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# Development of a Wideband EPR Spectrometer with Microstrip and Loop Antennas

**A. N. Ponomaryov[1], K.-Y. Choi[1], B. J. Suh[2], and Z. H. Jang[3]***

[1]*Department of Physics, Chung-Ang University, Seoul 156-756, Korea*
[2]*Department of Physics, The Catholic University of Korea, Bucheon 420-743, Korea*
[3]*Department of Physics, Kookmin University, Seoul 136-702, Korea*

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**We have developed a new non-conventional electron paramagnetic resonance (EPR) spectrometer, in which no resonant cavity was used. We previously demonstrated a wide frequency range operation of an EPR spectrometer using two loop antennas, one for a microwave transmission and the other for signal detection [1]. In contrast to Ref. [1], the utilization of a microstrip antenna as a transmitter enhanced a capability of wide-band operation. The replacement of conventional capacitors with varactor diodes makes resonance condition easily reproducible without any mechanical action during tuning and matching procedure since the capacitance of the diodes is controlled electronically. The operation of the new EPR spectrometer was tested by measuring a signal of 1,1-diphenil-2-picrylhydrazyl (DPPH) sample in the frequency range of 0.8-2.5 GHz.**

**Keywords :** electron paramagnetic resonance, microstrip, varactor diode, microwave tuning and matching circuit

## 1. Introduction

Since 1944, after the discovery of the electron paramagnetic resonance (EPR or ESR - electron spin resonance), the EPR has been applied in a variety of fields ranging from chemistry and biology to the stimulating areas of quantum computation and single spin detection in condensed matter physics. The EPR has excelled over any other techniques in determining magnetic parameters of a spin Hamiltonian as well as in obtaining g-factors, spin and orbital states of paramagnetic ions, which depend on their site symmetry, interactions between magnetic moments, and spin numbers [2].

The conventional EPR, based on the use of a resonant cavity, makes use of a field sweeping scheme to detect a resonance at a fixed microwave frequency. This scheme is employed mainly due to the limitation of the sweeping range of the traditional microwave sources and the calibration problem at microwave frequencies to obtain the absolute transmission/reflection. Although the conventional EPR operating at a fixed frequency has an advantage in sensitivity, it has certain restrictions: to lower the microwave frequency, one has to increase cavity size, which is often practically impossible [3].

On the one hand, this scheme makes it difficult to study very small zero-field splitting (zfs) parameters, which are often existent in single molecule magnets [4-10]. On the other hand, a field sweeping EPR introduces a complexity in the spectrum of magnetic compounds, if the compounds show field-induced novel phenomena. Examples of the novel phenomena encompass field-induced antilevel crossings and coherent tunneling of the magnetization reported in molecular antiferromagnetic rings, and a skyrmion of the chiral helimagnet MnSi stabilized at a finite field [11, 12]. These obstacles can be circumvented by performing EPR experiments in a sweeping frequency at a fixed field.

Recently, instrumental progresses have been made toward a frequency sweeping EPR [1, 13-15]. Either tunable microwave sources or vector network analyzers are used to provide variable frequency microwave sources. For a microwave transmission and a signal detection, two loop antennas, striplines, and a microcoil are employed. However, each scheme has drawbacks. For example, when the loop antennas are used, one has to apply tuning and matching networks for both transmitting and detecting antennas. In other word, for changing operation frequency it is necessary to tune and match both antennas simultaneously, which is

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*Corresponding author: Tel: +82-2-910-4768
Fax: +82-2-910-4728, e-mail: zeehoonj@kookmin.ac.kr

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usually a complicated procedure. Here we present a newly developed non-conventional EPR spectrometer, in which microstrip antenna and loop antenna are used as a transmitter and a detector, respectively. With the use of the microstrip antenna, we could avoid using tuning and matching network on a transmitter side and widened the frequency range of operation.

## 2. Experimental Setup

A new non-conventional EPR spectrometer is schematically shown in Fig. 1. As a source of microwave, Agilent Technologies E8257D PSG analog signal generator is used. For a microwave transmission, a commercially manufactured microstrip antenna is used. The antenna is made of 0.5 mm thick G10 circular substrate. On one side of the substrate, a 2 mm wide copper microstrip is deposited and the ground plane is located at the opposite side. The antenna has been matched to 50Ω impedance by the manufacturer and terminated with a 50Ω chip resistor. The microstrip antenna is connected to the signal generator with 0.085 inch outer diameter semi-rigid coaxial cable and the same semi-rigid cables were extensively used as interconnecting microwave transmission lines in other parts of the spectrometer. For detecting the microwave signal, a single turn loop antenna, made of 0.8 mm diameter copper wire, is used. The diameter of the loop is 5 mm and the value is being chosen from a numerical calculation for the best reception sensitivity considering the operation frequency range of 0.1-3 GHz.

For tuning the loop antenna to the target frequency and matching it to a standard impedance of 50Ω, a tuning and matching circuit is designed and constructed (see Fig. 2). In contrast to a standard tuning and matching circuits where mechanically variable capacitors are usually used, we developed a circuit which is based on varactor diodes and two bias-tees. The bias-tees are used to apply bias voltages to the varactor diodes for the capacitance change, and are powered with a programmable power supply. The power supply can supply voltage in the range from 0 to 30 V with 0.01 V steps, making tuning and matching very precise. On the other hand, the power supply is controlled by computer through RS232 serial port which makes tuning and matching procedure automatic, simple and easily reproducible.

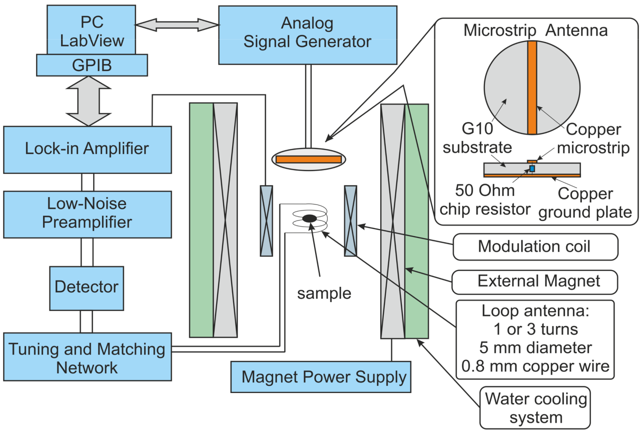

**Fig. 1.** (Color online) Schematic representation of the non-conventional EPR spectrometer: a 50 Ohm terminated microstrip antenna is used as a microwave transmitter and a single-turn 5 mm loop antenna is used as a receiver.

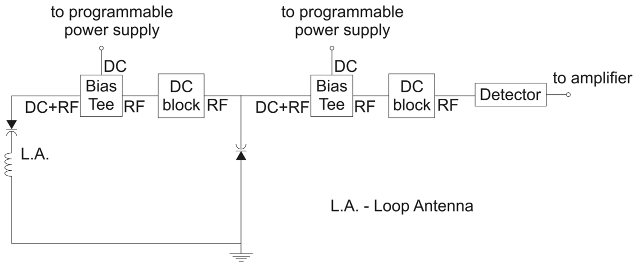

**Fig. 2.** Tuning and matching circuit at the receiver.

At output of tuning and matching circuit, the DC current is cut off by Pasternack PE8210 DC-block to protect detector from DC. By using Pasternack zero bias Schottky diode detector PE8012, the microwave signal is converted into DC, of which the voltage is proportional to the power of the microwave entered into the detector. After that, the signal from the detector is amplified with the SR560 low-noise preamplifier and then fed into SR830 DSP lock-in amplifier for the phase lock signal detection.

The lock-in amplifier is basically tuned amplifier and is used to detect signals of a low signal-to-noise ratio and functions as following. With internal oscillator, the amplifier generates sinusoidal reference signal of known frequency. Applying the reference signal to a modulation coil, a modulation magnetic field is generated. With the modulation field, the signal proportional to the field derivative of the EPR absorption is detected. Because of the tuned amplifier feature of the lock-in amplifier, the signal-to-noise ratio is increased and, since the detected signal is proportional to the slope of the EPR absorption signal, it is further enhanced. For a more detailed description of lock-in amplifier work principles we refer, for example, to Ref. [16].

For the generation of the modulation field, a modulation coil is made of 0.11 mm copper wire wound on a 10 mm long plastic bobbin. The coil consists of approximately 1000 turns and generates a modulation field of 9 Gauss at the frequency of 1 kHz when 5 V is applied. The modulation coil is placed in the spectrometer in such a way that the modulation field is parallel to the external DC magnetic field.

To generate an external DC magnetic field, a homemade magnet is made. The magnet consists of 290 turns of 3 × 2 mm copper wire wound on a 115 mm long bobbin. Inner diameter of the bobbin is 35 mm. In order to power the magnet, we used the Oxford Instruments superconducting magnet power supply IPS 120-10. To prevent damage of the magnet by the heat, the magnet was protected with homemade water-cooling system. The system is made of a metal hollow cylinder and two plastic caps. The magnet is put in the cylinder and the composite is hermetically sealed. Inlet and outlet at the cylinder allowed water to flow through the inner space of the cylinder. When 30A direct current is applied, the magnet generated magnetic field of 800 Gauss. The numbers were estimated from test measurement of EPR signal of DPPH sample at microwave frequency of 2.5 GHz.

For EPR data acquisition a special LabView program has been developed. The program allowed us to control the lock-in amplifier and the magnet power supply remotely, as well as to change their settings, such as sensitivity and time constant for the lock-in amplifier and magnetic field sweeping rate and range for the magnet power supply. By simultaneously reading out of the values of the current from the magnet power supply and the signal strength from the lock-in amplifier, we were able to record the derivative of EPR absorption as a function of current. By using the calibrated conversion factor, we were able to convert the current to the magnetic field in Tesla unit.

## 3. Characterization and Test of the EPR Spectrometer

When working on the design of the EPR spectrometer, we found that for the measurements in a wide frequency range, one has to apply a transmitter, which would have frequency independent transmittance in the required frequency range. In addition, we also found that a microstrip antenna is the most reasonable solution of the problem. Ideally, such antenna must have flat standing wave ratio (SWR), but, in practice, it is hard to make such antenna. By using the Agilent Technologies E5071A network analyzer, we tested characteristics of the microstrip antenna that we used for the EPR spectrometer. Fig. 3 shows the transmittance of the microstrip antenna as a function of frequency measured in the range from 0 to 8.5 GHz. As it is seen from the plot, the transmittance nearly equals to 1 in the range from 0 to 3 GHz, and decreases with frequency, reaching its minimum around 7 GHz.

An ideal microstrip antenna is known to have an infinite bandwidth if the impedance is matched to the characteristic impendence of the connecting transmission line, 50 ohm in usual coaxial cable. Practically the impedance matching condition can not be satisfied perfectly. The commercially manufactured microstrip has an impedance error of +/– 5 ohm which deteriorates the impedance matching condition and reduces the bandwidth of the microstrip. Additionally, because of the circular shape of our microstrip, the impedance at the edge deviates from the right value of 50 ohm. We believe that those are the reasons why our microstrip antenna has limited bandwidth of ~3 GHz. In future, a precision engineered microstrip with careful impedance control procedure will enhance the bandwidth of the microstrip and further extend the frequency range of the spectrometer.

We also performed measurements of the microwave power, transmitted by a 50 Ohm terminated microstrip antenna and detected by two different loop antennas. Fig. 4 shows the frequency dependence of microwave power, detected by three-turn loop antenna in the frequency range from 0.1 to 10 GHz. Note that, although the loop antenna was roughly tuned and matched to 1.546 GHz, several maxima of the detected power are observed. Moreover, some peaks are more intense than the one at 1.546 GHz. This can be explained with the characteristics of the microstrip antenna, of which the value of SWR is smaller at frequencies other than 1.546 GHz. Noticeably, in the frequency range from 0.1 to 3 GHz, the power maxima are separated evenly by approximately 130 MHz, while at

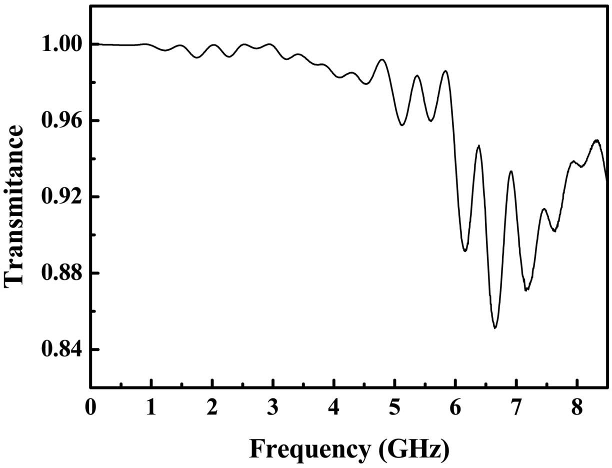

**Fig. 3.** Tranmittance of the microstrip antenna, which was used as a transmitter for the EPR spectrometer, measured as a function of frequency in the range from 0 to 8.5 GHz.

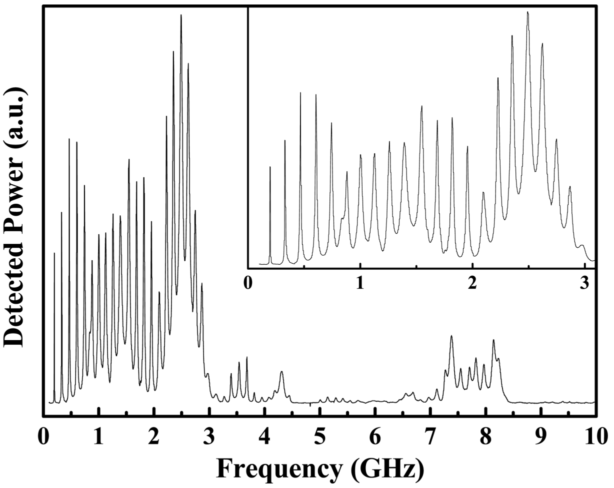

**Fig. 4.** Frequency dependence of microwave power, transmitted by a 50 Ohm terminated micristrip antenna and detected by three-turn loop antenna. The loop antenna was tuned and matched to 1.546 GHz. The measurements were done in the frequency range from 0.1 to 10 GHz.

higher frequencies the maxima are weak or even absent.

The frequency dependence of microwave power, detected by a single turn loop antenna in the frequency range from 0.1 to 2.6 GHz, is shown on Fig. 5. The antenna was tuned and matched to 1.106 GHz. As in the case of the three-turn loop antenna, several power maxima are also detected. However, the separation between them is around 145 MHz, which is by 15 MHz larger than that for the three-turn loop antenna.

We would like to note that maxima of the detected power are also observed for those frequencies, to which the loop antennas were tuned and matched, and thus, the EPR spectrometer operation frequency can be easily changed by changing tuning and matching characteristics of the loop antenna. Besides that, one also can measure EPR spectra at all those frequencies, for which the power maxima are observed. Below we present the test results of the EPR spectrometer, performed with a single-turn loop antenna at selected microwave frequencies for which the power maxima are observed (see Fig. 5). Since the single-turn loop antenna detected more microwave power in absolute values than the three-turn loop antenna, it was decided to perform the test of the whole EPR spectrometer with single-turn loop antenna.

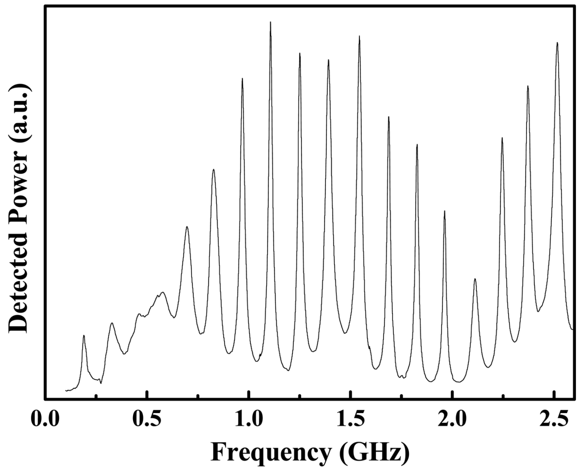

**Fig. 5.** Frequency dependence of microwave power, transmitted by a 50 Ohm terminated microstrip antenna and detected by a single-turn loop antenna. The loop antenna was tuned and matched to 1.106 GHz. The measurements were done in the frequency range from 0.1 to 2.6 GHz.

For testing newly developed EPR spectrometer, we used 11.4 mg of DPPH, put in a gelatin capsule. The resonance condition (frequency vs. field) of DPPH is well known with the calibrated g-factor of 2.0036 and thus DPPH is most popular reference sample for EPR spectrometer testing [17]. The single-turn loop antenna was used. Tuning and matching conditions were adjusted to the frequency of 1.106 GHz. Since for the same tuning and matching condition several power maxima are observed, it was decided to measure EPR spectra at those frequencies, for which the detected microwave power is significant. The measurements were done at room temperature.

Fig. 6 shows EPR spectra of DPPH measured at 0.824, 1.106, 1.542, and 2.244 GHz, respectively. The roughly estimated signal-to-noise ratios are 77, 300, 400 and 290, respectively. As one could expect, the spectra are of different intensities and their ratios are in a very good agreement with detected power values in Fig. 5. The most intensive

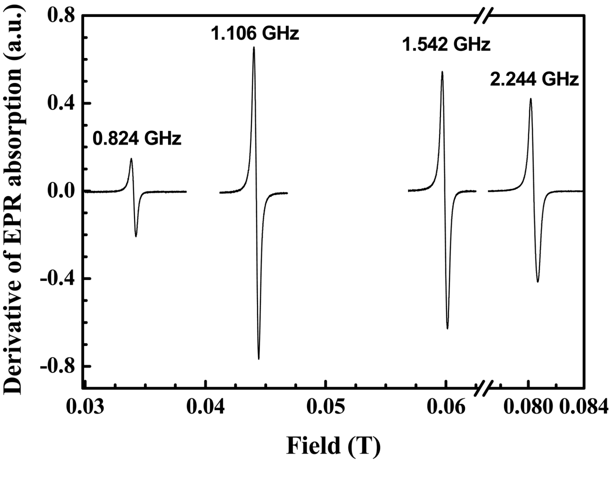

**Fig. 6.** EPR spectra of 11.4 mg of DPPH, measured at 0.824, 1.106, 1.542, and 2.244 GHz at room temperature. The single-turn loop antenna was tuned and matched to 1.106 GHz.

EPR spectrum was recorded for the frequency, to which the loop antenna was tuned and matched, and thus, to optimize measurement conditions for other frequencies one simply needs to tune and match the receiver loop antenna to a desired frequency.

## 4. Conclusions

In this paper we have presented a new non-conventional EPR spectrometer, in which microstrip and loop antennas were used as a transmitter and a detector, respectively. We have demonstrated that such a scheme allows measuring EPR spectra in a wide frequency range. Both the capability of wide-band operation and the sensitivity have been improved compared to the previous EPR spectrometer with two loop antennas. In addition, by replacing capacitors with varactor diodes the precision and the reproducibility of tuning/matching procedure has been enhanced. The operation of the spectrometer has been tested successfully in a frequency range from 0.8 to 2.5 GHz using a homemade magnet.

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