Supplementary Material (ESI) for The Analyst

A Novel Electroreduction Strategy for the Determination of Sulphite

Anita Isaac, Andrew J. Wain, Richard G. Compton, Callum Livingstone and James Davis[*]

Supporting Information

Experimental Details

Reagents: All reagents were of the highest grade available and used without further purification. Stock solutions of sulphite (10mM in deionised water) and ascorbate (10mM, pH 7) were prepared daily. The analysis solutions consisted of Britton-Robinson buffer (acetic, boric and phosphoric acids - each at a concentration of 0.04 M) to which 0.1M KCl was added.

Instrumentation: Electrochemical measurements were conducted using a μAutolab type III computer controlled potentiostat (Eco-Chemie, Utrecht, The Netherlands) with a standard three electrode configuration generally used throughout.

Laminate Fabrication: Copper foils (100um) were thermally sandwiched between commercial resin backed polyester laminating pouches (75-125 um thick). The design approach used in the construction of the integrated electrode system. The ring electrodes were initially completely encapsulated between the insulating laminate, baked at 100°C for 1 hour and then drilled (1-5mm diameter) to reveal the ring surfaces. The drilling process was clean in that there was no evidence of inter-layer smearing – corroborated by examining for "shorts" between individual strips with a digital multi-meter. The reference electrode was constructed through electroplating silver (1 mM $AgNO_3$, 0.1M HNO_3, -1V) on to the middle copper layer. The silver layer was then oxidised by holding the electrode a +1V in 1M KCl. These electrode modifications were conducted using a separate Ag|AgCl reference (BAS Technicol, 3M Cl^-) and platinum counter wire rather than the electrodes held within the laminate.

---

[*] Chemistry, School of Biomedical and Natural Sciences, Nottingham Trent University, Nottingham, NG11 8NS, +44 (0) 115 848 3218, Email: james.davis@ntu.ac.uk

ESR Data: In-situ electrochemical ESR experiments were carried out using a copper tube flow cell of internal diameter 1 mm, the characterisation of which has been reported previously. A platinum gauze counter electrode was positioned downstream of the 3 mm long copper working electrode and an SCE reference electrode was placed upstream. ESR spectra were obtained using a JEOL JES-FA100 X-band spectrometer with a cylindrical (TE011) cavity resonator, employing a magnetic field modulation width of 0.1 mT and a microwave power of 1 mW.