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## Low-volume electrochemical cell for in-situ measurement of XAS spectra of reactive metal compounds and biomolecules

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The design and operation of a low volume spectroelectrochemical (SEC) cell for the measurement of X-ray absorption spectra (XAS) of solutions at room temperature will be described. The XAS-SEC cell is suitable for operation with fluorescence detection where the  $2 \times 2 \times 12$  mm working electrode chamber doubles as the solution cell for XAS measurements. A key element of the design of the experiment is the control of the flow properties of the solution under electrochemical/XAS interrogation where a continuous pulsing of the solution with a small net flow rate ensures both rapid electrosynthesis and that photoreduction can be controlled. The operation of the XAS-SEC cell is demonstrated by the X-ray absorption near edge spectra (XANES) of solutions of  $K_3[Fe(C_2O_4)_3]$  under static and dynamic flow conditions and the XANES of  $[Fe(C_2O_4)_3]^{4-}$  obtained by electrochemical reduction. The suitability of the approach for the study of protein samples is demonstrated by preliminary measurement of the ubiquitous electron transport protein cytochrome c.

### Keywords

XAS; XANES; Electrochemistry; Metalloproteins; iron complexes

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