Investigating the stability of the TEMPO radical in the presence of phenol molecules

Ibukunolu Shofolu, Tomas Orlando

National High Magnetic Field Laboratory, Florida State University

Background

Hyperpolarization is a method used to transfer energy from one molecule (the hyperpolarization agent) to another molecule (target molecule), This energy transfer sets the target molecules in a hyperpolarized state that is easier to detect with conventional NMR instruments.

Molecules with -OH groups are usually quite reactive and they can easily destroy the hyperpolarization agents(organic radical). Therefore, it is important to find a target molecule which carries an -OH group that doesn't react with the hyperpolarization agent. The goal of this experiment is to characterize organic radicals at high magnetic fields as well as obtain EPR data on DNP samples to find out the optimal formulation of these systems (solvent, concentrations, type of target molecule) that allows for these experiments to be performed.

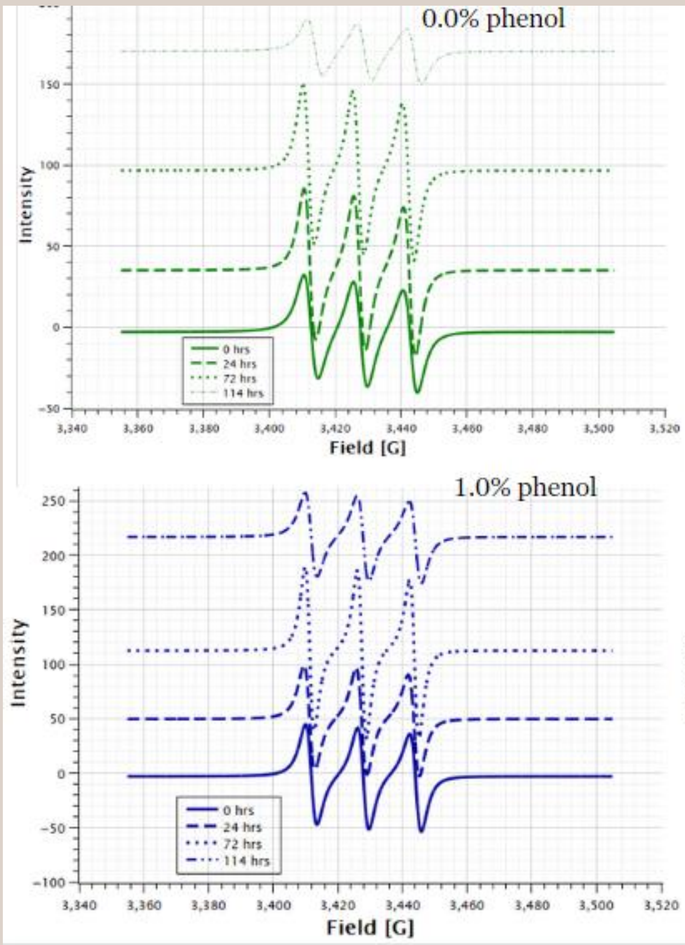
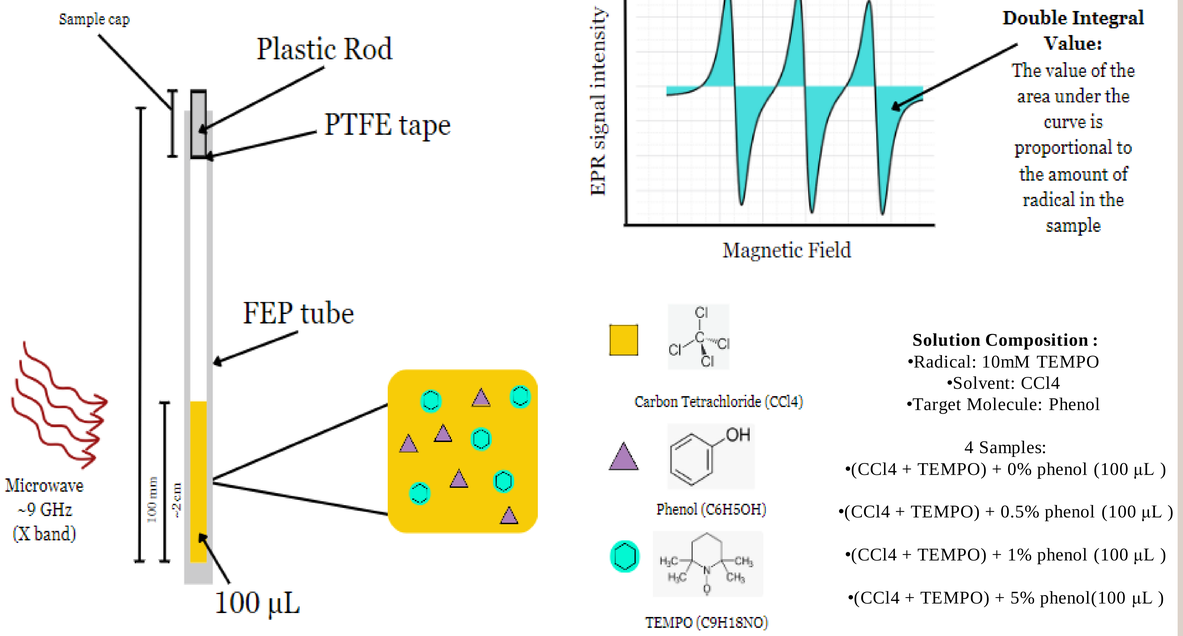


Figure 2. CW-EPR spectra of the investigated samples at 0, 24,72, and 114 hours



TFE= Teflon, FEP= flourinated ethylene propylene (not MW absorbing)

Figure 1. Sample preparation

Methods

The stability of TEMPO in the presence of phenol was tested with a systematic procedure. Continuous wave electron paramagnetic resonance (CW EPR) was performed at six intervals (0, 24, 72, 77, 90, & 114 hours)over a span over four days to monitor the amount of radical present in the solution.

Experimental Parameters:

- Mod. Amp: 0.500 G
- Mod. Frequency: 100.00 kHz
- Receiver Gain: 20 dB
- Number of Scans: 4
- Center Field: 3430.00 ± 75 G
- Sweep Time: 60.00 s

Results

- The CW EPR machine produced a spectrum for each sample analyzed at every time interval.
- The double integral value of each spectra was taken to obtain sample intensity.
- Analysis of the data showed that the samples were stable for the first 48 hours, however as the time increased, the stability of the radical was increasingly diminished.
- The graphs in Figure 2 show the increasing intensity readings of each sample as time progresses. This indicates decreasing stability as sample stability typically refers to the ability of the sample to maintain its paramagnetic properties over time without degradation or significant changes
- The largest gap in double integral values was seen in the solution containing 5% phenol(as seen in Figure 3), showing that the reactivity between phenol and radical is not as significant unless the concentration of phenol is 5% or higher.

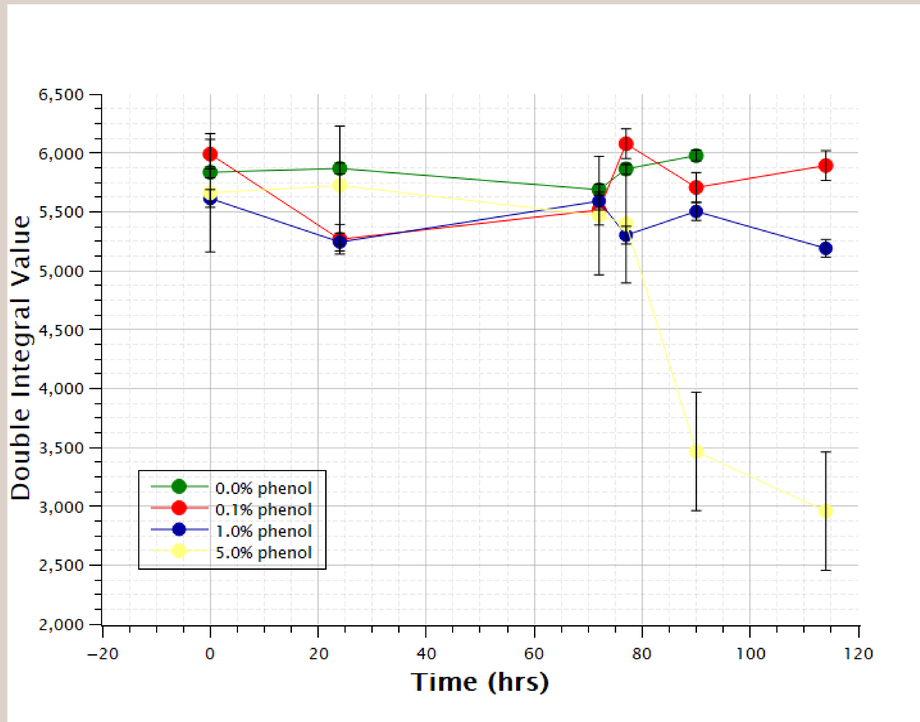


Figure 3. Double integral values extracted from CW- EPR spectra (The lines depicted solely serve as guides)

References

Bennati, M. and Orlando, T. (2019). Overhauser DNP in Liquids on 13C Nuclei. In eMagRes (eds R.K. Harris and R.L. Wasylishen). https://doi.org/10.1002/9780470034590.emrstm1581

Corvaja, Carlo. (2008). Introduction to Electron Paramagnetic Resonance. Pg 1-22. 10.1002/9780470432235.ch1.