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Registration of spectra in a high-frequency spectrometer of electron paramagnetic resonance using a cylindrical waveguide without a cavity

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The article presents a simple design of a device used in a high-frequency EPR spectrometer operating at 94 and 130 GHz. The device is used to transmit microwave energy over a long distance without noticeable attenuation from a microwave generator to a sample in a helium cryostat. This design allows recording EPR–ODMR spectra in normal and pulsed modes without using a resonator.

Keywords: high-frequency EPR spectrometer, cylindrical waveguide, registration of EPR spectra without a resonator.

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The electron paramagnetic resonance (EPR) phenomenon has been discovered more than 80 years ago [1] and quickly became a powerful method for studying substances. It still retains its significance, since the experimental techniques are being improved constantly, allowing for deeper analysis. One of the main reasons why magnetic resonance research remains relevant in various fields of physics, chemistry, biology, and medicine is that it provides an opportunity to obtain data on the processes occurring in materials at the atomic level. The number of published papers reporting EPR results is increasing steadily. EPR-related techniques, such as optically detected magnetic resonance (ODMR) [2], electron nuclear double resonance (ENDOR) [3], and pulsed detection methods [4], have also been improved over the years.

The instrumentation industry has long produced spectrometers operating in the X (9 GHz) and Q (35 GHz) bands of microwave frequencies. This is explained, on the one hand, by the ease of manufacture of microwave resonators for maintaining a high microwave field strength H_1 on the sample and, on the other hand, by the relative simplicity of fabrication of electromagnets with an induction up to 2 T that are required for operation in the specified microwave ranges. The advent of semiconductor Gunn diodes made it possible to reduce the voltage in a microwave generator to several volts compared to high-voltage klystrons operating at 500–1000 V). However, the introduction of Gunn diodes did not change qualitatively the design of microwave generators.

The idea of using higher microwave frequencies stems from the very nature of the EPR effect, since an increase in frequency leads to an increase in sensitivity and resolution in measurement of g-factors, which is especially important in organic chemistry and biology. The first high-frequency spectrometer operating in the two-millimeter range (D band) has been designed by Soviet scientist Ya.S. Lebedev [5] in 1977. It operated at a frequency of 130 GHz with a single-mode cylindrical resonator TE_{011} [6]. The sensitivity of Lebedev's spectrometer was as high as $4 \cdot 10^7$ spin/G.

A magnet with an induction of at least 5 T (a g-factor of 2 corresponds to a magnetic field of 4.64 T) was needed in order to observe electron paramagnetic resonance within the 130 GHz range. Lebedev used a superconducting solenoid as a magnet. In turn, this superconducting magnet required a special helium cryostat. Until the mid-80s, Lebedev's spectrometer had no comparable counterparts in the world. One major factor limiting the applicability of such high-frequency EPR spectrometers is the need for large quantities of liquid helium.

The design and construction of high-frequency EPR spectrometers is still the domain of enthusiasts. Bruker [7] is the only company to release a commercial EPR spectrometer operating in the X (10 GHz), Q (35 GHz), and W bands (95 GHz). The main disadvantage of this device is that it is very expensive.

The microwave field at the sample ($\mathbf{H}_1$) in modern spectrometers is produced by generators with a frequency stability on the order of 10^{-6} for frequencies upward of 90 GHz and an extremely narrow frequency band (just several hundred kHz [8]). Such generators provide an opportunity to alter the design of the device that transfers microwave energy from the generator to a sample. First, it is no longer necessary to use automatic frequency control. Secondly, a cylindrical single-mode resonator makes the design significantly more complicated in this case, since the fabrication of such a resonator involves complex mechanical processing. It is also difficult to match the cavity to the waveguide and adjust its settings inside the cryostat.

In the present study, we propose a device for transmitting microwave power to a sample placed in a closed-loop helium cryostat [9] without the use of a resonator. Since

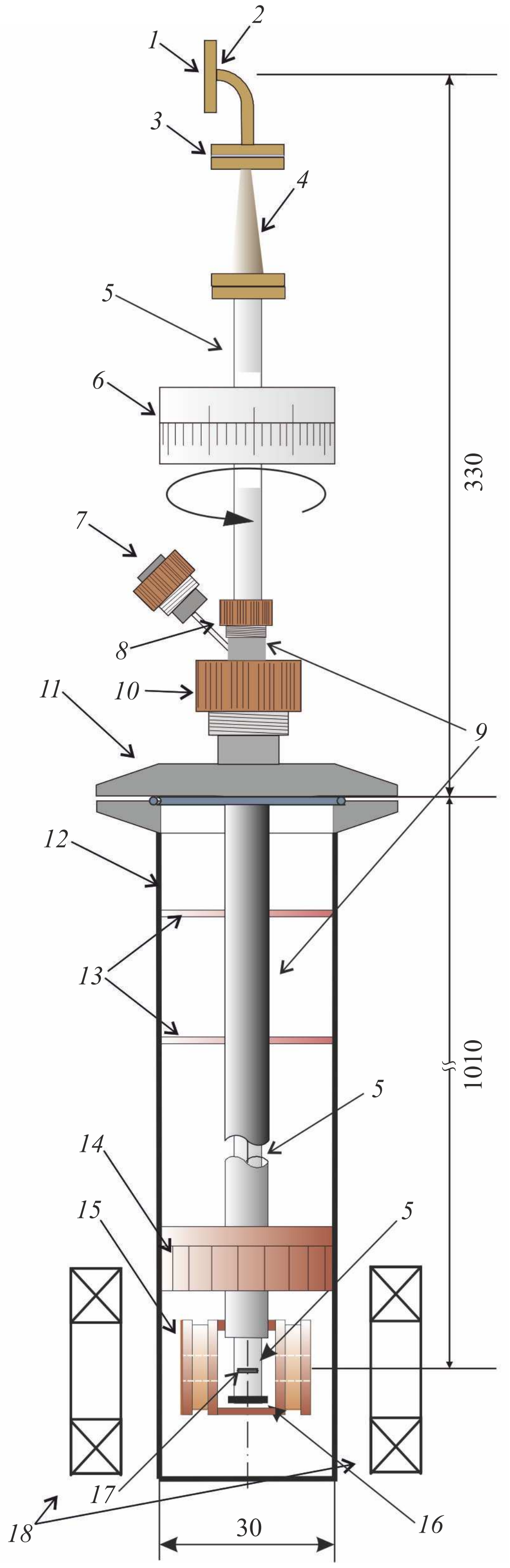

Figure 1. Schematic diagram of the device for microwave energy transmission and spectra recording in the high-frequency EPR–ODMR spectrometer without a resonator. The dimensions of the helium cryostat (in millimeters) are indicated below and on the right.

the generator emits a very narrow frequency band and all the energy is accumulated within this band, we managed to obtain a high microwave field strength at the sample under the condition of low losses in the transmission line. The device provides a high level of sensitivity in steady-state operation and supports pulsed operation and optical detection of magnetic resonance. When using a radio-frequency coil, electron nuclear double resonance may be detected.

A cylindrical waveguide is used in the presented design, which is implemented in the high-frequency (94 GHz) EPR spectrometer, to produce microwave field H_1 at the sample. The choice of a cylindrical waveguide was due to the fact that it allows one to transmit microwave power with almost no attenuation over a long distance. This is a crucial factor, since the sample is located far from the generator in a long sized cryostat. In addition, a cylindrical waveguide makes it easier to rotate the sample in the applied magnetic field when registering the angular dependence.

A non-magnetic stainless steel tube with a wall thickness of 0.2 mm was used as a waveguide. This choice is attributable, first, to the low heat capacity and thermal conductivity of stainless steel, which is very important for cryogenic applications. A low thermal conductivity is needed to minimize heat transfer from the top of the waveguide inside the cryostat down to its very bottom. The high quality of the inner surface, which is not subject to corrosion, is the second factor conditioning the choice of the tube. Although the electrical conductivity of stainless steel (specific resistance of steel is $0.103\,\mu\Omega\cdot\mathrm{m}$) is lower than that of, e.g., copper ($0.01724\,\mu\Omega\cdot\mathrm{m}$), it was found experimentally to be sufficient to obtain optimum results.

The backbone of the structure is a thin-walled tube 8 mm in diameter made of non-magnetic stainless steel. All structural elements are attached to it. Another tube with a diameter of 5 mm, which is the actual waveguide and is also made of stainless steel, is inserted coaxially inside the first one.

Figure 1 shows the schematic diagram of the device for transmitting microwave energy from a microwave generator to a sample. It features flange *1* and bent (90°) section of a rectangular waveguide *2*, which is connected with one flange to the microwave generator and with the other flange to waveguide section *4* (transitional from the rectangular waveguide to a cylindrical one). The approximate length of this section is 30 mm, which is equal to ten wavelengths (3 mm).

A thin Teflon gasket is inserted between cylindrical waveguide *5* and the flanges connecting transition *3* from the rectangular waveguide to the cylindrical one. This gasket seals waveguide *5* and isolates it electrically from the generator. A rotating joint with a goniometer *6* is required to rotate the sample in a constant magnetic field. This joint consists of two round flanges, one of which has a quarter-wave groove and a groove with a rubber ring for vacuum sealing of the joint parts inserted into it. The rotating joint is enclosed in the goniometer housing:

one of the flanges is connected to the rotating part of the goniometer, while the other is connected to the stationary part. The vacuum electrical connector *7* is used for connecting the temperature sensor and heater, which are housed in the thermostat *13*, with measuring instruments and other electrical connections. Vacuum connection *8* serves to adjust the height of waveguide *5* relative to supporting tube *9* and is used for quick sample change. Connection *10* is used to adjust the height of the supporting tube with a diameter of 8 mm relative to quick-release flange connection *11*, which secures the structure in shaft *12* of the cryostat. Tube *9* houses most of the structural elements, including copper screens *13* that obstruct convection flows of gaseous helium filling the internal shaft of the cryostat. Thermostat *14* (a massive copper cylinder) and magnetic field modulation coils *15* are also secured to this tube. The sample is positioned at the very bottom of the waveguide shorted by cover *16*. Since the cover is removable, it is easy to replace samples and adjust their position relative to the axis of superconducting solenoid *18*. Slit *17* is made in waveguide *5* to enable ODMR experiments and optical illumination of the sample.

Three main types of transverse waves may propagate along microwave transmission lines: electromagnetic (TEM), electric (TE), and magnetic (TM) ones. Rectangular and cylindrical waveguides support TE and TM waves only. These waves have the following features:

$$\text{TE}: \qquad E_z = 0, \quad H_z \neq 0,$$

$$\text{TM}: \qquad H_z = 0, \quad E_z \neq 0.$$

Waves having only the H_z (TE) or the E_z (TM) component may propagate in waveguides. The dominant wave propagating in a rectangular waveguide is TE_{10}. It has the lowest critical frequency. When a wave passes from a rectangular waveguide to a cylindrical one, a wave with dominant mode TE_{11} should be generated in the latter. However, owing to the electrical discontinuity of the waveguide and the long rectangle–cylinder transition, wave TE_{01}, which has no counterpart in the rectangular waveguide, is generated in our design. The critical wavelength for this mode is $\lambda_g = 1.64r$, where r is the waveguide radius. The radius of the waveguide made of a stainless non-magnetic steel tube with a diameter of 5 mm and a wall thickness of 0.2 mm is 2.3 mm. The critical wavelength is $\lambda_g = 3.77$ mm. The generator wavelength is 3 mm. This wave fits well into the proposed waveguide. The structure of its field is shown in Fig. 2.

The presented magnetic field line diagram indicates that the sample placed in the center of the waveguide (as in Fig. 2, *a*) will be subjected to the influence of magnetic field H_1 of the same configuration as in the cylindrical resonator (TE_{011}). Since electric currents do not flow along the waveguide walls, this mode has minimum attenuation and is used to tranmitt microwave energy over long distances, with the attenuation of the wave decreasing with increasing frequency [10]. This is very important, since the distance from the microwave generator to the sample in the cryostat is close to 1.4 m. Since the electric currents in wave TE_{01} flow in the direction normal to wave propagation (Fig. 2, *b*), it is possible to cut slits in the waveguide along its diameter. These slits are needed for penetration of low-frequency modulation, radio frequency (in ENDOR detection), and light (in optical magnetic resonance detection) into the waveguide.

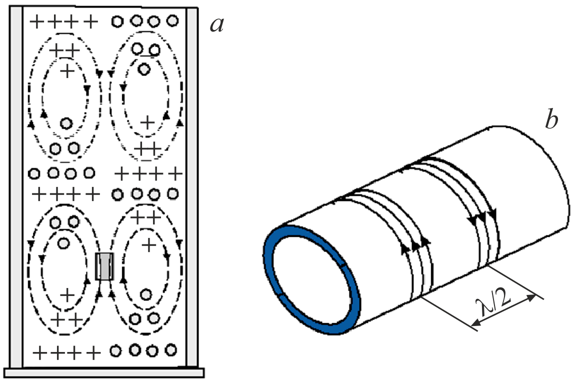

Figure 2. *a* — Structure of the TE_{01} wave that transfers energy to the sample. Magnetic lines are represented by dotted curves, while electric lines are denoted by „+“ and „o“ symbols. The rectangle marks the location of the sample. *b* — Directions of electric currents.

The TE_{01} mode may propagate in a waveguide with a diameter of 5 mm alongside with, e.g., TE_{02}, TE_{03}, and modes of even higher orders; however, their intensity decreases significantly with an increase in ordinal number. Their main feature — the flow of currents perpendicular to the direction of wave propagation — is preserved and does not alter the overall pattern of magnetic fields. Everything stated for the frequency of 94 GHz is also true for the frequency of 130 GHz, which is also used in our spectrometer.

Figure 3 illustrates the operation of the proposed device by presenting EPR spectra recorded with it.

Figure 3, *a* shows examples of EPR spectra of a Tb^{3+} ion in yttrium aluminum garnet recorded in the same sample at frequencies of 94 and 130 GHz in the conventional regime. Four lines in the spectra correspond to the hyperfine structure of the ion (nuclear spin $I = 3/2$). At 130 GHz, the Tb^{3+} spectrum is recorded in a stronger magnetic field. The spectra are presented in the same scale. Figure 3, *b* shows the electron spin echo detected EPR spectrum of Mn^{2+} in a BaF_2 crystal. Since no magnetic field modulation is used in pulse mode, six hyperfine structure lines of the Mn^{2+} ion are recorded as absorption lines.

The presented design of a device for transmitting microwave power to a sample located in a helium cryostat (at a large distance from the microwave generator) is relatively simple. It allows one to produce a high microwave field intensity at the sample without using a resonator, change samples quickly, and record angular dependences with ease.

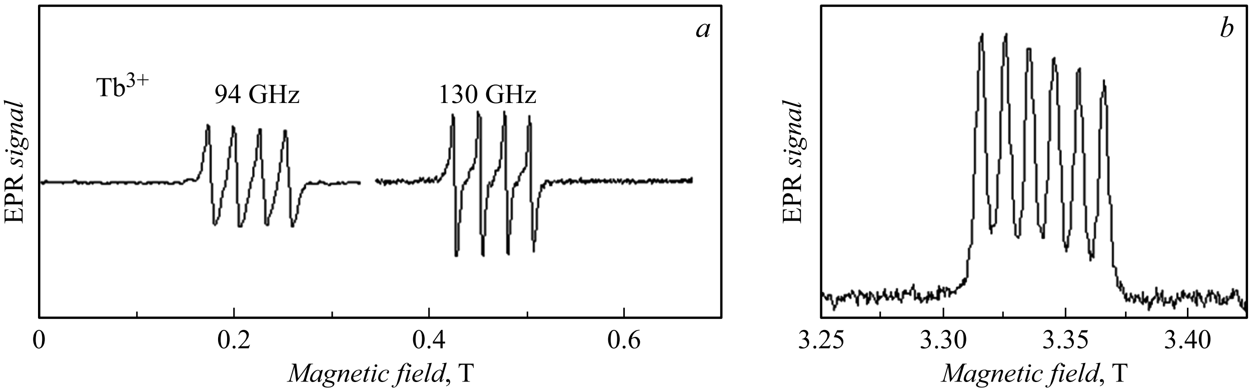

Figure 3. *a* — EPR spectra of a Tb^{3+} ion in yttrium aluminum garnet recorded in the same sample at frequencies of 94 and 130 GHz in the continuous regime; *b* — electron spin echo EPR spectrum of a Mn^{2+} ion in a BaF_2 crystal.

It provides an opportunity to record EPR, ODMR, and ENDOR spectra in continuous wave and pulse regimes. The sensitivity of the spectrometer with this device was no worse than that of the Bruker spectrometer. The use of a cylindrical waveguide with the TE_{01} mode and signal detection without a resonator offer great opportunities in the design of high-frequency EPR spectrometers with synthesizers of microwave frequencies upward of 50 GHz.

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Conflict of interest

The authors declare that they have no conflict of interest.

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