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Spectrometer for Optical Detection of the Electron Spin Resonance with an Increased Level of Microwave Power

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Abstract—A spectrometer for optical detection of the electron spin resonance (ESR) spectra of short-lived radical-ion pairs produced by ionizing radiation is described. The setup is based on a standard ESR spectrometer equipped with an adapter for optical detection of spectra and a microwave amplifier. In a solution of *n*-terphenyl in alkanes, the spin-locking effect has been observed. At a microwave power of 10 W, the microwave magnetic induction is 0.5 mT.

The method of optical detection of the electron spin resonance (OD ESR) is used in the investigation of rapid radiation–chemical primary processes initiated by ionizing radiation in solutions [1]. The method is based on recording the intensity of the luminescence that accompanies the recombination of singlet radical-ion pairs in constant (B_0) and alternating (B_1) magnetic fields.

Just with a conventional ESR spectrometer, when the intensity of the constant magnetic field is varied, the microwave field causes resonant transitions between the Zeeman sublevels of radical ions, which results in population depletion of the singlet state of radical-ion pairs. As a result, under the resonance, the intensity of the recombination fluorescence decreases.

The dependence of the luminescence intensity on the strength of a constant magnetic field provides data on the ESR spectra of recombining radical ions. In order to increase the sensitivity, a modulation technique is used with the signal registered by a synchronous-detection system at the modulation frequency (which is usually 12.5 kHz) of the constant magnetic field.

The intensity of the OD ESR spectra is proportional to the square of the microwave amplitude field [2]. Therefore, an increase in power is appropriate when weak signals are observed (owing to low-viscosity solutions, presence of quenchers of luminescence, etc.). Besides, with an increase in the microwave power, spin locking may occur, which manifests itself in variations of the OD ESR spectrum pattern and, in particular, in the appearance of opposite-phase lines [3].

Under these conditions, with an increase in the microwave power, the behavior of the spectrum pattern significantly depends on such characteristics of short-lived radical ions as their paramagnetic-relaxation times. This offers a possibility of measuring these times, which other wise is impossible when using conventional methods because of the short (1–100 ns) lifetime of radical ions. For this purpose, we developed an OD ESR spectrometer with an increased level of microwave power.

As in [4], the spectrometer is based on a standard ESR spectrometer intended for a 3-cm-wave band (a frequency of 9.5 GHz). In the device developed, while retaining the main of the characteristics spectrometer (spectrum recording through the use of the modulation technique and synchronous detection), the power level is increased and can be varied over a wide range using an additional high-power broadband amplifier (traveling-wave tube, TWT). The sample is irradiated by an X-ray tube.

The block diagram of the device is given in Fig. 1. As a base model, a Bruker ER 200D electron spin resonance spectrometer was used. The microwave signal from the klystron *K* is further amplified by the TWT amplifier *Amp*. The variable attenuators A_1, A_2 adjust the power level. The signal amplified is applied to the measuring cavity *Cav* via a circulator *Circ*. The calculated microwave-field induction in the cavity is 0.5 mT at a power of 10 W. The signal reflected from the cavity

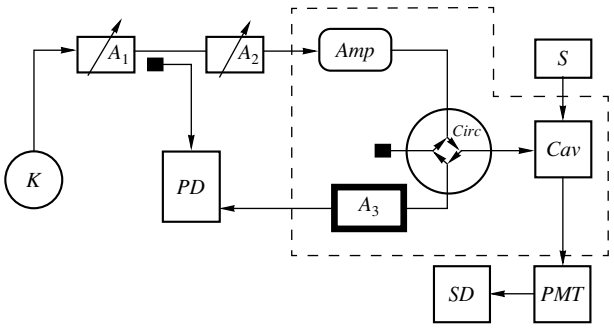

Fig. 1. Block diagram of the device.

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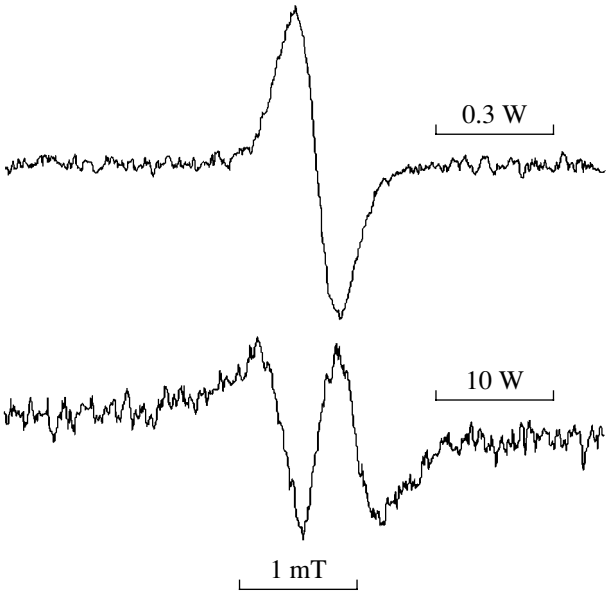

Fig. 2. OD ESR spectra of radical-ion pairs formed under X-ray irradiation in the solution of 10^{-3} M of *n*-terphenyl-d_{14} in dodecane at various levels of microwave power.

is combined with the klystron microwave signal in the phase detector *PD* and is used for frequency stabilization.

The part of the diagram outlined with a dashed line contains components with a high power-handling capacity (in this case, 12 W). To match the levels of the microwave signals, the attenuator A_3 is included into the microwave transmission line.

As the X-rays source *S*, a BSV-23 X-ray tube is used. The luminescence signal is registered by a ФЭУ-130 photomultiplier *PMT* and is isolated by the system of synchronous detection *SD*.

Using the device, we registered the radical-ion pairs $(n\text{-terphenyl-}d_{14})^{+\bullet}/(n\text{-terphenyl-}d_{14})^{-\bullet}$ in dodecane and in other alkanes at room temperature at a microwave power of 0.1–12 W. In Fig. 2, the OD ESR spectra of this system are given for two different power levels. It can be seen that, at a high power level, an inverse-phase line appears at the center of the spectrum, which is indicative of spin-locking. The assessment based on the theory of this phenomenon allows one to conclude that the behavior of the spectrum pattern is qualitatively consistent with the expected one at the given microwave-field induction in the cavity.

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