Active Spectrum's Benchtop Electron Spin Resonance Spectrometers

ACTIVE SPECTRUM

Extended Range Benchtop ESR

- 4000G sweep range (air cooled)
- 6500 G sweep range (water cooled)
- Compact benchtop design
- Competitive pricing
- Variable temperature accessories available

Technical Data

Frequency	9.6 to 9.8 GHz
Resonator	Cylindrical Dielectric
Sample Tube Diameter	Up to 5.8 mm
Sweep Range	Up to 6500 Gauss (water cooled); Up to 4000 Gauss (air cooled)
Supply Voltage	120/240 VAC
Data Interfaces	Ethernet, USB, and 802.11 WiFi
Screen	21 cm Touch Panel display with Windows 7 Embedded DVI/HDMI/VGA
Dimensions	30.5 x 35.5 x56 cm^3
Mass	54 kg

Micro-ESR Spectrometer

- **Easily Transported Anywhere**
- **Plug In, Power On, Acquire Spectra**
- **Versatile: Applications in Research, Education, and Industry**
- **Variable temperature accessories available**

Technical Data

Frequency	9.6 to 9.8 GHz
Resonator	Cylindrical Dielectric
Sample Tube Diameter	Up to 5.8 mm
Sweep Range	Over 330 Gauss Centered at g=2
Supply Voltage	15 VDC (120/240 V Wall Adapter Included)
Data Interfaces	Ethernet and USB
Screen	21 cm Touch Panel display with Windows 7 Embedded DVI/HDMI/VGA
Dimensions	30.5 x 30.5 x 30.5 cm^3
Mass	10 kg

Online Benchtop ESR

- World's first industrial process monitoring instrument
- Provides real-time, embedded, non-destructive measurements of free radicals and some transition metals
- Designed to work under harsh environmental conditions
- Easily transported to any site
- Hazardous location rated enclosures available(Class 1, Div. 1, and ATEX)

Technical Data

Frequency	9.6 to 9.8 GHz
Resonator	Cylindrical Dielectric
Sample Tube Diameter (Flow Mode)	Up to 6 mm OD
Sample Tube Diameter (Tube Mode)	Up to 3 mm OD
Sweep Range	Over 300 Gauss centered at g=2
Supply Voltage	15 VDC (120/240 V wall adapter Included)
Data Interfaces	Ethernet and USB
Screen	21 cm Touch Panel display with Windows 7 Embedded DVI/HDMI/VGA
Dimensions	30.5 x 15 x 23 cm^3
Mass	10 kg
Operating Temperature	-30 to 85 degrees C
Maximum Pressure	100 psi
Maximum Fluid Inlet Temperature	160 degrees C

Signal to Noise

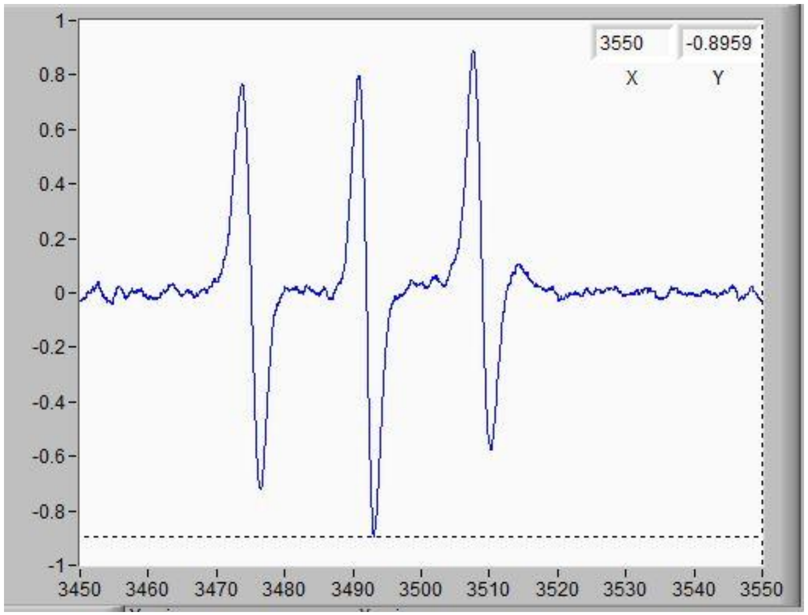
NOTE: Signal and noise measurements are made using the same conditions. The signal is calculated as the average of the amplitude of the three TEMPOL peak heights divided by two times the RMS noise in the same spectrum.

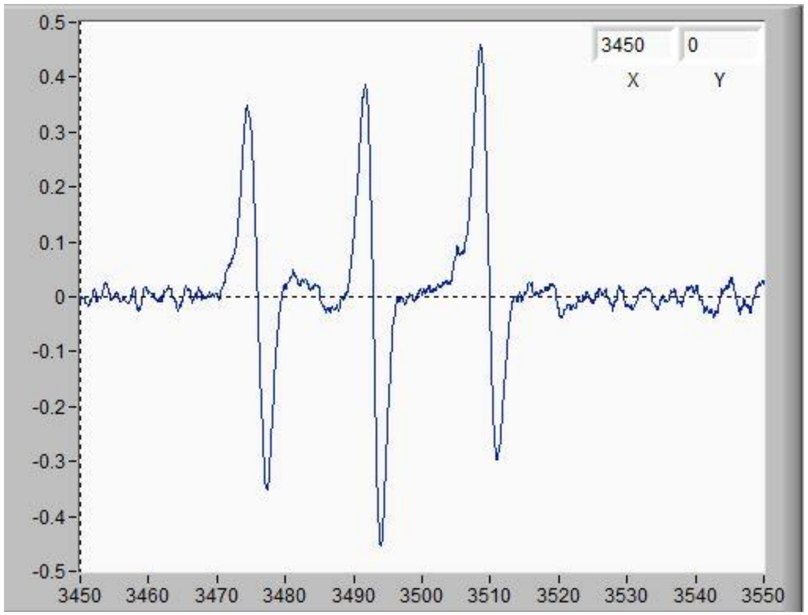
Signal to Noise = (peak amplitude) / (2*RMS noise)

50 μM TEMPOL in Water

1 scan, 4k data points, 15 mW
S:N > 200:1
Extended Range Benchtop

1 µM TEMPOL in Water





Flat Cell
Signal to Noise 70:1

Capillary, 1mm ID
Signal to Noise 30:1

64 scans, 4k points, 15 mW, 100 G sweep width

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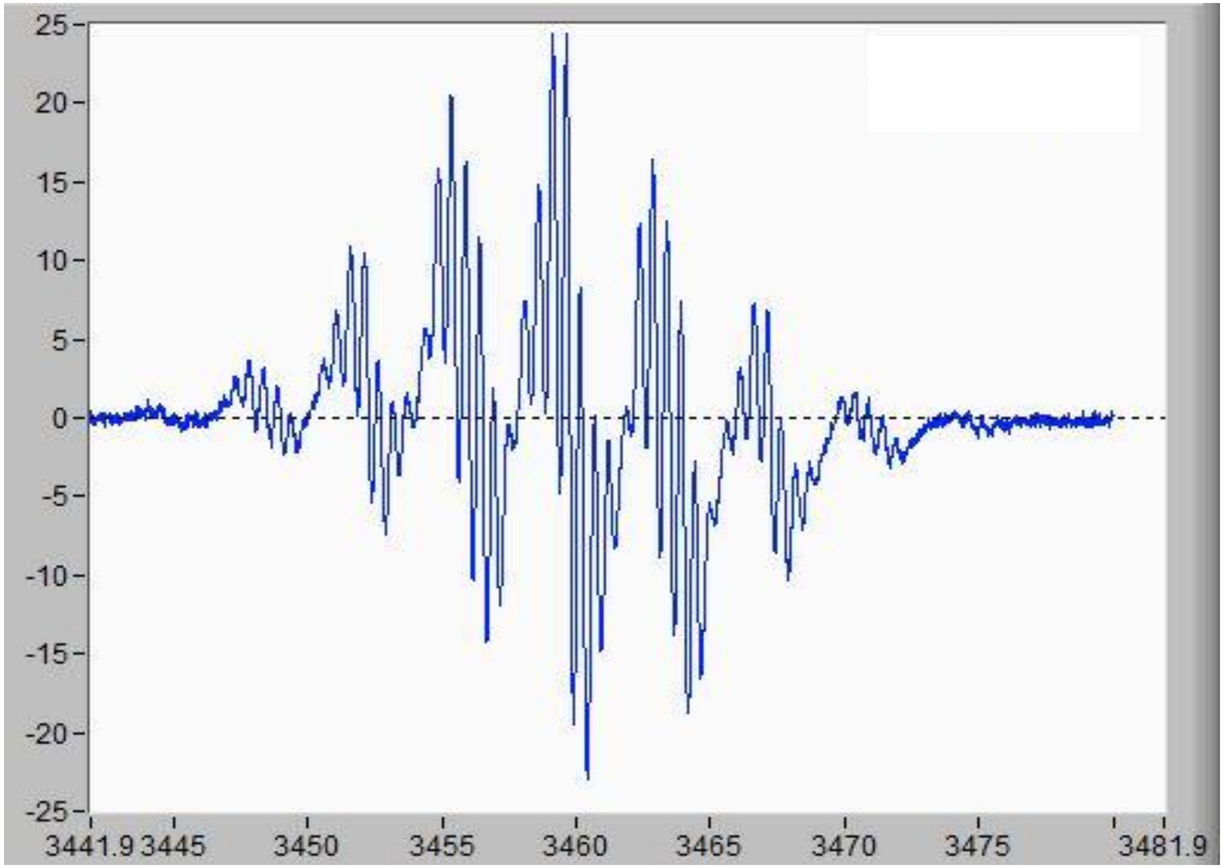
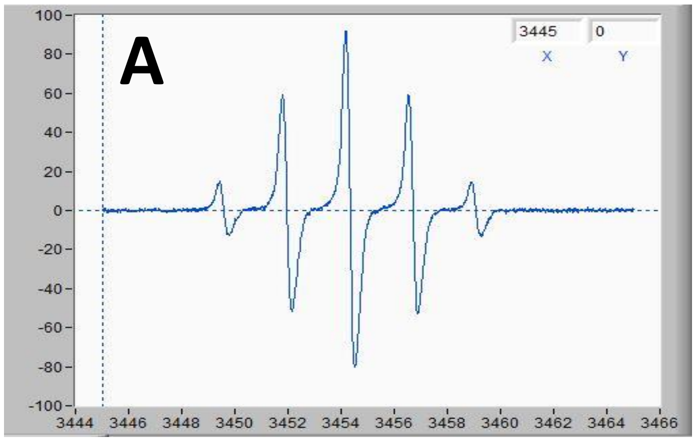
Resolution

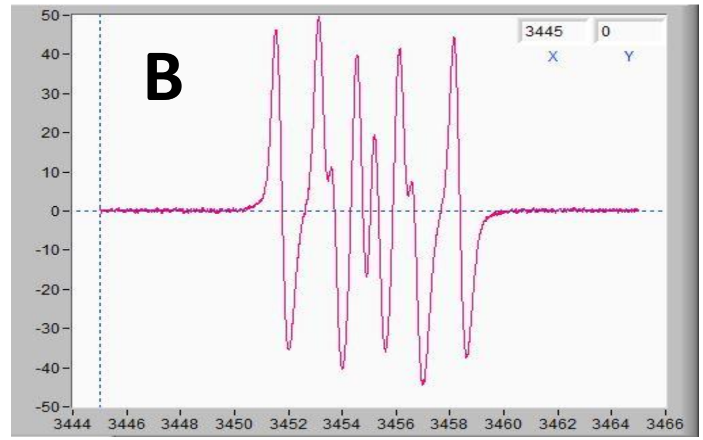
Perylene Radical Cation



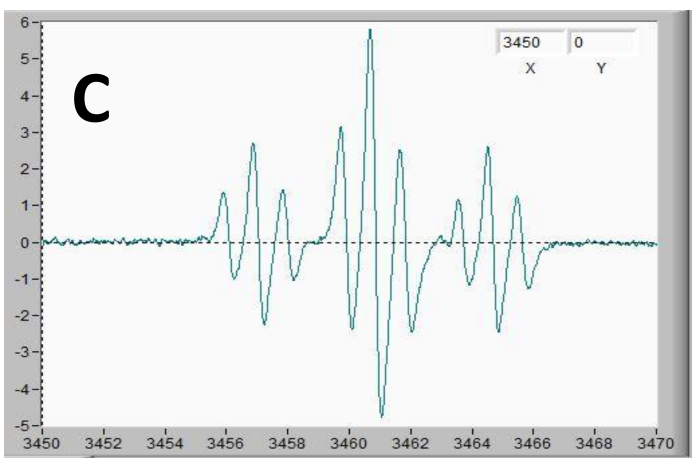
Semiquinone Radical Anions



Benzosemiquinone



2-butylbenzosemiquinone



catechol

	a_{H1}	a_{H2}	a_{H3}
A	2.28		
B	2.01	1.67	1.38
C	3.38	0.74	

Some Applications

- Spin Trapping
- Kinetics
- Coordination Chemistry
- Antioxidant Radicals in Lubricants
- Dosimetry
- Crystal Defects
- Education

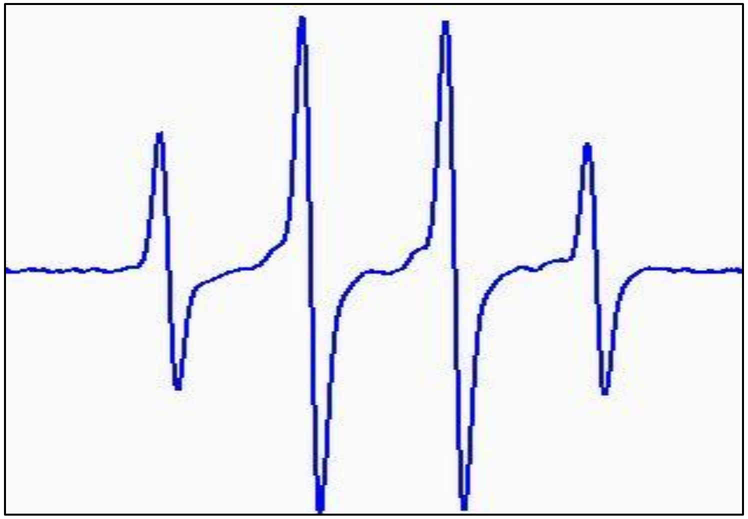
DMPO as a Spin Trap For $OH^{\cdot}$

- Electron spin resonance spectroscopy is a well established technique for measuring the signal from hydroxyl radicals that are spin trapped by DMPO. These hydroxyl radicals are generated via the Fenton Reaction:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{\cdot} + OH^{-}$$

$$DMPO + OH^{\cdot} \rightarrow DMPO\text{-}OH^{\cdot}$$

- Micro-ESR provides a portable, cost-effective, and sensitive spectrometer that can measure DMPO-OH radicals from this reaction.



DMPO-OH$^{\cdot}$ Spin Adduct

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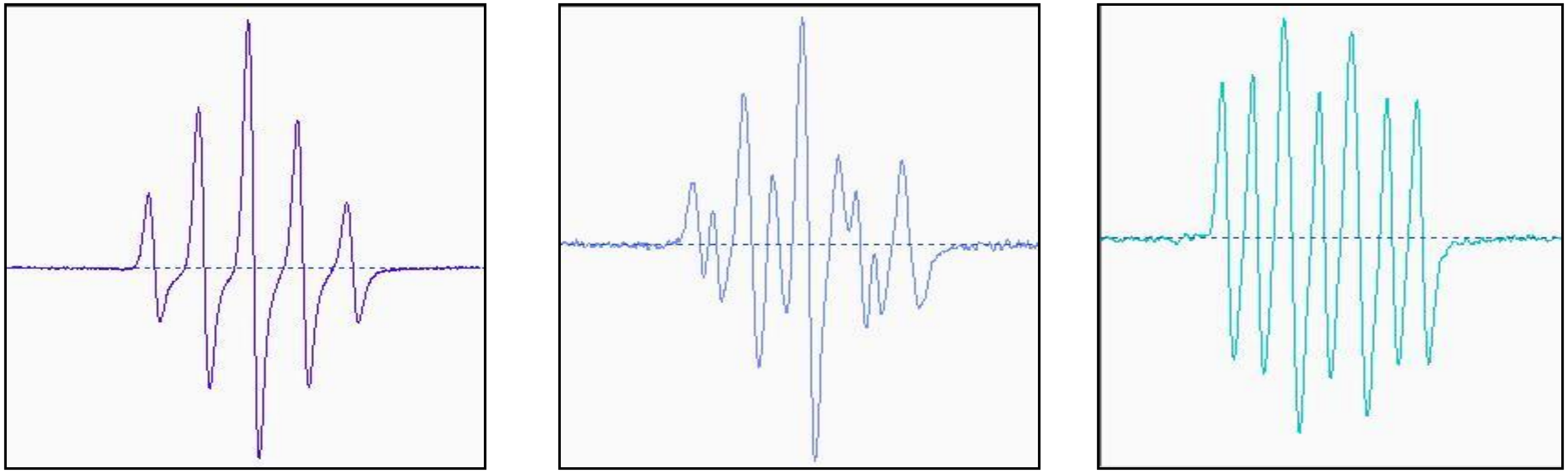
PTIO as a Spin Trap for Nitric Oxide



1. ESR signal of α-Phenyltetramethylnitronly nitroxide (PTIO)
   - PAPA NONOate (NO source) is added to the tube
2. The ESR signal changing from the nitronyl nitroxide to the imino nitroxide
3. The imino nitroxide ESR signal

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Determine Reaction Mechanisms by Observing Spin Adducts

- Using PBN (t-butyl-α-phenyl nitrone) as a spin trap, we follow a Fenton Reaction in DMSO.
- The Fenton Reaction generates hydroxyl radicals, which react with the PBN, but also the DMSO
- The PBN-OH• spin adduct is too short lived to observe directly, but methoxyl (OCH_3•) radicals are also produced
- The PBN-OCH_3• spin adduct is long lived, and easily observed by ESR
- As the reaction proceeds, the major spin adduct changes from methoxyl to methyl
- MNP, 2-methyl-2-nitrosopropane, a spin trap itself, is formed during the reaction
- MNP forms a third spin adduct with methyl radicals

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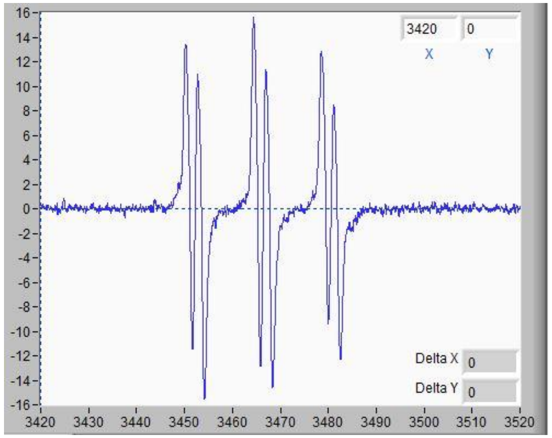
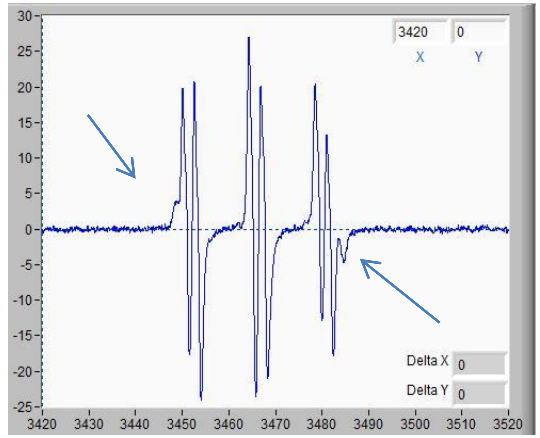
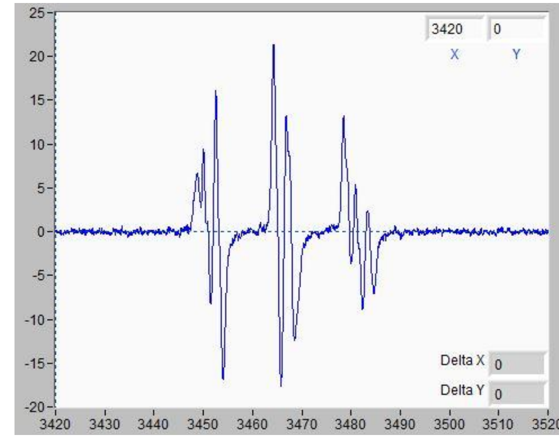


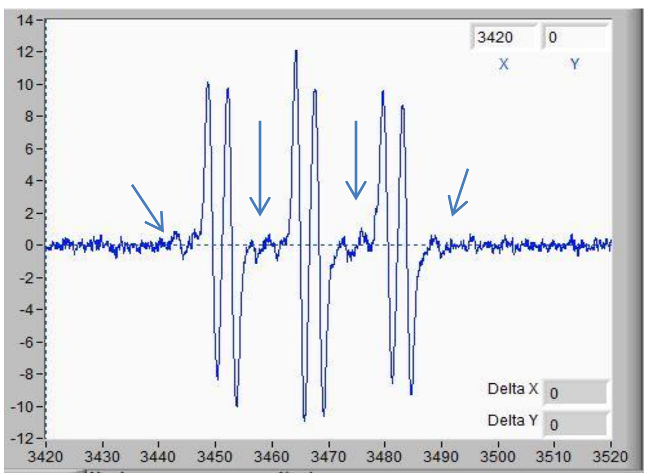
PBN-OCH_3 Spin Adduct



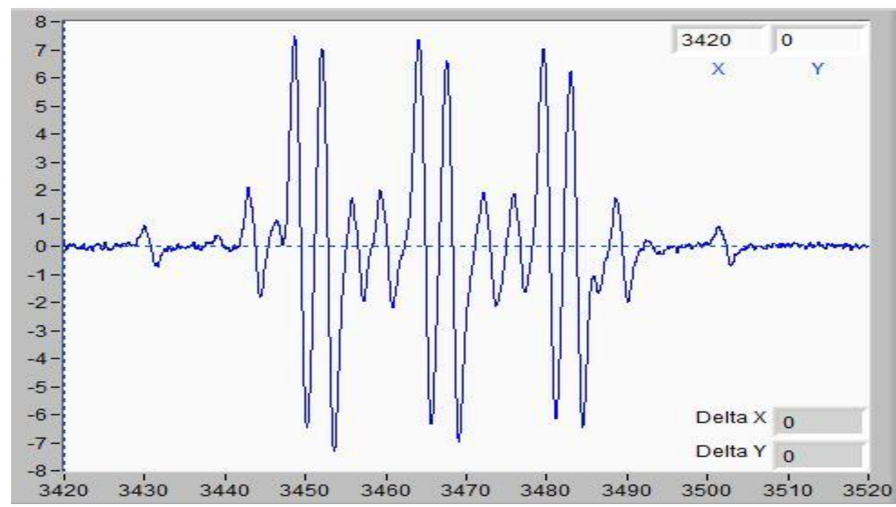
A second spin adduct starts to form



Both PBN-OCH_3 and PBN-CH_3 Spin Adducts are present



The PBN-CH_3 spin adduct remains, and another spin adduct is forming



A third spin adduct, MNP-CH_3 has formed, and is superimposed on the PBN-CH_3 spin adduct

Identifying Spin Adducts

- The hyperfine coupling constants help to identify the trapped radicals, and give information about the electron spin density
  - The hyperfine coupling constants are smaller both for the nitrogen and the proton in the PBN-OCH_3 spin adduct than the PBN-CH_3 spin adduct because the oxygen in the methoxy pulls spin density away from the nitrogen
- More direct information can be obtained about the radical from the nitroso spin adduct because the radical adds directly to the nitrogen atom
  - We see coupling from the methyl protons only in the MNP-CH_3 spin adduct
- We know that it is the methyl, not the methoxyl, radical that had added to the MNP because MNP does not form oxygen centered radicals

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Kinetics and Spin Traps

- Stable radicals such as TEMPOL can be used to monitor reaction rates
- TEMPOL has an intense ESR signal, and can react with other radicals to form ESR silent products
- The rate at which the TEMPOL signal disappears can be used to determine reaction rates and mechanism
- Active Spectrum's user friendly acquisition software allows for quick and easy set up of kinetics experiments
- Easy to use processing and analysis software allows for quick analysis of data

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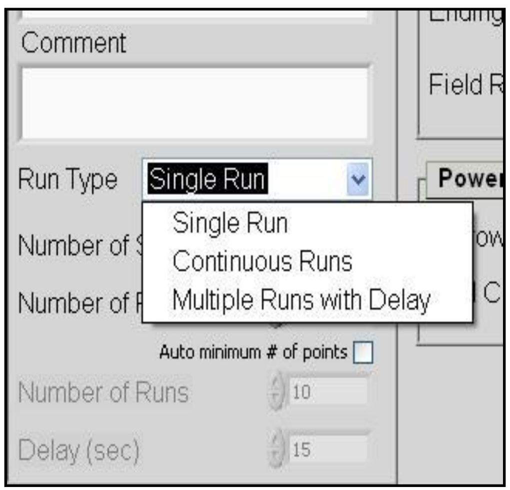
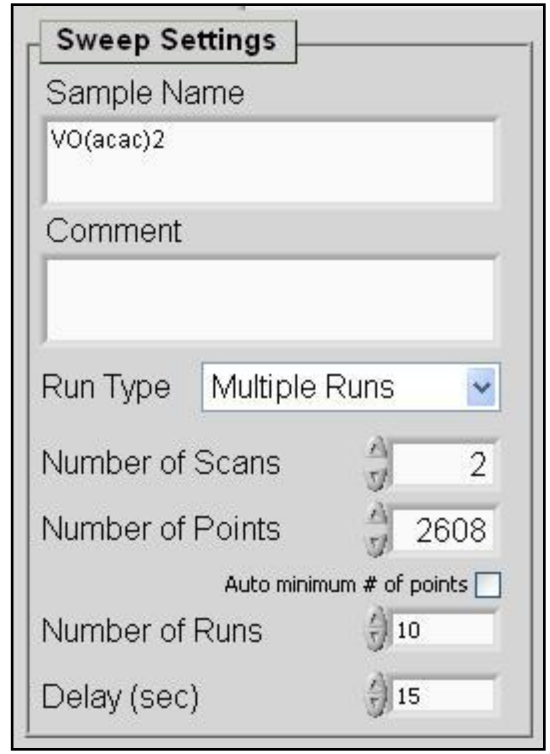
Data Acquisition

1. **Insert Sample into Spectrometer**

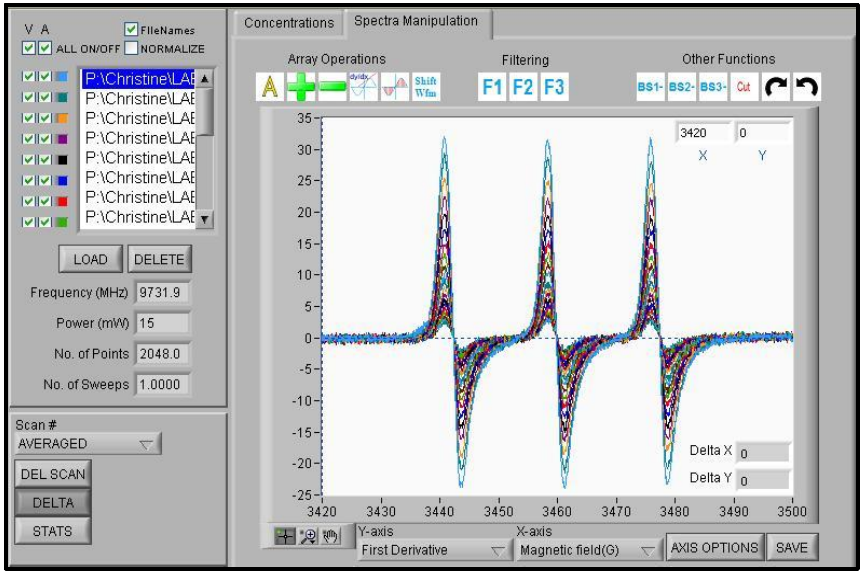


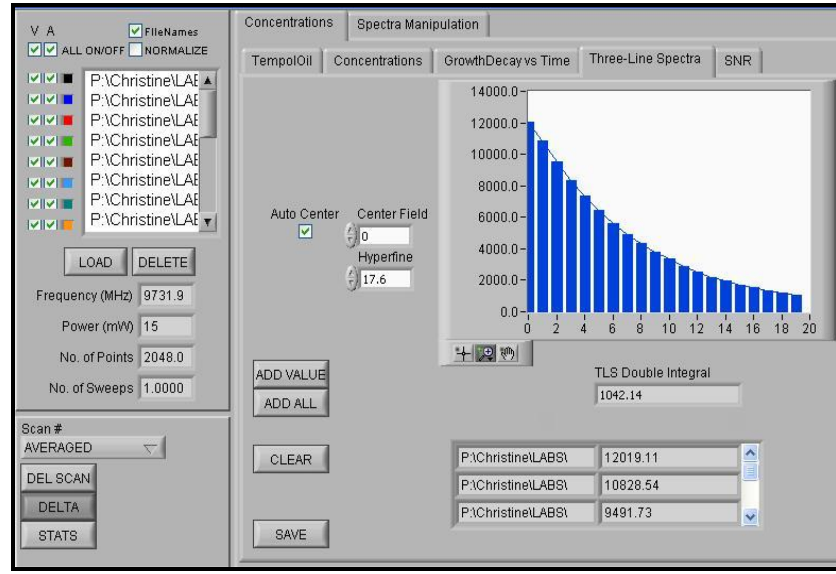
2. **Choose Multiple Runs with Delay.**



3. **Set up the Experiment**
   a) **Enter the number of scans**
   b) **Enter the number of points per scan**
   c) **Enter number of runs**
   d) **Enter the delay time between runs**
   e) **Start acquisition**

Data Processing



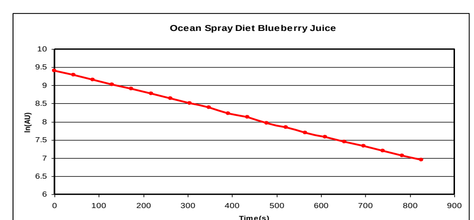


Load as many or few files as needed into the Micro-ESR processing software

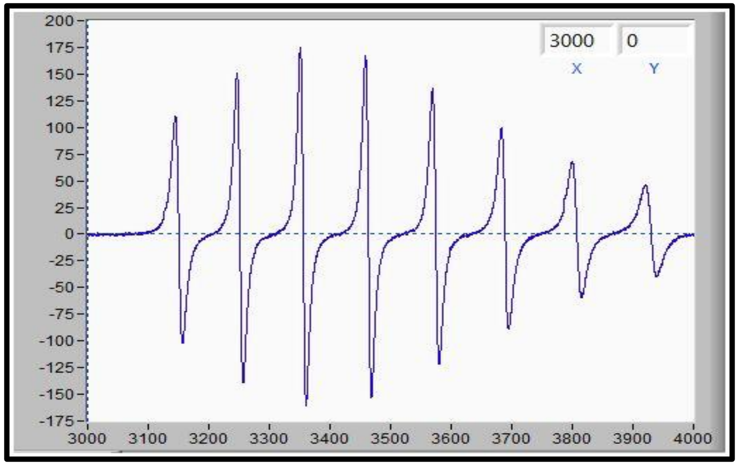
Display a single spectrum or overlay as many spectra as needed.

Processing software automatically calculates the double integral of all selected spectra, and displays the values in a bar graph.

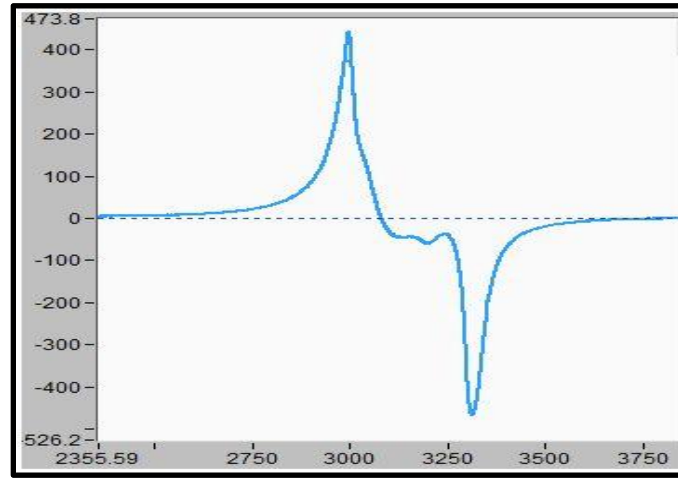
Data is saved is csv format, so can easily be read into other processing programs if desired.



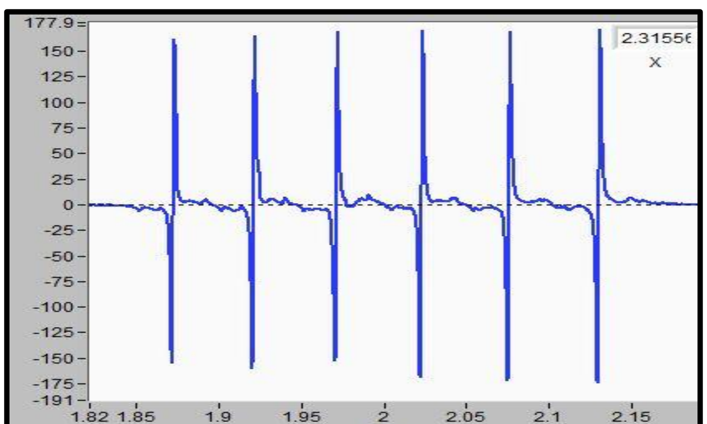
Transition Metal Compounds (Extended Range)



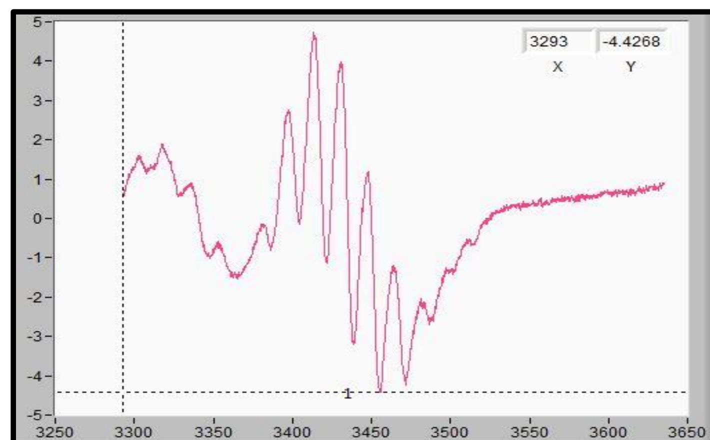
$VO(acac)_2$



$CuSO_4$

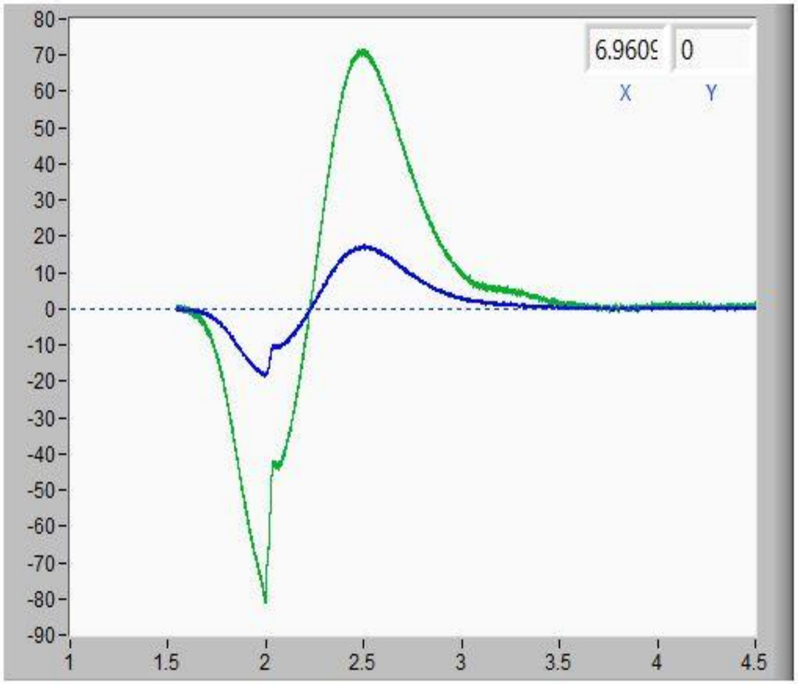


MnO

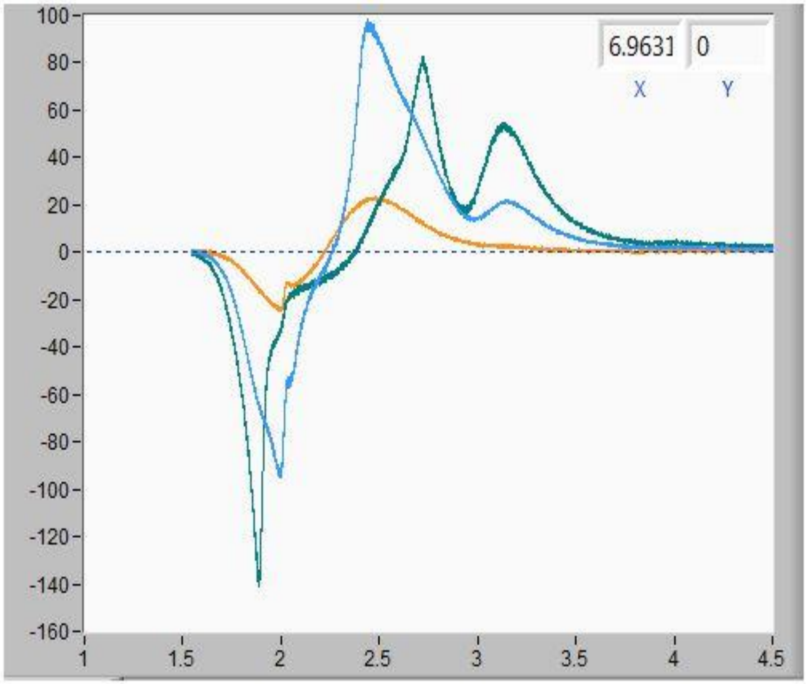


CuTPP

Ferrofluids
(Extended Range)

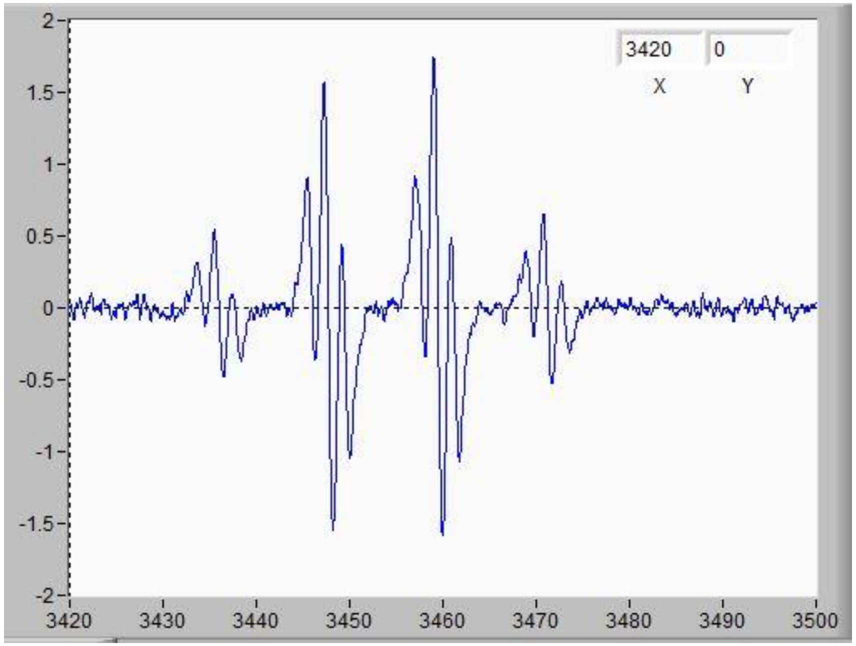


0.1 and 0.5 % Ferrofluid in Toluene



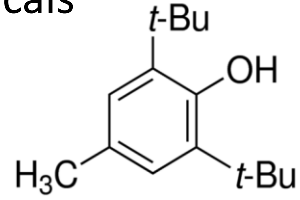
1, 5, and 10% Ferrofluid in Toluene

Antioxidant Radicals in Lubricants



- ESR is a very effective way to monitor and identify antioxidants in lubricants
- Peroxy and other radicals formed as thermal degradation products react with antioxidants, often forming stable radicals which are visible by ESR
- Elucidate reaction mechanisms through radical adducts

Butylated Hydroxy Toluene (BHT)
Loses the OH proton to free radicals



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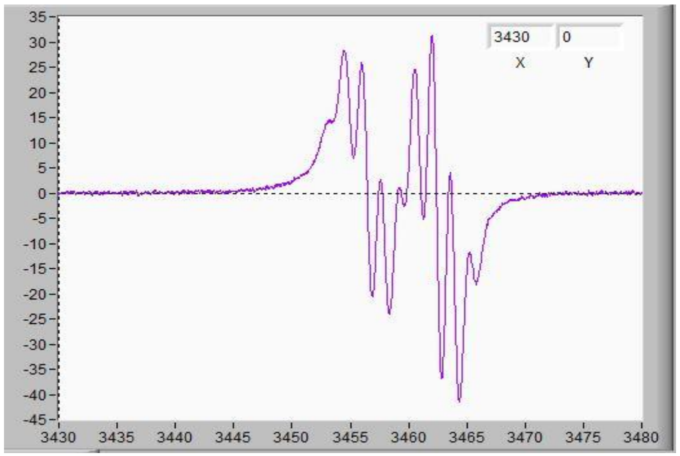
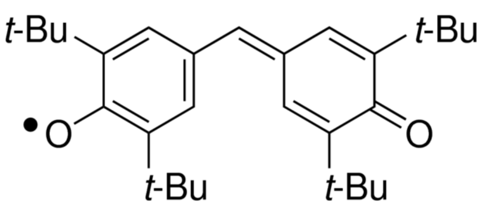
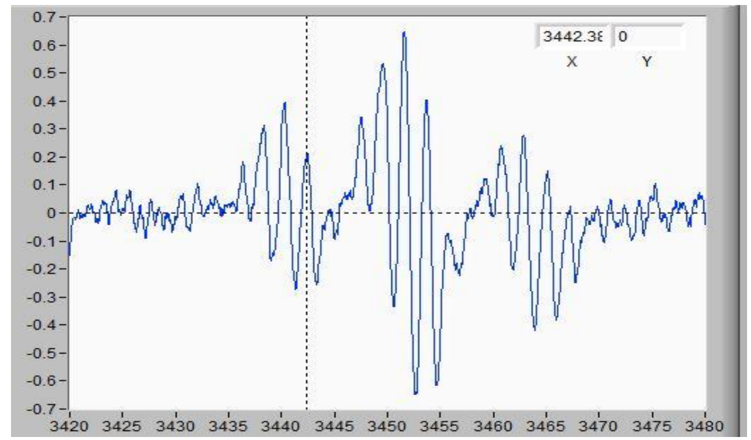
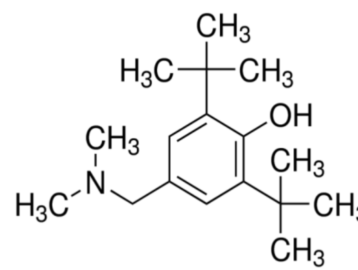




Galvinoxyl radical

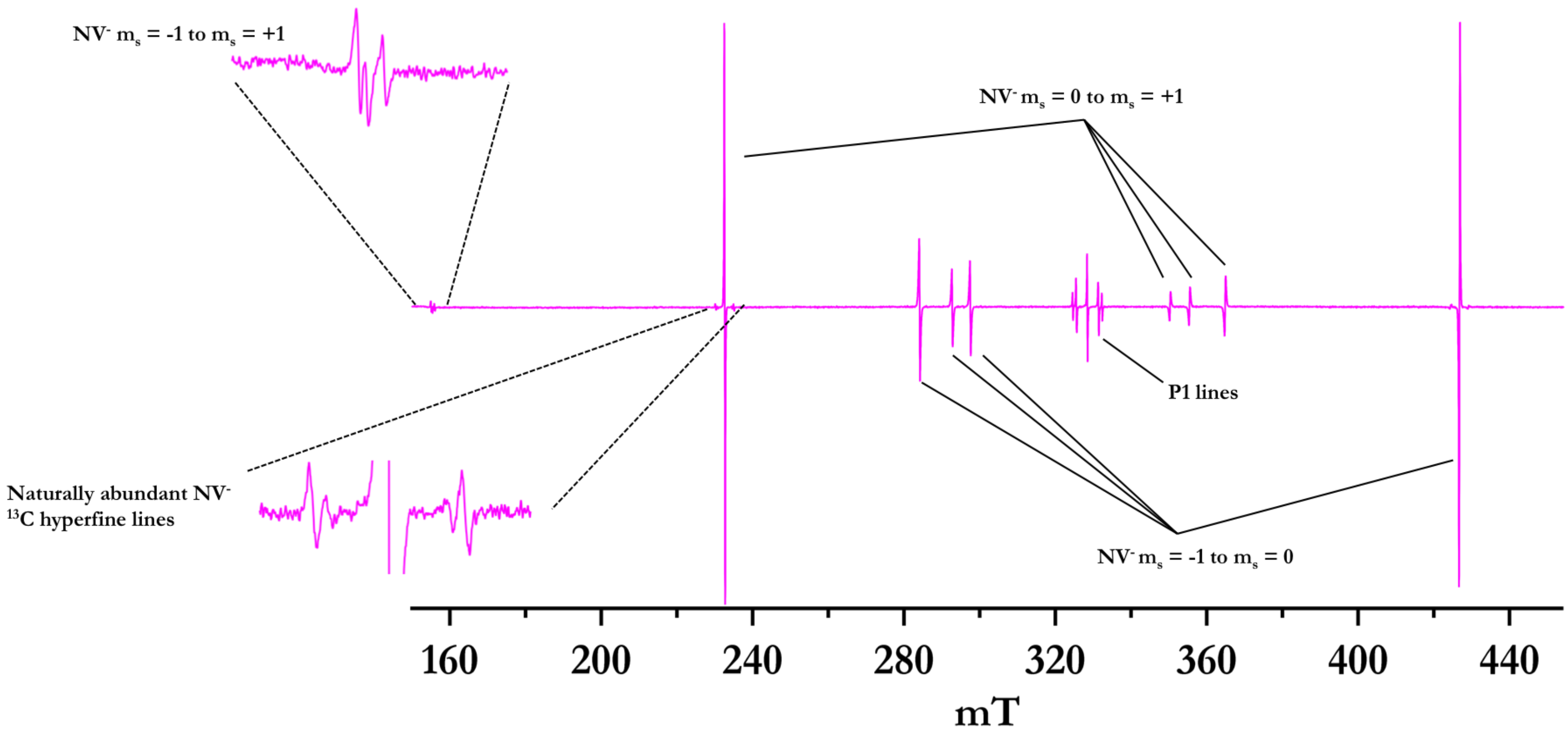




4,4-methylenebis (2,6-Di-*tert*-butylphenol)

(Loses the OH proton to free radicals)

Optically Polarized Nitrogen Vacancies in Diamond

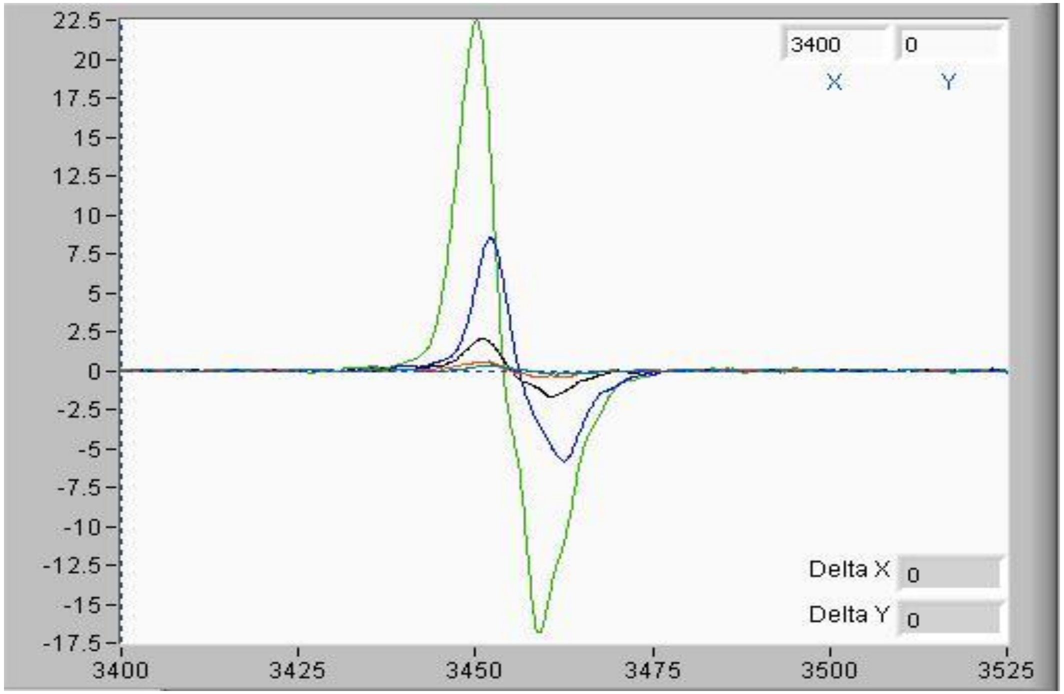


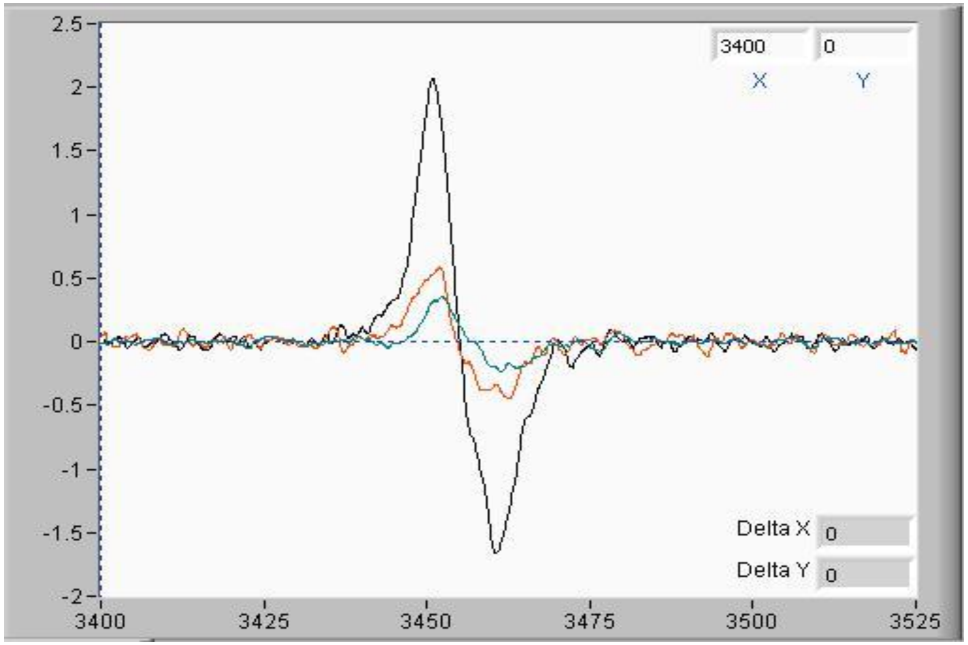
Special mirror allows laser light to be shown in to the resonator cavity to induce optical polarization.

Data courtesy of Eric Scott, Jeff Reimer's Lab, UC Berkeley. Extended Range Benchtop.

Dosimetry

- Electron Spin Resonance can be used to measure radiation exposure
- Signal amplitude of irradiated sample is proportional to the dose (sample specific, and true only for good dosimetry samples)





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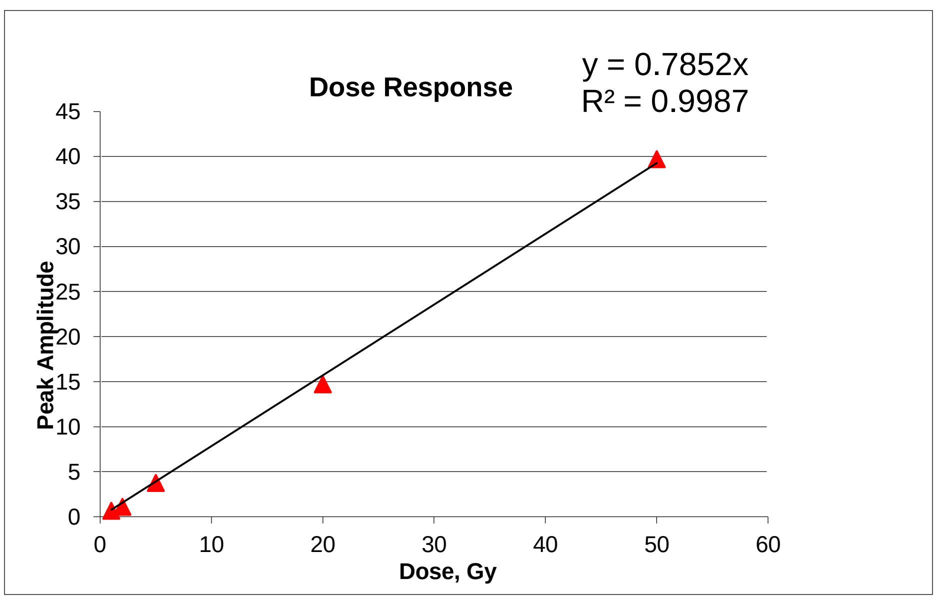
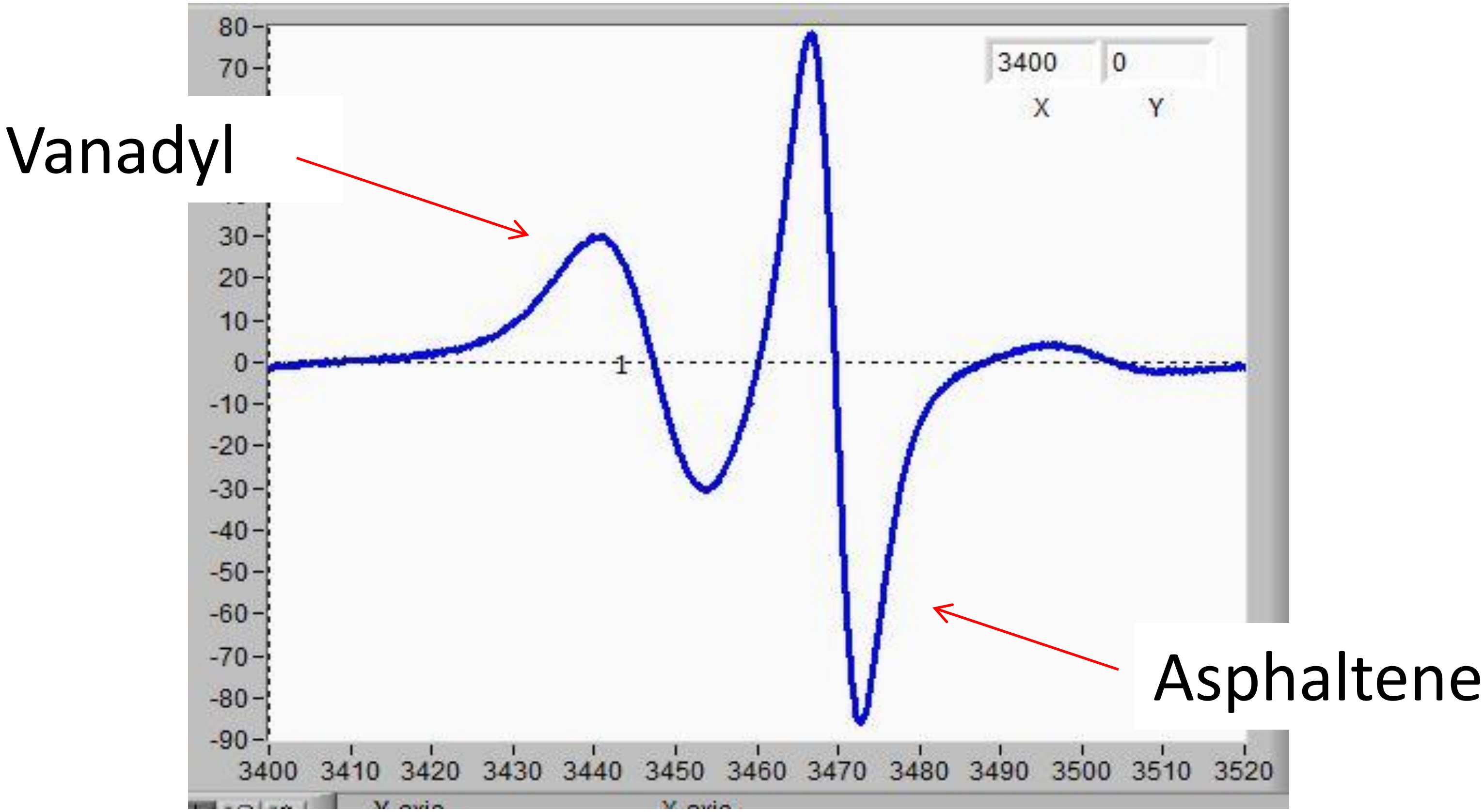
Spectra of sample irradiated with 1,2, and 5 Gy from a ^{60}Co source



Linear dose response of sample for radiation doses from 1 to 50 Gy

Crude Oil Analysis



Asphaltenes contain radicals, and vanadyl porphyrins are commonly found in crude oil

Education

- Active Spectrum's Micro-ESR is an ideal undergraduate teaching tool
- It can easily be moved to labs, classrooms, or shared
- Easy to use, so students can start to acquire data quickly
- Can be purchased with an Education Package

Micro-ESR Education Package

- **Micro-ESR Spectrometer**
- **Micro-ESR Experiments Manual with Seven Labs**
- **Micro-ESR Instructor's Guide**
- **Lab Accessory Kit**
- **Micro-ESR User's Manual**
- **Micro-ESR Analysis and Processing Software with Manual**

http://www.activespectrum.com/Micro-ESR_Education_Pkg2B_Summary.pdf

ACTIVE SPECTRUM

Contact

- These are only a small number of ESR applications
- For more information:

  http://www.activespectrum.com/about-us.shtml

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