Neutralization of fluctuations in resonance conditions during registration of NMR spectra in the Earth's magnetic field

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NMR-spectra in weak fields are not split due to chemical shifts and they would seem uninformative for analysis of substances. Nevertheless, NMR in the Earth magnetic field allows ones to register the record resolution of spectra which are split due to heteronuclear interactions (J-coupling). Thus, the NMR in Earth magnetic field provides an opportunity to measure J-coupling constant with the highest accuracy. The proton spectrum has a symmetrical structure and a distance between lines determines the degree of spin interactions. As examples, several J-coupling NMR-spectra which were obtained with the home-built equipment [1] are represented.There are some actual nuclear isotopes which interact with protons in liquid compounds (it is impossible to detect NMR in the Earth magnetic field in solids). The main such isotopes are presented in Table 1.

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Nucleus	Abundance,%	Spin
^{19}F	100	1/2
^{29}Si	4.7	1/2
^{31}P	100	1/2
^{13} C	1.1	1/2

Table 1. *The actual isotopes which interact with protons and form the indirect spin-spin J-coupling observed on in the Earth field NMR-spectra.*

J-coupling spectra which recieved with small averaging
Fluorine-containing liquids

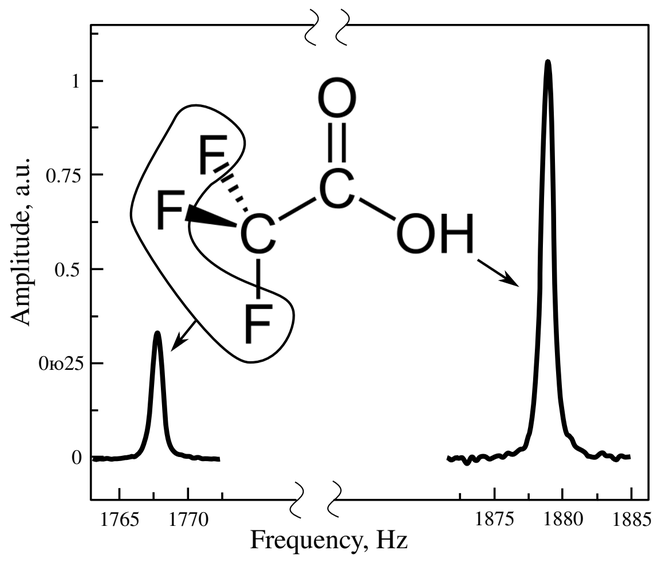

Fig. 2. *The spectrum of 2,2,2-Trifluoroacetic acid. The sensor of the NMR-spectrometer is tuned to the proton frequency.*

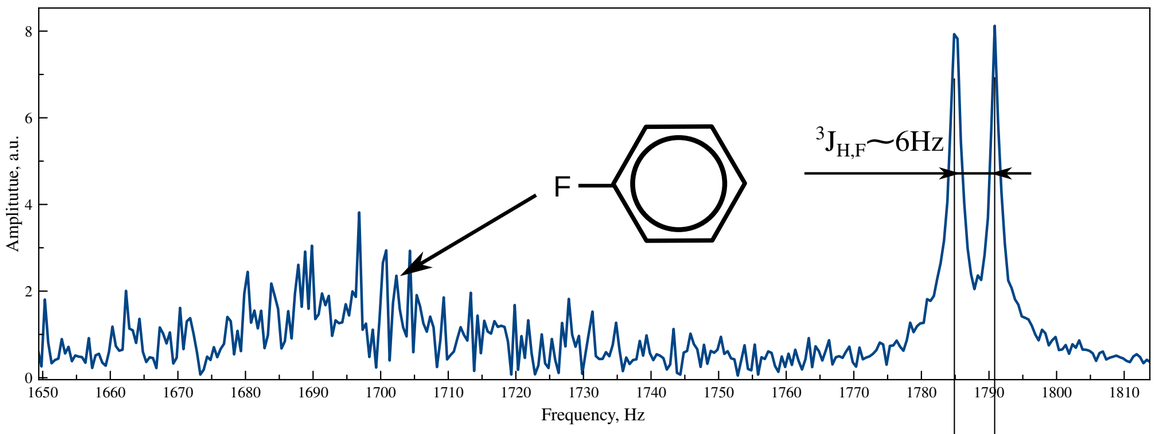

Fig. 3. *The spectrum of fluorobenzene. The sensor of the NMR-spectrometer is tuned to the fluorine frequency. Averaging is 200 shots. The relation of signal-to-noce is not enougth.*

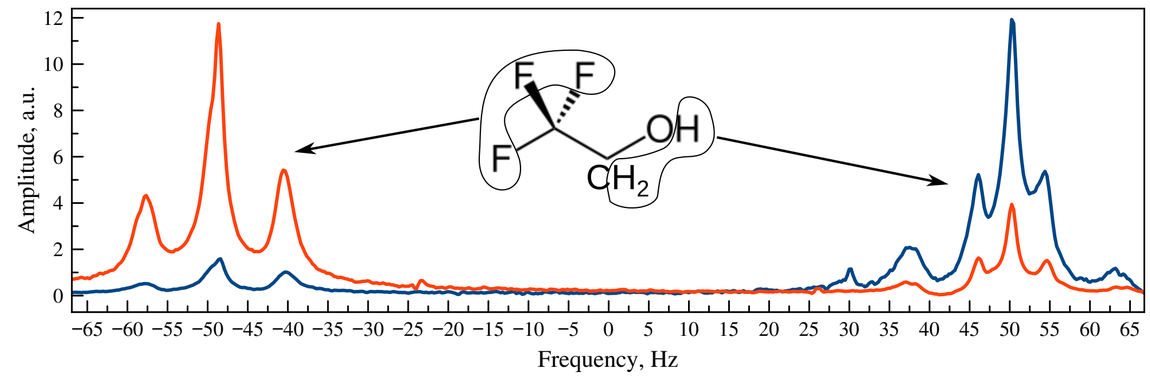

Fig. 4. *The spectra of 2,2,2-trifluoroethanol. The sensor of the NMR-spectrometer is tuned to the fluorine frequency (red) or to the proton frequency (blue).*

Phosphates

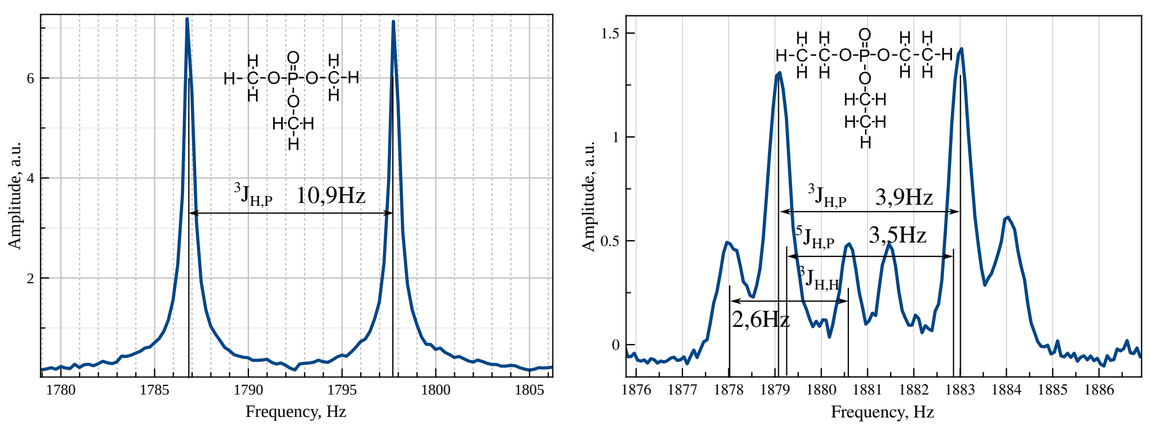

Fig. 5. *The spectra of Trimethilphosphate (left) and Triethilphosphate (right).*

Silicates

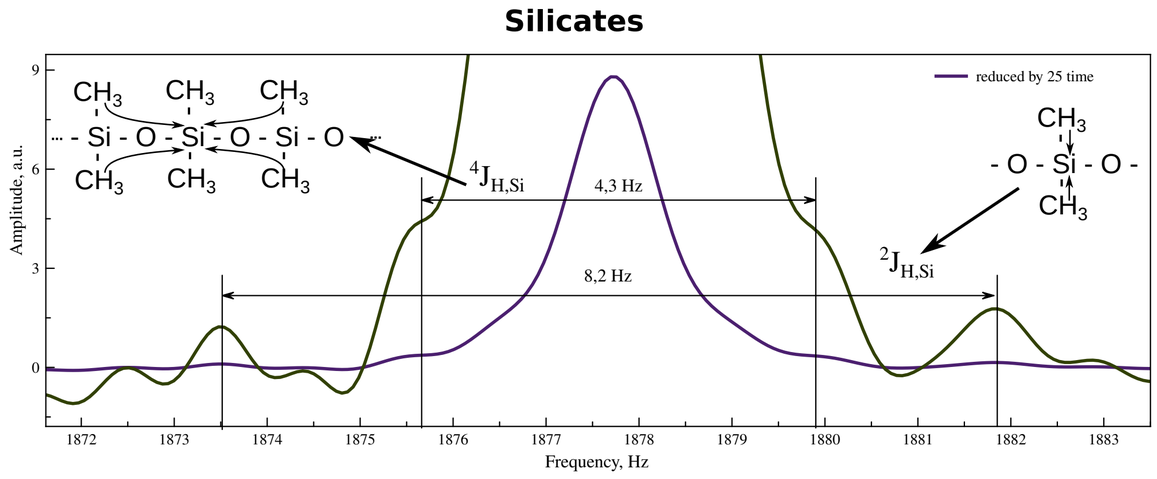

Fig. 6. *The spectra of Polimethilsiloxan, 36 time averaging*

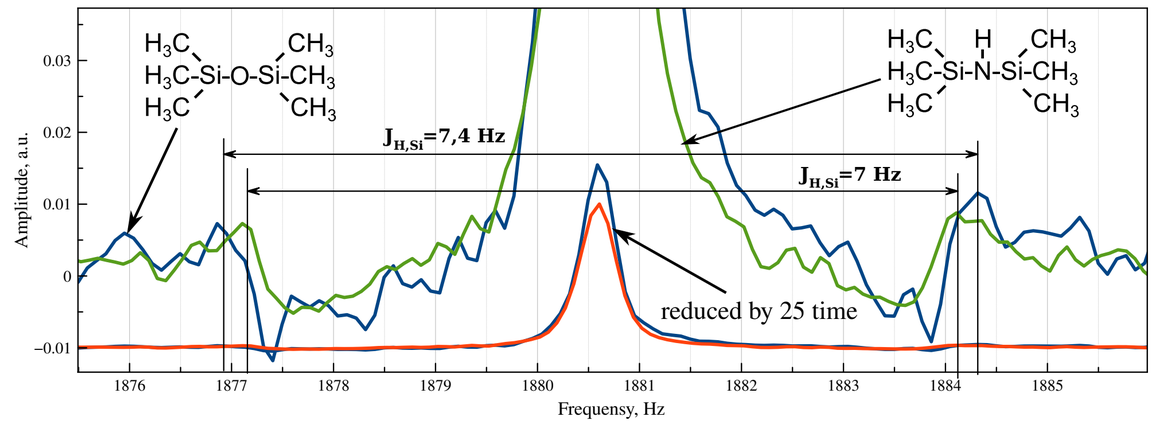

Fig. 7. *The spectra of Hexamethilsiloxan (red) and Hexamethilsilozan (blue),*

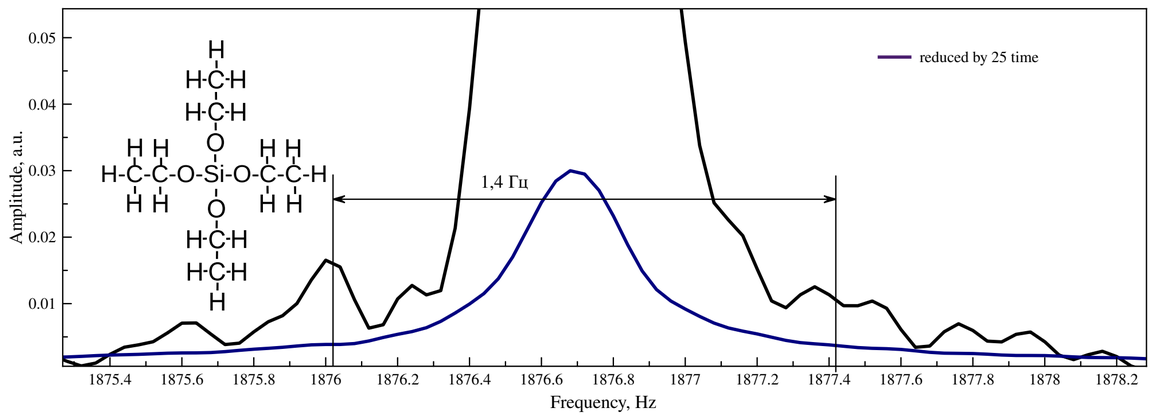

Fig. 8. *The spectra of Tetraetoxysilan (red), 4 time everaging.*

Problems in the case of small natural abundance of interacting nuclei

It is very important to analyze organic liquids but the isotope 13C has natural abundance of 1.1 % and satellites in proton spectra are very weak. In this case it would be effective the signal accumulation. Unfortunately, the Earth magnetic field is not stable enough, especially in laboratory conditions. To neutralize the fluctuations of the Earth magnetic field we developed and patented the method of the stabilization of resonance conditions [2].

The principle is based on two-sensors scheme [3]. The first sensor contains an investigated sample. A sample in the second sensor is a proton liquid which possesses a single intensive line in the NMR-spectrum. The signal from the second sensor carries an The main feature is detection method of test probe signal (1). As signal (9) for quadrature detection (10), the synthesized sin with frequency (8) from second sensor probe (2). The device is patented (RU175 974U1).

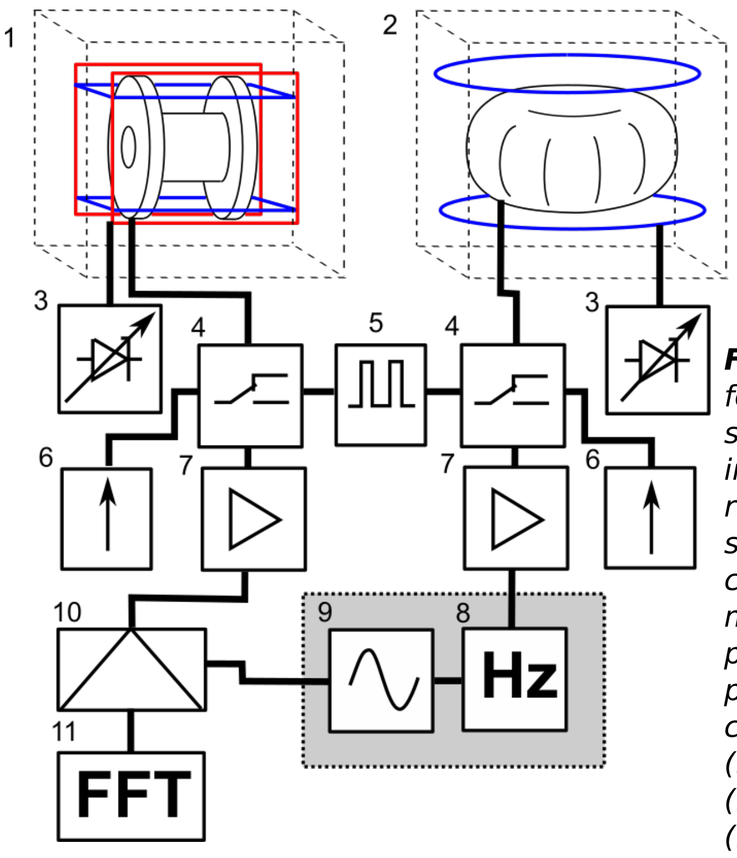

Fig. 1. *The device scheme for NMR-frequency stabilization: sensor for an investigated sample (1), reference sensor (2), shimm systems (3), commutation blocks (4), microcontroller (5), pre-polarization power siply (6), preamplifier (7), frequency counter (8), sine generator (9), quadrature detector (10), spectrum visualizer (11).*

References

1. P. A. Kupriyanov, K. A. Dmitriev, A. V. Chizhik. On some improvements of nuclear magnetic resonance in the earth’s magnetic field registration. Vestnik SPSU, T. 3. № 1. pp. 59-69. 2016.
2. V. I. Chizhik, P. A. Kupriyanov. Equipment for stabilizing the frequency of nuclear magnetic resonance in the Earth's magnetic field. Patent of Russian Federation № 175974. Bull. № 36. 2017.
3. A. Mohoric, G. Planinsic, M. Kos, A. Duh, J. Stepisnik. Magnetic Resonance Imaging System Based on Earth magnetic field". Instrumentation Science and Technology, Vol. 32, No. 6, pp. 655-667, 2004.