

# МЕТОДЫ И ПРИБОРЫ КОНТРОЛЯ И ДИАГНОСТИКИ МАТЕРИАЛОВ, ИЗДЕЛИЙ, ВЕЩЕСТВ И ПРИРОДНОЙ СРЕДЫ



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## HIGH-Q LITZ WIRE NQR SENSOR FOR MEDICAL APPLICATIONS

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**Abstract:** *RELEVANCE.* The design and development of radio frequency (RF) coil sensors is an important engineering and, at the same time, fundamental task for those radio spectroscopic instruments that require an increase in sensitivity, measured as a signal-to-noise ratio (SNR). Radio spectroscopy of nuclear quadrupole resonance (NQR), especially in nitrogen compounds, in which the resonant frequency is very low and ranges from a few megahertz or lower to hundreds of kilohertz, requires the use of special solutions to increase the sensitivity. *PURPOSE.* Theoretical substantiation and search for a technical solution that allows achieving high sensitivity on standard equipment through the use of a high-quality sensor. *METHODS.* Methods for optimizing the design of sensors for NQR/NMR spectrometers are considered. The design of the sensor for the NQR spectrometer, which contains an inductance coil wound with a Litz wire, has been calculated and designed. *RESULTS.* A high-quality coil for the spectrometer sensor was made, which gives an increase in the quality factor by about 1.5 times. The use of a spectrometer with this coil made it possible to confidently record weak noisy signals of paracetamol at a low duty cycle. The sensitivity of the sensor made it possible to distinguish preparations from different manufacturers by their spectral characteristics. *CONCLUSIONS.* A solenoid sensor has been developed, modeled and manufactured, which has a high quality factor and allows recording quadrupole resonance signals of drugs (paracetamol) by a non-destructive method directly through the package. The possibility of using such a sensor for quality control of medicines, detection of falsified and counterfeit medicines is shown.

**Keywords:** NQR 14N; quadrupole resonance sensor; litz wire RF coil; authentication of medicines.

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## ВЫСОКОДОБРОТНЫЙ ЯКР СЕНСОР ДЛЯ МЕДИЦИНСКИХ ПРИЛОЖЕНИЙ

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**Abstract:** *АКТУАЛЬНОСТЬ.* Проектирование и разработка радиочастотных (РЧ) датчиков катушек является важной инженерно-технической и, одновременно,

фундаментальной задачей для тех радиоспектроскопических приборов, в которых требуется увеличение чувствительности, измеряемое как отношение сигнал/шум (SNR). Радиоспектроскопия ядерного квадрупольного резонанса (ЯКР), особенно в соединениях азота, в которых резонансная частота очень низка и составляет от единицы мегагерц или ниже - до сотен килогерц, требует применения специальных решений для увеличения чувствительности. **ЦЕЛЬ.** Теоретическое обоснование и поиск технического решения, которое позволяет добиться высокой чувствительности на стандартном оборудовании за счет применения высокодобротного датчика. **МЕТОДЫ.** Рассмотрены методы оптимизации конструкции датчиков для ЯКР/ЯМР спектрометров. Рассчитана, спроектирована конструкция сенсора для спектрометра ЯКР, содержащая катушку индуктивности, намотанную проводом типа литцендрат. **РЕЗУЛЬТАТЫ.** Изготовлена высокодобротная катушка для датчика спектрометра, дающая увеличение добротности примерно в 1,5 раза. Использование спектрометра с данной катушкой позволило уверенно регистрировать, слабые зашумленные сигналы парацетамола при низком коэффициенте заполнения. Чувствительность датчика позволило различить по спектральным характеристикам препараты разных производителей. **ВЫВОДЫ.** Разработан, смоделирован и изготовлен соленоидный датчик, обладающий высокой добротностью и позволяющий регистрировать сигналы квадрупольного резонанса лекарственных средств (парацетамол) неразрушающим методом непосредственно через упаковку. Показана возможность использования такого датчика для контроля качества лекарственных средств, выявления фальсифицированных и поддельных лекарств.

**Ключевые слова:** ЯКР  $^{14}\text{N}$ , датчик квадрупольного резонанса, литцендрат, рч, аутентификация лекарственных препаратов.

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### **Introduction**

The problem of pharmaceutical drugs quality is relevant all over the world. According to the World Health Organization in developing countries, one pill in 10 is adulterated. It may contain no active ingredient at all or contain less than necessary. According to official statistics, the share of fake (counterfeit, adulteration) drugs in the Russian Federation is 2-4%, according to unofficial data, the number of counterfeit drugs is much higher and reaches 10% for some positions. In Europe and North America, the percentage of low-quality drugs is lower than in Russia; in Asia and Africa, the proportion of counterfeit drugs reaches 50% for some popular types of drugs [1-5]. Another factor influencing the spread of counterfeit products is the increase in the online trade in medicines. Exercising control organizations use physical and chemical methods to test drugs. An obligatory set, as a rule, are methods of chemical analysis, chromatographic analysis, methods of IR spectroscopy, mass spectroscopy. Most of these research methods allow a qualitative analysis of the medicinal product for compliance with regulatory documentation. However, the existing analytical equipment has a number of disadvantages. For example, generally preliminary preparation of a test sample is required. The equipment is bulky and requires qualified personnel. The modern analytical method based on Raman scattering spectroscopy is devoid of the main disadvantages as non-destructive testing is provided, i.e. analysis can be carried out without damaging the packaging, and the scanners are portable and easy to operate [8-11]. At the same time, their use is limited by the requirements of the optical transparency of the medium, which means that it is impossible to study drugs in opaque packages and most tablets or powders in capsules. Therefore, the technique of nuclear quadrupole resonance (NQR) has outlook as a method for determining the quality of drugs, as it allows the study of chemical compounds in the solid phase directly in the package (in blisters, plastic and cardboard boxes and tubes) [10-12].

### **Literature Review**

Nuclear quadrupole resonance (NQR) is a radiospectroscopy method based on the resonant absorption of electromagnetic energy in crystals, due to transitions between energy levels resulting from the interaction of the electric quadrupole moment of the nucleus with the electric field gradient (EFG) at the location of the nucleus. NQR is observed in solid mono- and polycrystalline compounds (also in frozen liquids) containing quadrupole nuclei, and the energy levels arising

from this interaction absorb and emit in the range from 0.1 to 10 MHz. Typical nuclei on which NQR is observed are  $^{35}\text{Cl}$ ,  $^{37}\text{Cl}$ ,  $^{79}\text{Br}$ ,  $^{81}\text{Br}$ ,  $^{127}\text{I}$ ,  $^{121}\text{Sb}$ ,  $^{123}\text{Sb}$ ,  $^{75}\text{As}$ ,  $^{63}\text{Cu}$ ,  $^{65}\text{Cu}$ ,  $^{14}\text{N}$ ,  $^{10}\text{B}$ ,  $^{11}\text{B}$  etc. Since the NQR resonance frequency is determined by the interaction between the quadrupole moment of the nucleus and the EFG tensor around the given nucleus, it is unique for a given chemical compound, the so-called passport. This circumstance made it possible to develop entire areas of research related to the search and detection of explosives and narcotic substances [13-15] since all explosives and most drugs contain quadrupole nitrogen  $^{14}\text{N}$ . In the early 2000-s, a group of scientists from King's College proposed to use the NQR method to determine the authenticity of drugs. In addition, with the help of NQR it is possible to study the polymorphism of medicinal compounds [16].

The physics and technology of NQR is in many ways similar to nuclear magnetic resonance (NMR), with one very significant difference: pure NQR does not require a constant magnetic field. Therefore, NQR equipment is more compact and cheaper to manufacture. The main and serious problem that limits the use of NQR for such studies is the low sensitivity or the associated low signal-to-noise ratio. The amplitude of the NMR/NQR signals is related to the resonant frequency, and in NQR, for example, nitrogen frequencies lie in the range of 0.6–6 MHz, as a result, the transient signals induced in the sensor coil have amplitude of several  $\mu\text{V}$ , which is commensurate with the noise of the device itself. There are a large number of ways to increase the sensitivity, such as the use of multiple signal accumulation, the use of special multi-pulse sequences, the use, together with NQR, of sensors based on other methods, such as giant resistive resistance or SQUID, a combination of NQR and NMR, two- and three-frequency techniques, improvement of the sensor design [17-20].

#### **Materials and Methods NQR Probe design**

The NQR sensor of the spectrometer must simultaneously perform two main functions. On the one hand, it must create a high radio frequency field to excite all quadrupole nuclei, and on the other hand, it must be sensitive to register very small (a few microvolts) NQR signals. Traditionally, RLC oscillatory circuits tuned to the resonant frequency are used for these purposes. The test sample is placed inside, as a rule, a solenoidal inductor. For a number of specific tasks, such as mine detection, flat coils are used, with a sample containing quadrupole nuclei located outside the coil. The main parameters of the oscillatory circuit are its active and reactive resistances, impedance and also the quality factor of the circuit. A detailed calculation of the circuit elements for parallel and series oscillatory circuits is given in [21, 22]. For a series oscillatory circuit shown in Fig. 1, the expressions are as follows:

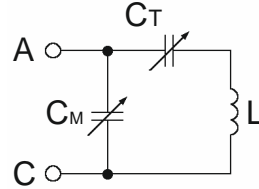


Fig.1 The series oscillatory circuit

The impedance between points A and C is:

$$Z_{AC} = \frac{\left( R + j\omega L + \frac{1}{j\omega C_T} \right) \cdot j\omega C_M}{\left( R + j\omega L + \frac{1}{j\omega C_T} \right) + j\omega C_M} \quad (1)$$

where  $R$  is the resistance of the coil at the resonant frequency  $\omega = 2\pi f$ , where  $f$  is the frequency;  $C_M$  and  $C_T$  capacitances of the matching and resonant capacitor, respectively,  $L$  is the inductance of the coil. Simplifying the expression and removing negligible terms and separating the real and imaginary parts separately, we get:

$$Z_{AC} = \frac{R}{\omega^2 C_M^2 R^2} - j \frac{(\omega^2 L C_T - 1)(\omega^2 L C_T C_M - C_M - C_T)}{\omega^2 C_M^2 C_T^2 R^2} \quad (2)$$

In order to match with the output stage of the power amplifier, it is necessary to provide a resistance of the real part of expression (2) equal to  $50 \Omega$ . To do this, it is necessary that the circuit be tuned to the resonant frequency by adjusting the values of  $C_M$  and  $C_T$  in such a way that the relation is fulfilled:

$$\frac{R}{\omega^2 C_M^2 R^2} = 50 \Omega \quad (3)$$

The value should be:

$$C_M = \frac{1}{\omega \sqrt{50R}} \quad (4)$$

For maximum sensitivity, it is necessary to achieve several indicators simultaneously. The oscillatory circuit must be tuned to resonance and, at the same time, be matched with the output stage of the spectrometer; it must have a low ohmic resistance and a high quality factor, must withstand the impact of powerful radio frequency pulses (up to several kW), retaining its characteristics, while being easily tuned to other frequencies. Therefore, the design of the sensor, its material, winding method, shape and cross-section of conductors are carefully calculated and selected to achieve optimal parameters.

The sensor scheme is shown in Fig.2. Here  $C_M$  is a capacitor for matching the output impedance of the power amplifier with the input impedance of the sensor - a Jennings 5-500 pF variable vacuum capacitor,  $C_T$  is a similar capacitor for tuning the circuit into resonance. Additionally, high-quality ceramic capacitors from ATC were used. The cross-diodes are designed to protect the preamplifier from high RF pulses.

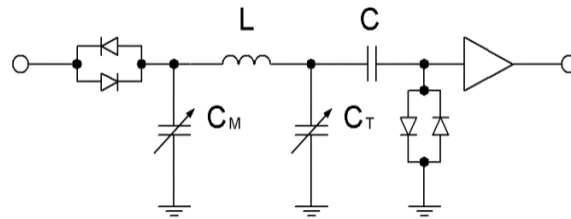


Fig.2. The NQR sensor scheme

The sensor coil is one of the most critical elements. Requirements are also imposed on it for low active resistance on the one hand and a sufficiently high inductance on the other. The dimensions of the coil must allow for completely non-destructive measurements for various types of drugs. Their packaging can be in the form of a rectangular cardboard box in which blisters with compressed tablets are packed, or in the form of a plastic tube containing gelatin capsules.

The quality factor of a series circuit consisting of a coil and a capacitor depends on the quality factor of both of them. But taking into account the fact that the quality factor of the capacitor is much greater than the quality factor of the inductor coil, it is the quality factor of the coil that turns out to be decisive for the oscillatory circuit. The quality of the capacitor has a strong influence on the quality factor of the circuit as a whole, but since the further selection of the inductor used the same set of capacitors for both cases, the further calculation concerns only the coil.

The sensor coil was wound with  $N = 65$  turns of copper wire with a diameter of 0.6 mm on a frame made of PVC pipe with a diameter of  $D = 50$  mm. All available samples of paracetamol were placed in such a coil, both in blister packs and in plastic tubes. There are several formulas that allow you to calculate the inductance from a solenoidal coil, taking into account its parameters. The range of inductance values was from  $140 \mu\text{H}$  to  $240 \mu\text{H}$ . The most accurate value of  $169 \mu\text{H}$  also takes into account not only the geometric dimensions of the frame, but also the ratio of the coil winding length to its diameter as follows:

$$L = \frac{\mu_0}{4\pi} N^2 D \Phi \quad (5)$$

In this expression

$$\Phi = \frac{\pi^2}{\alpha} \left( 1 - \frac{4}{3\pi\alpha} + \frac{1}{8\alpha^2} \right) \quad (6)$$

Where  $\alpha = l / D = 0,84$  is the ratio of the length of the winding to the diameter of the frame.

The quality factor of the coil is calculated by the formula

$$Q = \frac{2\pi fL}{R_{AC}} \quad (7)$$

In this expression, the denominator is the loss impedance for alternating current at a given frequency. Despite a large number of studies on this topic, there is still no single algorithm for calculating losses with alternating current from the basic works of Butterworth 1926 [23] and R.G.Medhurst [24].

At the same time, the exact calculation of losses is not an easy task. The most profound and detailed consideration of this issue is given by David W. Knight in his works [25, 2].

Losses at radio frequencies are caused by three factors: dependence on the material of the conductor, the influence of the skin effect, the influence of the proximity effect. The first factor can be ignored, since the copper material used has a rather low resistivity value and, in comparison with the skin effect, the influence of this effect is much less. Of course, you can use metals with lower resistivity, such as silver, but then this will lead to an increase in cost, with a very small gain. The phenomenon of the skin effect is manifested in the fact that at radio frequencies the currents in the conductor flow in a thin near-surface layer, increasing the resistance to alternating current. Despite the fact that the frequency is "boundary" to take into account the influence of the skin effect, it was decided to take into account the skin effect.

The proximity effect is due to the interaction of the radio frequency field of adjacent turns of the coil, eddy currents arise in them, which, in combination with skin effect currents, add losses. The value is determined by the ratio between the winding pitch and the diameter of the wire, as well as between the length and diameter of the coil. According to the table from [27], for a coil with the parameters used in the article, this coefficient is equal to  $\Psi=5.8$ .

Thus, total resistance is

$$R_{AC} = R_{DC} \cdot \Xi \cdot \Psi \quad (8)$$

The thickness of the layer  $\delta$  in which the entire current is enclosed more precisely, the depth at which the current density decreases by  $e=2.71$  times is determined by expression (9):

$$\delta = \sqrt{\frac{\rho}{\pi f \mu}} \quad (9)$$

Where  $\rho$  is metal resistivity [Om.m],  $f$  is frequency [Hz],  $\mu = \mu_0 \mu_r$ ,  $\mu_0 = 4\pi \times 10^{-7} \text{H/m}$ ,  $\mu_r$  - relative permeability.

The influence of the skin effect is introduced as a coefficient  $\Xi$ , which shows how many times the resistance to alternating current at a given frequency is greater than the resistance to direct current.

$$R_{AC} = R_{DC} \cdot \Xi$$

Taking into account the influence of losses on proximity, the total resistance will be considered as (8)

$$R_{AC} = R_{DC} \cdot \Xi \cdot \Psi$$

The classical calculation using the area of the ring in which the current passes, limited by

the radius of the wire  $r$  and the penetration depth  $\delta$  gives:

$$A_{eff} = \pi(2r - \delta^2) \quad (10)$$

Then, taking into account the classical expression for the resistance to direct current  $R_{DC} = \rho l / A$ , where  $A = \pi r^2$

We obtain

$$\Xi = r^2 / (2r - \delta^2) \quad (11)$$

Having carried out calculations taking into account losses due to the skin effect and the proximity effect, we obtain an increase in losses by about 8 times. The inductance of the solenoid coil is calculated by formula (5). Having carried out calculations for our coil, we obtained the value of the coefficient  $\Xi$  equal to 3.9. The value of the coefficient  $\Psi$  turned out to be 5.8. Thus, the quality factor of the coil, taking into account losses, turned out to be 191. The adequacy of the calculations was also confirmed by calculations in the online calculators of the Coil32.com portal. In addition, having carried out measurements on the device “Q-meter E4-4”, the values  $L = 156 \mu\text{H}$ ,  $Q = 195$  were obtained, which also confirms the adequacy of the calculations. This is a fairly good value for RF coils used for NQR spectroscopy. However, for our purposes, where the fill factor of the coil will be very small, due to the fact that the volume of tablets in the blister will be uneven and occupy a very small part of the volume of the coil, this value turned out to be insufficient to obtain transient signals.

As an optimization of the coil parameters, it was decided to reduce the loss resistance, get rid of the skin effect and reduce the influence of the proximity effect by using a Litz wire. Such solution should lead to an increase in the quality factor of the coil and, consequently, the oscillatory circuit, the sensor of the spectrometer as a whole.

### Result

As a result, the calculation of a litz wire coil is given below. To reduce the losses associated with the skin effect in practice the replacement of a single-core wire with a litz wire is used 3, which is a bundle of a large number of thin conductors, where each has lacquer insulation in a common silk or lavesan insulation. Thus, there is a gain due to the larger surface area of a large number of conductors.

The coil was wound with 105/48 AWG litz wire. Loss resistance parameters were calculated using New England Wire Technology <https://www.newenglandwire.com/traditional-litz-wire-theory>.

For this wire containing 105 strands with a diameter of 0.03 mm each, the total diameter was 0.43 mm, taking into account the outer silk insulation. According to the calculations using the above method, the loss resistance to the skin effect and the proximity effect is:

$$\Xi = 1,0076 \quad \Psi = 3,9$$

$$\text{While } R_{DC} = 0,237 \text{ OM/M}$$

$$\text{Including losses } R_{AC} = 0,93 \frac{OM}{M}$$

The inductance of the coil wound with a litz wire appeared 172  $\mu\text{H}$  and the final quality factor calculated by expression (5) turned out to be 291. Here for the total resistance value, a wire length of 10.21 m is taken into account.

This value turned out to be about 50% higher than the quality factor of a coil with a conventional wire. This difference was sufficient to obtain an NQR signal from paracetamol [28, 29]. If we evaluate the effect of the quality factor on the measurement sensitivity, then according to [14] the voltage amplitude of the induced signal is approximately equal to:

$$V = \omega IM \sqrt{QZ_{AC} / \omega L},$$

where  $\omega = 2\pi f$  is frequency,  $I$  is the magnitude of the current induced by the sample,  $M$  is the magnetization induced in the coils by currents  $I$ .

For a coil wound with a litz wire, the amplitude value turned out to be 30% larger compared to a conventional wire.

Comparison with conventional coils made according to the technology published in [11,12] and our Litz wire coil shows an increase in the quality factor by about 1.5-1.7 times, which leads to an increase in the signal-to-noise ratio and, consequently, to a reduction in detection time by about 1.5 times. This is important as a large number of measurements are required for drug authentication purposes.

Further research involves expanding the range of drugs containing other quadrupole nuclei and creating a prototype of a compact drug authenticity scanner.

### Experiment

Measurements were performed on a spectrometer Apollo Tecmag. NQR experiments were carried out on Apollo Tecmag NQR/NMR console (0.1-100 MHz) with two-channel transmitter and one-channel receiver modules. Two Tomco BT-00500-Beta power amplifiers with output power of up to 500 W have been used. The detector unit includes transcoupler, a quarter wave lines  $\pi$ - filter 1.5-3.6 MHz bandwidth, a low-noise single-channel preamplifier Miteq and signal sensor. The coil described above was used as a sensor, complete with Jennings 5-500 pF vacuum capacitors and fine tuning with ATC capacitors. For measurements, samples of the medicinal compound "Paracetamol" of various brands from different countries - Turkey, Italy, Czech Republic, Russia, India, etc. containing 500 mg and 1000 mg of the active substance were purchased. The measurements were carried out at room temperature using a multi-pulse sequence of double spin-locking, which is a sequence of two series of 90-degree pulses separated by an interval for signal accumulation and recording. The pulse duration and the interval between pulses varied over a wide range and were selected according to the echo signal maximum. At the NQR transition frequency of 2.564 MHz, the most optimal pulse durations were 30  $\mu$ sec, the interval between pulses was 2000  $\mu$ sec. The number of pulses in the 1024 series, the signal recording time is 2.56 ms (the number of gates is 512 with an interval of 5  $\mu$ s). The experiment using such a sensor makes it possible to obtain spin echo signals. The width of the lines for different manufacturers is different, which allows you to confidently distinguish paracetamol from different manufacturers (see Table1). The reasons for this difference can be different - the presence of moisture, impurities, the difference in the composition of the auxiliary ingredients, the density of the tablet, etc. Discussion of these reasons is beyond the scope of this article.

Table1

Spin echo linewidth of paracetamol from different manufacturers

Medicine name	The linewidth, Hz
Parol_A_(Tur)	865
Parol_(Tur)	793
Panadol_(Che)	1305
Panadol_(Rus)	1080
Paracetamolo_(Ita)	350
Paracetamol_(Rus)	1245
Paralen_(Che)	980
Tachspirina1000_(Ita)	827
Tachspirina500_(Ita)	970
Merimol650_(Ind)	940

### Conclusion

Measurement of quadrupole resonance signals of very small amplitude, in most cases covered by noise of various nature, is a rather tricky engineering problem. Most often, this problem is solved in a complex way - equipment, multi-pulse sequences, mathematical signal processing. The choice of the optimal sensor design for the spectrometer is one of the main ways to solve this problem. Therefore, solutions that give even a small, few percent gain, can help to pull the signal out of the noise, especially when measuring the NQR characteristics of low nitrogen compounds (0.5 - 6 MHz). The quality factor of coil makes it possible to increase the sensitivity, and therefore the proposed solution of replacing the wire with a litz wire turned out to be effective, despite the fact that the measured frequencies approached the limit of applicability of the influence of this effect.

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