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Supporting Information

A Green Approach to Fenton Chemistry: Mono-Hydroxylation of salicylic acid in aqueous medium by the electrogeneration of Fenton's reagent

Janjira Panchompoo,[1] Leigh Aldous,[1] Mikhail Kabeshov,[2] Ben S. Pilgrim,[2] Timothy J. Donohoe[2] and Richard G. Compton,[1,]*

[1] Department of Chemistry, Physical and Theoretical Chemistry Laboratory, University of Oxford, South Parks Road, Oxford OX1 3QZ, United Kingdom

[2] Department of Chemistry, Chemistry Research Laboratory, University of Oxford, Mansfield Road, Oxford OX1 3TA, United Kingdom

*Author to whom all correspondence should be addressed.

Email: richard.compton@chem.ox.ac.uk

Tel: +44 (0) 1865 275 413

Fax: +44 (0) 1865 275 410

I. Titration of hydrogen peroxide by $KMnO_4$

The most commonly used method for the quantification of hydrogen peroxide is titration with potassium permanganate.[1, 2] In acidic aqueous solution, potassium permanganate is a stronger oxidising agent than hydrogen peroxide and it can quantitatively react with hydrogen peroxide as shown below in Equation 1.[1, 2]

$$2MnO_4^- + 5H_2O_2 + 6H^+ \rightarrow 2Mn^{2+} + 5O_2 + 8H_2O \qquad (1)$$

Once all hydrogen peroxide is consumed, the next drop of potassium permanganate added into the analyte solution will cause an easily detected colour change, going from colourless to a faint persisting pink solution. The stoichiometry of the reaction shows that 2 moles of potassium permanganate will react with 5 moles of hydrogen peroxide. Thus, if the concentration of potassium permanganate is known, we can figure out the exact concentration of hydrogen peroxide presented in the solution ($C_{H_2O_2}$) by using Equation (2).

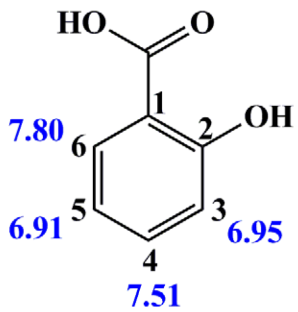
$$C_{H_2O_2} = \frac{5}{2} \cdot \frac{C_{KMnO_4} \cdot V_{KMnO_4}}{V_{H_2O_2}} \qquad (2)$$

where $C_{H_2O_2}$ is the concentration (mol/L) of the unknown hydrogen peroxide that reacted with potassium permanganate in the titration. C_{KMnO_4} and V_{KMnO_4} are the concentration (mol/L) and volume (mL) of potassium permanganate used as titrant. $V_{H_2O_2}$ is the volume (mL) of hydrogen peroxide solution being titrated.

It is important to note that a significant amount of hydrogen ion (H^+) is consumed during the titration process, as presented in Equation (1). Consequently, in this study we added 10.00 mL of 1 M H_2SO_4 into each 5.00 mL of hydrogen peroxide solution being titrated in order to make sure that hydrogen ion needed for the reaction is readily available in a sufficient amount.

II. ^{1}H NMR spectra

The electrolysed mixture before (Figure 1) and after (Figure 2) controlled potential electrolysis reaction was analysed by proton nuclear magnetic resonance (^{1}H NMR) spectroscopy in order to confirm the reaction products.



salicylic acid

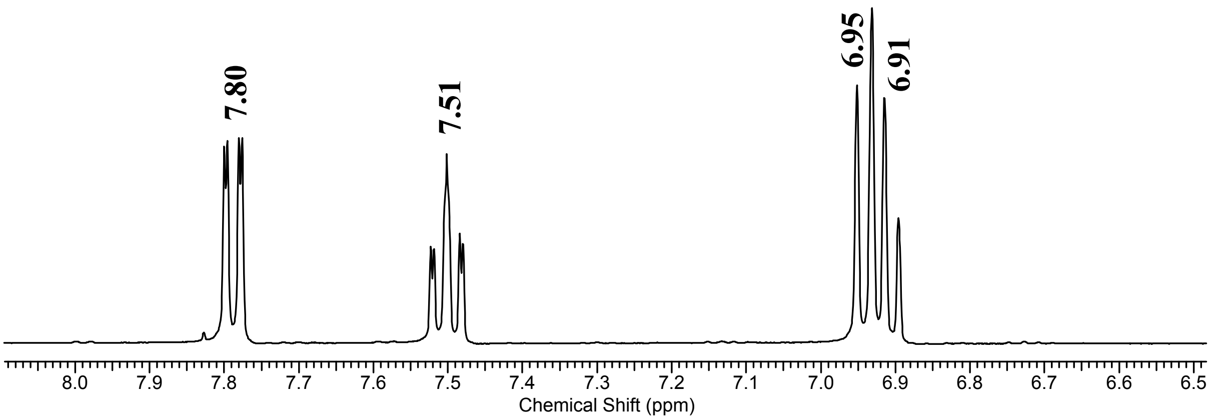
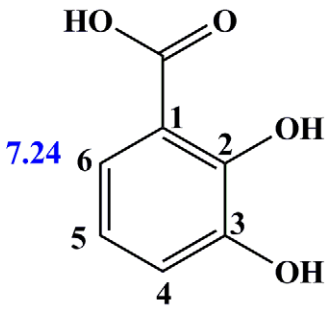
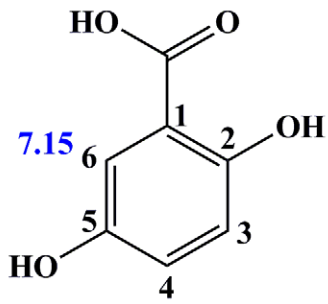


Figure 1: ^{1}H NMR spectra (zoomed in and cut to the region of interest) of an O_2-saturated 0.1 M Na_2SO_4 pH 3.0 solution containing 5 mM salicylic acid and 5 mM Fe(II) before the controlled-potential electrolysis.



2,3-dihydroxybenzoic acid



2,5-dihydroxybenzoic acid

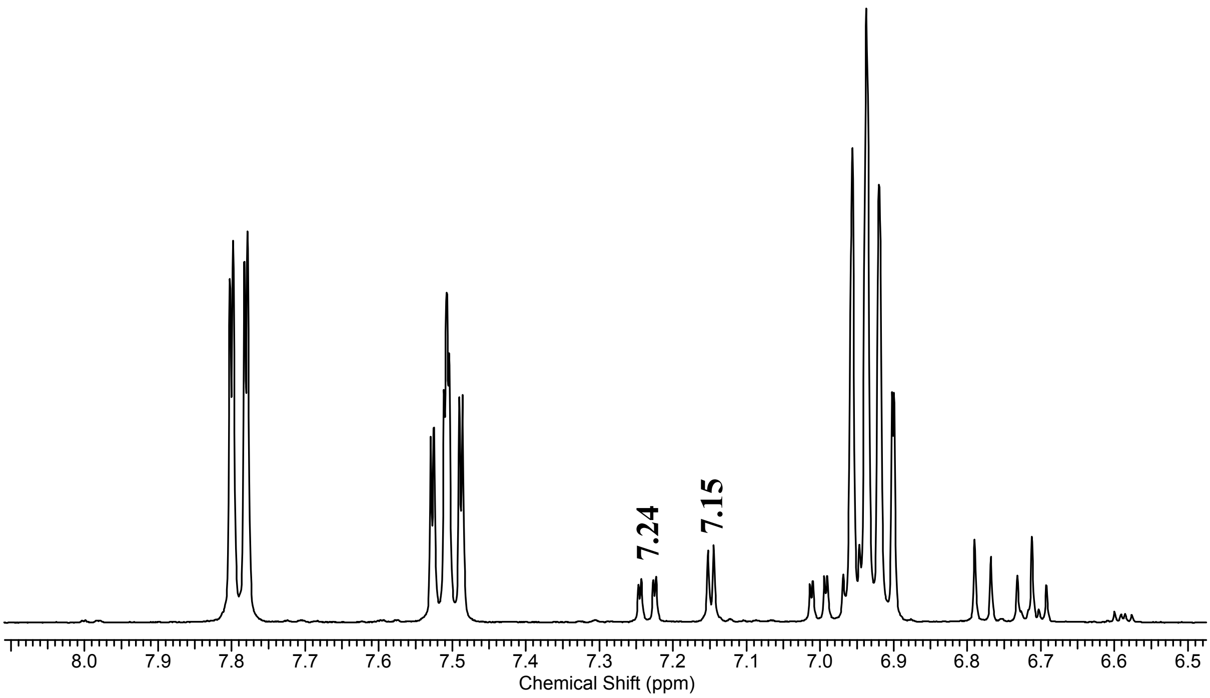


Figure 2: [1]H NMR spectra (zoomed in and cut to the region of interest) of an O_2-saturated 0.1 M Na_2SO_4 pH 3.0 solution containing 5 mM salicylic acid and 5 mM Fe(II) after the controlled-potential electrolysis.

III. References

1. J. S. Reichert, S. A. McNeight and H. W. Rudel, *Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 194-197.

2. G. H. Jeffery, J. Bassett, J. Mendham and R. C. Denney, *Vogel's textbook of quantitative chemical analysis*, Longman Scientific & Technical, Harlow, 1989.