlling them (fig. 2). The abilities of this program are: the generation of the pulse sequences and data acquisition.



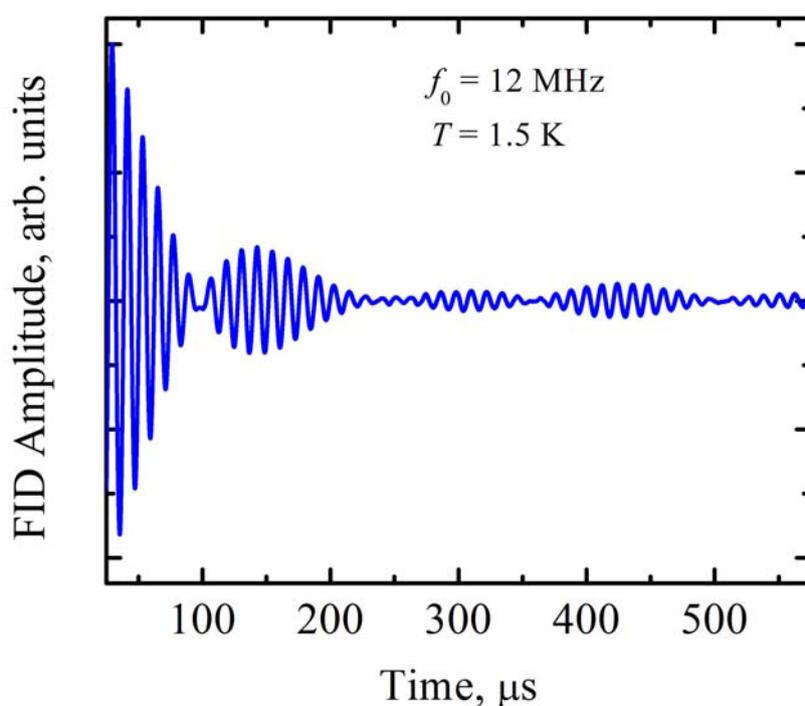
**Figure 2.** Interface of pulse NMR spectrometer.

Main features of PB and ADC are shown in the table 1.

For testing of the program the carbonate geological sample was used. The method of sample preparation is described in [1]. The free induction decay of  $^3\text{He}$  is presented in fig. 3.

**Table 1.** Main features of PB and ADC.

Parameter	BORDO 221	Parameter	PB16-32k
Bit depth	10 bit	Number of channels	16
Input voltage	0-8 V	Pulse duration	50 ns - 2 year
Band	0-150 MHz	The time between pulses	10 ns

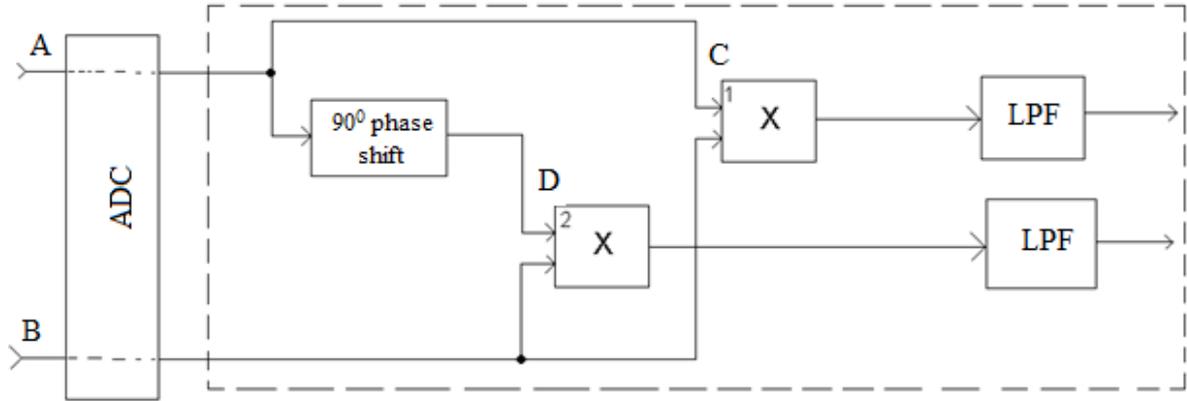


**Figure 3.** FID of liquid  $^3\text{He}$  in geological sample (4 scans).

#### 4. Digital quadrature detector

A signal, the spectrum of which does not contain frequencies above  $f_e$  (Hz), can be recovered if the reference values of the signal, taken at regular intervals of time  $\Delta t = 1/2f_e$  (s), is known. In the spectrometer the ADC with sampling period  $\Delta t = 10$  (ns) is used, so we can correctly digitize the signal the spectrum of which does not contain frequencies above 50 MHz. The block diagram of the digital quadrature detector is shown in fig. 4.

The signal from the harmonic reference generator Rohde&Schwarz SML01 is applied to the input  $A$  of the double channel ADC. The obtained array is supplied to the  $90^\circ$  phase shift unit and to the multiplier  $C$ . The signal from the pre-amplifier is applied to the input  $B$  of the ADC. This array of data is supplied to the two multipliers,  $C$  and  $D$ . After multipliers data sets arrive to the low-pass filters (LPF) from LabVIEW library.



**Figure 4.** The block diagram of a digital quadrature detector.

Let us describe the operation of the  $90^\circ$  phase shift unit. If we have the signal  $\sin \omega t$  as the digitized array then we need to have array  $C$  with the signal  $\cos \omega t$ . Let us create an additional array  $B$  which is obtained from the array  $A$  by shifting its elements into two index up, i.e.  $B[i] = A[i + 2]$ . Obviously, the array  $B$  can be written as  $\sin(\omega t + \varphi)$ , where  $\varphi = 2\omega\Delta t$  is the phase shift,  $\Delta t$  is the sampling period. Using the following trigonometric equations:

$$\sin(\omega t + \varphi) = \sin \omega t \cos \varphi + \cos \omega t \sin \varphi, \quad (1)$$

$$\cos \omega t = -\frac{\sin \omega t}{\tan \varphi} + \frac{\sin(\omega t + \varphi)}{\sin \varphi} \quad (2)$$

we can conclude that any element of the array  $C$  can be obtained as the sum of the corresponding element of the array  $A$  and the corresponding element of the array  $B$  with the following coefficients:

$$\alpha = -\frac{1}{\tan \varphi}, \quad (3)$$

$$\beta = \frac{1}{\sin \varphi}, \quad (4)$$

i.e.  $C[i] = \alpha A[i] + \beta B[i]$ .

As a result, the output of the quadrature detector has the following signals - channel "A" and "B" (the results of the two synchronous detectors, the reference signals are out of phase), and the channel "C" which is called quadrature (or  $C^2 = A^2 + B^2$ ).

## 5. Conclusion

We have developed and described here the experimental setup of home-build pulse NMR spectrometer. The digital spectrometer gives the following advantages: absence of additional noise in a quadrature detection and low frequency amplifiers, using of digital filters with sharp characteristics, flexible realization of the spectrometer operation scheme.

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