Research Highlight #152

A new multi-arm W-band (94 GHz) electron paramagnetic resonance (EPR) spectrometer

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Introduction: Technical advances embodied in a new multi-arm W-band (94 GHz) EPR spectrometer are described by Strangeway et al.[1] The instrument can be configured for a variety of continuous wave (CW) experiments including field- and frequency-swept EPR, saturation-recovery EPR, and pulsed electron-electron double resonance (ELDOR). It utilizes a loop-gap resonator (LGR). The superconducting magnet has a horizontal magnetic field and provides access through a side port. The bridge is mounted on a table that rests on a granite slab. The table is supported on air bearings and is readily moved in a horizontal plane to introduce the sample through the side port of the magnet. The microwave power chain contains four microwave sources: microwave synthesizers in the 2 GHz range, two low-noise Gunn diode oscillators (33 and 51 GHz), and a yttrium iron garnet (YIG) oscillator (6.5-9.5 GHz). The synthesizers are time-locked and provide digital accuracy of frequency differences between arms; the in-house developed Gunn diode oscillator pair dominates the microwave phase noise, which is exceptionally low, and the YIG oscillator provides frequency agility. There are two sample-irradiation arms and a receiver arm. Path-length equalization occurs at the intermediate frequency of 59 GHz. A directional coupler is favored for separation of incident and reflected power between the bridge and the LGR. Oversize waveguide with hyperbolic-cosine tapers couples the bridge to the LGR, which results in reduced microwave power loss. Emphasis is placed on the utility of the spectrometer for free-radical studies in the room-temperature range, including spin labels.

Results: CW experiments are shown in Fig. 1 for data collected from a 1 μM TEMPO sample at 25°C in a 0.15 mm inner and 0.25 mm outer diameter quartz capillary. Comparative data were taken from work by Sidabras et al,[2] shown in Fig. 1A. In Sidabras et al,[3] a cylindrical TE011 was used and the total sample volume was 30 nL. Spectrometer parameters were 3 G field modulation at 100 kHz, 0.5 mW rf power, 50 ms time constant, and 128 averages at 30 sec per scan for a total of 45 minutes. Each scan has 1024 points with a sweep of 5 mT. Spectra in Figs. 1B and C were collected on the current W-band system using a five-loop–four-gap LGR and the same capillary and sample volume. Spectrometer parameters were 3 Gp field modulation at 433 Hz, 0.5 mW rf power, 50 ms time constant. Each scan was 1024 points over 4.75 mT. The five-loop–four-gap LGR has no modulation slots; therefore, a low audio frequency (433 Hz) was used for field modulation. A single scan was taken over 1 min and 30 sec, and a five-point moving average filter was used to remove high frequency noise, shown in Fig. 1B. Finally, a spectrum was collected using 128 averages at 1 min and 30 sec per scan for a total scan time of 3 hr and 45 min, shown in Fig. 1C. A five-point moving average filter was used to remove high-frequency noise. The noise profile of the spectra with 128 averages is shown in the inset.

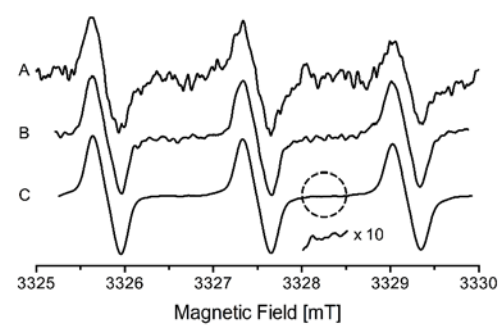

Fig. 1. Sensitivity data at 1 μM TEMPO. From Sidabras et al:[4] A) 128 scans, 45 min total time. New benchmark spectra: B) single scan, 1 min 30 sec, five-point moving average filter. C) 128 scans, 3 h 45 min, five-point moving average filter, with 10x noise inset.

Implications: Long-term stability of the bridge, allowing signal averaging of data for up to 24 hours, as well as substantial improvement in concentration sensitivity, are great accomplishments. These improvements, coupled with the very small volumes required, will allow for the analysis of single eye lenses from patients, limited numbers of stem cells or other cells and tissues from transgenic animals or humans, as well as low concentrations or limited expression of proteins to advance the knowledge of biomedical processes and disease diagnosis. Highlight #153 showcases one important application.

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[1] Strangeway RA, Camenisch TG, Sidabras JW, Mett RR, Anderson JR, Ratke JJ, Hyde JS. (in submission) W-band EPR Spectrometer with a Two-Arm High-Bandwidth Multistage Frequency Conversion Bridge. J Magn Reson.
[2] Sidabras JW, Mett RR, Froncisz W, Camenisch TG, Anderson JR, Hyde JS. (2007) Multipurpose EPR loop-gap resonator and cylindrical TE011 cavity for aqueous samples at 94 GHz. Rev Sci Instrum. 78(3):034701.
[3] Ibid.
[4] Ibid.