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Uniform Field Loop-Gap Resonator and Rectangular TE_{U02} for Aqueous Sample EPR at 94 GHz

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Abstract

In this work we present the design and implementation of two uniform-field resonators: a seven-loop–six-gap loop-gap resonator (LGR) and a rectangular TE_{U02} cavity resonator. Each resonator has uniform-field-producing end-sections. These resonators have been designed for electron paramagnetic resonance (EPR) of aqueous samples at 94 GHz. The LGR geometry employs low-loss Rexolite end-sections to improve the field homogeneity over a 3 mm sample region-of-interest from near-cosine distribution to 90% uniform. The LGR was designed to accommodate large degassable Polytetrafluorethylen (PTFE) tubes (0.81 mm O.D.; 0.25 mm I.D.) for aqueous samples. Additionally, field modulation slots are designed for uniform 100 kHz field modulation incident at the sample. Experiments using a point sample of lithium phthalocyanine (LiPC) were performed to measure both the uniformity of the microwave magnetic field and 100 kHz field modulation, and confirm simulations. The rectangular TE_{U02} cavity resonator employs over-sized end-sections with sample shielding to provide an 87% uniform field for a $0.1 \times 2 \times 6$ mm³ sample geometry. An evanescent slotted window was designed for light access to irradiate 90% of the sample volume. A novel dual-slot iris was used to minimize microwave magnetic field perturbations and maintain cross-sectional uniformity. Practical EPR experiments using the application of light irradiated rose bengal (4,5,6,7-tetrachloro-2',4',5',7'-tetraiodofluorescein) were performed in the TE_{U02} cavity. The implementation of these geometries providing a practical designs for uniform field resonators that continue resonator advancements towards quantitative EPR spectroscopy.

Keywords

Electron paramagnetic resonance spectroscopy; microwaves; uniform field cavity; aqueous samples; high frequency; light access

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1. Introduction

In this work we present two new geometries for electron paramagnetic resonance (EPR) of aqueous samples at 94 GHz (W-band): i) a seven-loop–six-gap (LGR) with enhanced end-sections, shown in Fig. 1A and ii) a rectangular TE_{U02} cavity with over-sized end-sections, shown in Fig. 1B. These resonators take advantage of uniform field methodology for improved quantitative experiments.

Methods to produce a uniform microwave field in cavity resonators were introduced by Mett *et al.* [1–4] for cavity and re-entrant geometries and later for loop-gap resonators [5].

Uniform field cavities are defined as resonant structures at cut-off with a strictly uniform microwave magnetic field over a region-of-interest extending parallel to the propagation vector of the cavity. In order for the central section waveguide at cut-off to satisfy Maxwell's equations, a perfect-magnetic boundary condition must be matched to the central section by each end-section. Three matching methods were shown to produce this effect: i) over-sized end-sections above cut-off, ii) re-entrant metallic structures that adding capacitance to the end-section, and iii) dielectric end-sections of length $\lambda/4$ placed at each shorted end. All end-section types present an rf open impedance boundary condition to the central cut-off section. These methods are illustrated in Fig. 2.

Sidabras *et al.* introduced a five-loop–four-gap LGR at 94 GHz [6]. The LGR at W-band permitted a number of experiments that required high resonator efficiency and bandwidth, such as ELDOR, saturation recovery, and frequency sweep experiments [7, 8]. The LGR was designed for a 0.15 mm I.D. quartz capillary with an active region of 1 mm. A method for degassing the sample outside of the resonator was devised. However, this only permitted 100% air or 100% nitrogen degassing. In order to permit air/nitrogen gas mixture degassing in the resonator, a design for Polytetrafluorethylen (PTFE) capillaries was devised. The seven-loop–six-gap resonator was designed with a 0.825 mm sample loop to accommodate Zeus Inc. (Orangeburg, SC, USA) extruded PTFE sample tubes with a 0.25 mm I.D. and 0.81 mm O.D.

Recently, Sidabras *et al.* introduced methodology to produce uniform 100 kHz field modulation incident on the sample by numerically optimizing the modulation slot depth [9]. In the present work, this methodology was used to optimize, simulate, and implement uniform 100 kHz field modulation slot design in the seven-loop–six-gap LGR geometry. By providing a strictly uniform 100 kHz field modulation and enhanced microwave magnetic field uniformity, we provide a model for resonator design for quantitative EPR at high frequencies [10].

The rectangular TE_{U02} , shown in Fig. 1B, was designed for light access experiments at W-band. Light access slots were designed to have 90% illumination with -100 dB microwave leakage [11]. A flat-cell sample with inner dimensions of $0.1 \times 2 \text{ mm}^2$ from VitroCom (Mountain Lakes, NJ, USA) was chosen. The large aspect-ratio provides a thin surface for reduced light refraction. The uniform field method of oversized end-sections, illustrated in Fig. 2A, is employed with sample shields to limit the microwave magnetic field incident on

the sample to only the region-of-interest. Sample shields provide a metallic sleeve that extends into the end-sections where the microwave magnetic field is non-uniform.

The seven-loop–six-gap LGR reported here is the first practical example of a uniform field LGR as introduced in Ref. [5]. The rectangular TE_{U02} geometry is the first practical implementation of the rectangular geometries introduced in Ref. [1] and extensively studied in Ref. [2] with two notable differences. First, the geometry described here provides light-access slots. Second, the null index is extended compared to a traditional rectangular TE_{102} cavity providing two dimensions of uniform field (the z -direction and the null x -direction).

The use of uniform field resonators provides a number of advantages to traditional resonators with a cosine dependence in the transverse z -direction. A uniform field resonator i) provides better quantitative measurements reducing the need to calibrate the resonator and modulation profile [10]; ii) allows the region-of-interest to be extended to provide a larger sample volume, increasing the EPR concentration sensitivity; iii) perform reliable CW saturation studies; iv) in pulse experiments with need for coherent pulses (such as ESEEM and HYSCORE) provides the same magnetic field excitation along the entire sample volume [12]; and v) provides uniform excitation for arbitrary-wave form shaped inversion pulses [13] and frequency modulation sweeps.

2. Methods

Finite-element simulations were performed on a Dell Precision T7500 workstation with dual six-core Xeon 5650 2.67 GHz processors with 12 MB of L2 Cache per chip and 124 GB of system DDR4 RAM. A RAM drive was set up with 8 GB of RAM in order to reduce hard-drive bottlenecks. This system has been optimized for simulations and new versions of ANSYS (Canonsburg, PA, USA) High Frequency Structure Simulator (HFSS; v. 17.2) are able to take advantage of all twelve CPUs during finite-element modeling matrix solving. The operating system was Windows 7 64-bit. The eigenmode and driven mode solvers were used and typical simulation times were 10 minutes. All simulations were performed at 95 GHz (W-band).

A sample holding technique using a slot and clamp structure has been implemented in the LGR to hold 0.81 mm outer diameter PTFE tubing [14]. Holding such tubing was found in the past to be challenging and would result in an unstable tune. In current experiments, the resonator and tuning structure has held exceptionally well for hours. The new resonator structure has a gas exchange port designed in the narrow face of the waveguide. In the rectangular TE_{U02} cavity, flat-cell sample holding is accomplished using PTFE tape to hold the sample in place.

Both geometries were designed using the 3D software tool, AutoDesk Inventor Professional. Integrity Wire EDM (Sussex, WI, USA) performed the electric discharge machining (EDM) manufacturing. Two types of EDM are available: die-sink and wire-cut. Wire-cut EDM uses a thin wire at a high potential (the part to be machined is grounded) under an oil bath to cut the through features. For high precision blind cuts, die-sink EDM is used. Die-sink EDM uses a mandrel to etch away a given pattern. EDM provides positional tolerances down to

0.001 mm. Wire-cut EDM allows feature sizes down to 0.05 mm (0.03 mm wire) and die-sink is limited to the mandrel manufacturing. All electrodes were tungsten. EDM is considered a “zero force” manufacturing technique and, as such, small features are easily manufactured compared to traditional machining. EDM fabrication techniques are essential to high-precision high frequency resonator development.

The LGR was fabricated from oxygen-free copper for resonator body and end-sections. The rectangular TE_{U02} cavity was fabricated from three-nine (0.999 pure) silver for the resonator body and over-sized end-sections. Both resonator bodies are encased in a graphite shield. The graphite shield minimizes microwave leakage and environmental rf noise.

Bench tests were performed on the Agilent Technologies (Keysight Technology; Santa Rosa, CA, USA) PNA-X model N5242A vector network analyzer with an Agilent N5262A millimeter head controller, two OML V10VNA2 T/R 75 to 110 GHz millimeter wave extender, and an OML V10-CAL calibration kit.

Each resonator iris is designed in Ansys HFSS to be over-coupled. Fine-tuning performed using a tungsten rod protruding into the wide face of the WR-10 waveguide and adjusted by a nylon screw. The tungsten rod placed $(n + 5/8)\lambda_g$ from the resonator iris, where n is 3 wavelengths and λ_g is the wavelength of the microwaves at 94 GHz. It was found that by placing a pill at $5/8\lambda_g$ from the resonator iris achieves minimal frequency pulling.

EPR measurements using a point sample of lithium phthalocyanine (LiPC) were used to quantify the uniformity of the LGR with enhanced end-sections. EPR measurements were performed on a home-built W-band spectrometer [15]. Measurements were made at room temperature, with a 2.5 mT sweep width over 90 seconds and a receiver time constant of 10 ms. The power was at 10 μ W, well below saturation. The field modulation was set to 0.1 mT at 100 kHz. The LiPC sample was placed in a quartz capillary and precisely moved at 0.5 mm increments through the region-of-interest. Measurements of the amplitude and peak-to-peak width were recorded. EPR signal intensity is proportional to B_1^2 , while the peak-to-peak linewidth is proportional to B_m if the sample is sufficiently over-modulated.

EPR measurements using 0.1 mM rose bengal (4,5,6,7-tetrachloro-2',4',5',7'-tetraiodofluorescein) in the presence of 0.5 mM ascorbate in the rectangular TE_{U02} cavity were performed with 10 mT sweep width over 190 seconds and a receiver time constant of 10 ms. The power was at 100 μ W, well below saturation. Field modulation was set to 0.4 mT at 433 Hz. A 532 nm wavelength laser was affixed outside of the magnet. An attenuating filter was attached to the laser to minimize bleaching of the sample during acquisition.

The effect of the magnetic field variation on the sample should be taken into account when calculating the resonator efficiency, which is normally defined at a point and assumes uniform field [16]. For example, a cylindrical TE_{011} cavity has an H_1 field that is cosine in the transverse z -direction. In this work, we define the resonator efficiency as an average over the sample volume,

$$\Lambda_{ave} = \frac{\int B_{1r} dV}{(P_s + P_w + P_t)^{1/2} V} [mT/W^{1/2}], \quad (1)$$

where B_{1r} is the clock-wise (or counter clock-wise) rotational component of the linear magnetic field perpendicular to the static magnetic field, in milliTesla, integrated over the sample volume, V . In addition, P_s , P_w , and P_t are the power losses associated with the sample, walls, and sample holder, respectively. The Λ_{ave} -to- Λ_{max} ratio can be used as a metric to the uniformity of the resonator.

EPR signal intensity and Λ values were calculated using ANSYS HFSS solutions [17] and tabulated for comparison with the previous resonators [6]. Two EPR signal conditions are calculated: signal unsaturable and signal saturable. Signal unsaturable is defined as the EPR signal intensity at constant incident power, while signal saturable is defined as the EPR signal intensity at constant microwave magnetic field.

3. Design

3.1. Uniform Field Rectangular TE_{U02} Cavity

Illustrated in Fig. 3A is a half-structure CAD drawing of the fabricated rectangular TE_{U02} cavity. The light access slots and over-sized end-sections are shown. The over-sized end-sections also include a sample end-section shield.

Testing the rectangular resonator uniformity is straightforward. Since the resonant frequency is highly dependent on the sample geometry we must test the resonator uniformity using a network analyzer and not by perturbing sphere methods or measuring the EPR signal of a point sample. In order to obtain the cut-off frequency of the region-of-interest flat end-sections were made and attached to the resonator body to form a typical rectangular TE₁₀₂ cavity. The frequency with flat-cell and aqueous sample was measured using the Agilent PNA-X network analyzer to be 96.2 GHz. From the measured frequency of the TE₁₀₂ cavity, the cut-off frequency can be calculated which would include mechanical imperfections, iris, and sample variations. The cut-off frequency was calculated to be 92.95 GHz. The over-sized end-sections are then installed and the resonator frequency with flat-cell and aqueous sample was recorded as 93.21 GHz. The resonator frequency being higher than the cut-off means the magnetic field profile is slightly cosine. In Ansys HFSS, one can adjust the geometry to match the region-of-interest cut-off frequency and the full assembly frequency. Simulating the modified geometry yields a microwave magnetic field that is 87% uniform.

The rectangular TE_{U02} cavity is coupled by a novel dual long-slot capacitive iris. Each slot is 0.1 mm wide and extends the entire height of a WR-10 waveguide (2.54 mm). The dual long-slot iris minimizes coupling to higher-order modes that arise from the long (6 mm) uniform region-of-interest and the wide 2.35 mm zero index. Coupling to higher-order modes degrades the uniformity of the resonator. With the dual long-slot capacitive iris, only the TE_{U02} mode is coupled. The symmetry of the magnetic field is shown in Figs. 3B and 3C.

Also shown in Figs. 3B and 3C are the evanescent fields in the light access slots. The inside geometry of the rectangular resonator edge is illustrated as a dotted line in Fig. 3C. The depth of the light slots is designed to have more than -100 dB of microwave leakage. Any remaining leakage is absorbed by the graphite shield.

3.1.1. Dual Iris Design—The need for dual iris design is qualitatively illustrated in Fig. 4 using a TE₁₀₂ rectangular cavity. Plotted in Fig. 4 are simulations of the x - and y -components of the electric field, denoted E_x and E_y . Our (x, y, z) coordinates have the corresponding mode indices (m, n, p) , which correlate to the conventional waveguide mode index order TE _{p m n} . The contour plots show the real part of the peak value of $(E_x + E_y)$ over the cross-section of the resonator. Here we compare a conventional TE₁₀₂ rectangular cavity cross-section which has an zero-index length ($x = a$) less than the two-index length ($y = b$), illustrated in Fig. 4A. The electric field is strictly in the x -direction.

In the typical TE₁₀₂ configuration, the resonant frequencies of the nearby TE₁₁₁ and TE₁₁₂ modes are sufficiently higher than the TE₁₀₂ mode and, therefore, no coupling occurs with a single iris excitation. However, as the zero-index length a is extended, as illustrated in Figs. 4B-E, the TE₁₁₁, TE₁₁₂ and TE₁₂₁ resonant frequencies can approach the resonant frequency of the TE₁₀₂ mode. The resonance frequencies of a TE (or TM) rectangular cavity mode are described by

$$f = \frac{c}{2} \sqrt{\left(\frac{p}{h}\right)^2 + \left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2}, \quad (2)$$

where c is the speed of light in the resonator, TE _{p m n} is the resonator nomenclature, (a, b, h) correspond to the (x, y, z) cavity dimensions and the indices (m, n, p) correspond to the number of half-wavelengths of microwave field in each dimension [18]. As a approaches b , the m and n indices can be interchanged without changing the resonance frequencies. Using the dimensions $a = 2.35$ mm, $b = 3.13$ mm and neglecting the p term, it can be shown from this equation that the nearest-neighbor modes to the TE₁₀₂ are the TE₁₁₁ and the TE₁₁₂ with frequencies 17% below 20% above that of the TE₁₀₂, respectively. The TE₁₁₂ is 42% above that of the TE₁₀₂. These frequency ratios match those produced by the simulations of Figs. 4B-E.

A necessary but insufficient condition for a cavity mode to couple to the iris is the same as the condition for the excitation of a second-order lumped circuit response, namely that the resonance frequency of the mode f_0 and the excitation frequency be within [18]

$$\frac{\Delta f}{f_0} \lesssim \frac{1}{Q}, \quad (3)$$

where Q is the Q -value of the mode. The Q values of the undesired modes can be very low because the electric fields of these modes pass through the aqueous sample, unlike the

TE₁₀₂. The simulated Q -values and frequencies, shown in Table 1, observed in the pure-mode simulations were 19, 236, and 66 for the TE₁₁₁, TE₁₁₂ and TE₁₂₁ respectively. One can compare these values to the reciprocals of the percent frequency deviations given above, suggesting strongest coupling to the TE₁₁₁ and TE₁₂₁ modes with a single iris. It should be noted that, this is further complicated by the introduction of the oversized end-sections needed to match the region-of-interest in uniform field resonators which will lower the frequencies of the higher-order modes and increase the coupling.

The other condition for coupling is that the iris microwave fields be of a similar polarization as the mode. The long-slot iris minimizes mode perturbation by having an excitation 180 degrees out of phase from the mode [19] creating a propagation into the cavity. The single and dual iris excitation is illustrated as an inset in Fig. 4. The coupling strength is proportional to the integral of the dot product of the iris fields and the mode fields over the volume [20]. As illustrated in Fig. 5A, a single long-slot iris couples to the TE₁₀₂ with some level of TE₁₁₁, TE₁₁₂ and possibly TE₁₂₁ modes also present causing significant distortions to the electric and magnetic fields. Because the fields of the single iris are so large and of proper polarization over a volume, the single iris effectively couples to the unwanted nearby cavity modes, illustrated in Fig. 5A.

In order to improve the coupling coefficient to the TE₁₀₂ mode and reduce the coupling to the unwanted modes, a perturbation can be introduced that is orthogonal to the unwanted modes. A dual iris, illustrated in Fig. 5B, meets this type of perturbation. With a dual iris, two perturbations with the same electric field direction (no phase shift) are introduced symmetrically spaced apart on the cavity wall. The perturbation introduced by one iris is largely canceled by the other for each of the unwanted modes. Extending this analysis to the TE_{U02} cavity, the purity of the TE_{U02} mode is significantly improved, as shown in Figs. 3B and 3C.

3.2. Seven-Loop–Six-Gap LGR with Enhanced End-Sections

Illustrated in Fig. 6A is a half-structure CAD drawing of the fabricated seven-loop–six-gap LGR. The seven-loop–six-gap geometry is illustrated in Fig. 6B. The large inductance of the sample loop requires a significant reduction in capacitance. A six-gap geometry was chosen to reduce the total capacitance by the number of gaps. To accomplish this a gap with a small parallel area was produced. The gap and the outside loops were connected by a flared gap. The magnetic field profile is shown in Fig. 7.

The creation of “imperfect” gaps produces a lower filling factor and efficiency. An Ansys HFSS simulation is shown in Fig. 7B, which shows the magnetic field is not fully contained within the sample loop. Due to the boundary conditions and standing electromagnetic waves of a resonant geometry, the electric field is concentrated at the magnetic field nulls (dark blue). This resonant structure could be described as a reentrant cylindrical TE₀₁₁ geometry. We chose to use the LGR nomenclature.

It was shown in Mett *et al.* that by increasing the size of the outer loops, compared to the inner sample loop, the input impedance of the LGR approaches the characteristic impedance of the WR-10 waveguide [19]. This important finding has two consequences. First, the long-

slot capacitive iris width approaches the width of the waveguide. This reduces the stored energy of the iris resulting in less magnetic field perturbation. Second, by reducing the stored energy of the iris, the frequency shift expected by matching with a tuning pill is significantly reduced. This is due to the lower reactance of the system. Using Ansys HFSS, it was found that a circular hole iris produces a 160 MHz frequency shift during matching. The frequency shift is reduced to less than a 80 MHz during matching with the long-slot iris design.

Illustrated in Figs. 6A and 6C are the dielectric enhanced end-sections. The enhanced end-sections are a practical compromise for creating uniform field LGRs at high frequencies [5]. By adding dielectric end-sections to a shorted LGR, a uniform field in a region-of-interest can be realized. As the physical size of the resonators decreases with frequency, it becomes impractical to machine dielectric end-sections with the loop-gap geometry of the shorted LGR. However, it is possible to design the LGR with an open end-section and a low-loss dielectric to approximate the boundary condition. This becomes possible as the number of gaps increases and the LGR approaches the magnetic field of a re-entrant cylindrical waveguide. Therefore, a seven-loop–six-gap resonator can approximately match a short transformed by a $\lambda/4$ dielectric slab [18], similar to the uniform field method of Fig. 2C. However, this method is only an approximation to an open impedance and a true uniform field may not be possible due to the mismatch. Ansys HFSS simulations, shown in Fig. 7A, have a 90% uniform magnetic field over the 3 mm region-of-interest.

As the LGR length increases and the inner sample loop becomes larger, a near-cosine distribution of the magnetic field to occurs. For instance, without end-sections the magnetic field is 60% uniform over the 3 mm region-of-interest, similar to a cylindrical TE_{011} . Therefore, the above methodology corrects for longer LGR geometries and large sample loop. With such a geometry an increase in EPR concentration sensitivity can be achieved. The 3 mm region-of-interest was chosen to maintain a good resonator efficiency and EPR concentration sensitivity.

Design of the field modulation slots is produced by the following: i) simulating the 100 kHz field modulation profile in the region-of-interest for n slots of equal depth, ii) reducing the central slots by approximately half, and iii) adjusting slots to minimize ripple along the sample [9]. After each step a simulation is run and uniformity along the axis of the sample is calculated. Using Ansys HFSS built-in optimization, this process can be automated. The slots are mirrored from the center of the resonator to reduce the parameter space. The designed slots are illustrated in Fig. 6 and are calculated to be 98% uniform over the region-of-interest. This solution is not unique.

4. Results

A comparison of calculated and measured characteristics of the new resonators and resonators of Ref. 6 is shown in Table 2.

Shown in Fig. 8 is the complex spectrum of rose bengal and ascorbyl radicals in sample containing aqueous solution of rose bengal and sodium ascorbate photo excited with green

light (532 nm) using the rectangular TE_{U02} . We report this test spectrum as the first experiment of rose bengal at 94 GHz. At X-band, under similar experimental conditions the rose bengal anion radical exhibits a three line spectrum with hyperfine splitting 0.31 mT, due to the interaction of unpaired electron with two equivalent protons [21, 22]. At W-band, the hyperfine splitting is broadened by the rotational correlation time of the rose bengal molecule and the lower resonator efficiency requires higher power resulting in potential source noise. Future studies are underway.

In the dark a single line EPR signal can be observed. This EPR signal could be attributed to the ascorbyl radical, which is always present in the aerated aqueous solution of ascorbate. Additionally, the apparent lack of the dependence of the ascorbyl radical signal intensity on sample irradiation can be explained by substantial differences in the decay kinetics of the ascorbyl and rose bengal anion radicals, resulting in different steady-state concentrations of the photoinduced radicals [22, 23].

Shown in Fig. 9A as solid and dashed is the B_1^2 for the seven-loop–six-gap LGR with and without end-sections, respectively. With the end-sections in place, the LGR field over the region-of-interest is calculated to be 67% uniform in B_1^2 (solid line), whereas the resonator without the end-sections is 30% uniform (dashed line). Additionally, for continuous-wave experiments, the applied 100 kHz field modulation is calculated to be 98% uniform over the region-of-interest, shown in Fig. 9B. Data measured is shown as circles (●) and connected by a dash-dot line.

5. Discussion

The five-loop–four-gap LGR in Ref. 6, as shown in Table 2, sets the standard. Here, we see an increase in both EPR signal saturable and unsaturable samples when using the seven-loop–six-gap LGR described in this work. This increase is due to two reasons. The first is the increase of volume using larger PTFE capillary diameter and length. The second is the uniformity, shown by the Λ_{ave} -to- Λ_{max} ratio of 0.90. In contrast, the five-loop–four-gap LGR has a Λ_{ave} -to- Λ_{max} ratio of 0.45. The low Λ_{ave} -to- Λ_{max} ratio can be attributed to the 0.65 mm sample diameter and 1 mm overall height. However, the increase of the volume lowers the overall Q_0 -value of the resonator and the larger inner diameter lowers the efficiency parameter. Overall, the seven-loop–six-loop LGR was found to be a good compromise in sample handling and degassing, resonator efficiency, and EPR signal. An example of degassing capabilities can be found in Ref. [14]

Due to the larger inner diameter and overall length of the seven-loop–six-gap LGR, the uniformity of the microwave magnetic field would be very poor if there were no end-sections correcting the microwave magnetic field profile, shown in Fig. 9A as a dashed line. The enhanced end-sections provide this adjustment shown in Fig. 9A as a solid line. The measured uniformity of both the microwave magnetic field, shown in Fig. 9A (data points as ●; dash-dot), and 100 kHz magnetic field, shown in Fig. 9B (data points as ●; dash-dot), shows very good agreement with the simulated data (solid line).

The rectangular TE_{U02} cavity has a Λ_{ave} -to- Λ_{max} ratio of 0.87, while the typical cylindrical TE_{011} cavity has a ratio of 0.6, which is consistent with a cosine distribution. The lower uniformity ratio for the TE_{U02} is due to the sample shield size needed to hold the sample. The shield creates a local roll-off region, shown in Fig. 3B. Additionally, this resonator has a lower efficiency parameter compared to the cylindrical TE_{011} cavity due to an extended region-of-interest that is larger than a wavelength. This was described in the seminal uniform field papers [1–3, 5] and is due to the decrease in stored energy over the larger volume.

The extension of the null-index direction creates the potential for higher-order modes to form in the oversized waveguide. The use of a dual iris in the rectangular TE_{U02} cavity decreases the coupling of neighboring modes that would degrade the microwave cross-section. Additionally, the use of the long-slot iris minimizes the perturbation in the cut-off region allowing the 6 mm region-of-interest in the z-direction. Ansys HFSS simulations show that a round iris significantly perturbs the mode and a uniform field mode is not possible.

Although the rectangular TE_{U02} cavity has no modulation slots, continuous-wave experiments can be performed using a low frequency (433 Hz) field modulation with little degradation to the continuous-wave EPR signal [15]. The uniformity of the field modulation was simulated to be 70% uniform over the region-of-interest.

6. Conclusion

A seven-loop–six-gap LGR with enhanced end-sections and optimized 100 kHz field modulation slots has been designed for better quantitative experiments of samples that require degassing or larger sample volumes. This resonator is a good compromise of efficiency and Q_0 -value for an increase in EPR signal intensity. The uniformity has been calculated and measured to be 90% for magnetic field and 98% for 100 kHz field modulation.

The rectangular TE_{U02} resonator provides a plane of uniform microwave magnetic field incident on a $0.1 \times 2 \times 6 \text{ mm}^3$ sample. The use of evanescent slots to allow 90% light to radiate the sample has been successfully implemented and tested with a rose bengal sample in ascorbate. A dual iris coupling system has been designed to significantly reduce coupling to neighboring modes that degrade the uniform fields providing an improvement in EPR concentration sensitivity.

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- Two new W-band uniform field cavities are introduced
- A seven-loop—six-gap loop-gap resonator with enhanced end-sections
- A rectangular TE_{U02} resonator with light-access slots
- Design methodology, fabrication, and EPR experimental results are discussed

Figure 1. Fabricated geometries of the A) seven-loop-six-gap LGR and B) assembled TE_{U02} cavity showing the 0.1×2 mm flat cell capillary and light access slot.

Figure 2.

Illustrations of three methods for creating uniform fields in cavities. Region-of-interest at cut-off is matched to A) an over-sized section, B) a reentrant end-section with capacitive posts (black circles), C) a dielectric section of length $\lambda/4$ placed at a shorted end. Magnetic fields vectors are illustrated with dotted lines.

Figure 3.

A) Half-structure CAD drawing illustrating the rectangular TE_{002} geometry with over-sized end-sections, sample access port, light access, and sample end-section shield, B) Magnetic field magnitude profile showing the 6 mm region-of-interest and C) Magnetic field magnitude profile showing the dual iris (thickness 0.1 mm) and sample placement (dashed). Dotted line represents the cavity wall illustrating the light access slots beyond cut-off. Red is large magnetic field and dark blue zero magnetic field.

Figure 4.

Simulations of the real part of the peak value of $(E_x + E_y)$ over the x - y cross-section of the resonator illustrating A) typical TE_{102} mode with small zero-index length, B) TE_{102} mode with large zero-index length, C) TE_{111} mode with large zero-index length, D) TE_{112} mode with large zero-index length, E) TE_{121} mode with large zero-index length. Plus (+) and Minus (-) show field maxima and minima, square (■) is zero field.

Figure 5. Simulations of the real part of the peak value of $(E_x + E_y)$ over the x — y cross-section of the resonator illustrating A) large zero-index TE_{102} mode with single capacitive slot iris, B) large zero-index TE_{102} mode with dual capacitive slot iris.

Figure 6.

A) Half-structure CAD drawing illustrating the LGR resonator assembly including the gas exchange port, resonator iris, Rexolite end-sections, and shorted end-sections. B) Illustration of the seven-loop–six-gap LGR geometry. The sample loop is in the center with a diameter of 0.825 mm, outside loops at 0.95 mm, and a gap of 0.29 mm. Waveguide and iris are shown as a dotted line. C) Side view of the LGR with a 3 mm active region. Rexolite end-sections are highlighted and modulation slots optimized for uniform 100 kHz incident on the sample are illustrated.

Figure 7.

A) Magnetic field profile for the seven-loop-six-gap LGR illustrating sample placement (dashed) and B) cross-section of magnetic field. Red is large magnetic field and blue is no magnetic field.

Figure 8.

Spectra of 0.1mM Rose bengal in the presence of 0.5 mM ascorbate photo-excited using the rectangular TE_{U02} shown with and without 532 nm wavelength excitation. Resulting residual spectrum is in red. Experiments were performed at room temperature with 10 mT sweep width over 190 seconds, 0.4 mT field modulation at 433 Hz, and a microwave power of 100 μ W.

Figure 9.

A) Normalized B_1^2 showing simulated data for the uniform field LGR (solid) and same LGR geometry without enhanced end-sections (dash). Measured data points shown (●). B) Normalized 100 kHz field modulation B_m showing simulated LGR with optimized field modulation slots (solid). Measured data points shown (●). Dash-dot lines are used to connect data points and vertical lines define the region-of-interest. Experiments were performed at 10 μ W microwave power and over-modulated field modulation at 0.1 mT at 100 kHz. Normalized B_1^2 is measured as peak-to-peak EPR signal and B_m is measured in change of linewidth.

Table 1

Resonator characteristics calculated for nearest-neighbor modes of the rectangular TE₁₀₂ with sample.

Mode	Freq. (GHz)	Q ₀ -Value
TE ₁₁₁	70.12	19
TE ₁₀₂	94.32	510
TE ₁₁₂	109.51	236
TE ₁₂₁	124.49	66

Resonator characteristics calculated and measured of new geometries compared to resonators of Ref. 6

Table 2

Geometry	Freq. Meas. [Ghz.]	Q ₀ -value Meas.	Signal Unsaturable (Calc.) [V]	Signal Saturable (Calc.) [V]	Λ_{max} Calc. [mT/W ^{1/2}]	Λ_{ave} Calc. [mT/W ^{1/2}]	Sample [mm]
Cylindrical TE ₀₁₁ Cavity	93.35	1630	8.41	1.18	0.3	0.18	0.151D. × 3
Five-loop–four-gap LGR	94.31	350	10.00	0.97	1.12	0.50	0.151D. × 1
Seven-loop–six-gap LGR	94.17	150	19.16	6.36	0.51	0.46	0.251D. × 3
Rectangular TE ₀₀₂ Cavity	93.21	510	6.82	4.56	0.15	0.13	0.1 × 2 × 6