L-21 **Thursday, 16.06., 10^{20} – 11^{00}**

The Hybrid XAS technique for dilute [1-10 mM] solutions at high accuracy

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XAFS can now be used to investigate electron inelastic mean free paths, dynamical and thermal bonding, to measure nanoroughness and most importantly to assess the significance of alternate hypotheses with derived experimental uncertainty.

A new approach is introduced for determining XAS spectra on absolute and relative scales using multiple solutions with different concentrations by the characterisation and correction for experimental systematics. This Hybrid technique is a development of standard XAFS along the lines of high-accuracy XERT but with applicability to solutions, dilute systems and cold cell environments. We have applied this methodology to determining absolute XAS of [bis(N-n-propyl-salicylaldiminato)] nickel(II) and [bis(N-i-propyl-salicylaldiminato)] nickel(II) complexes with square planar and tetrahedral structures in 15 mM and 1.5 mM dilute solutions. Dilute systems provide excellent XANES and XAFS spectra by transmission, and we confirm that transmission measurements can be superior to conventional fluorescence measurements even for dilute systems. For the first time, we have determined XAS of the isomers from low concentration solutions on an absolute scale with a 1%-5% accuracy, and with relative precision to 0.1% to 0.2% in the active XANES and XAFS regions after inclusion of systematic corrections. This allows a range of new insights for multi-phase systems, for going beyond fingerprinting to theoretical validation and for species identification in dilute systems using XAFS.

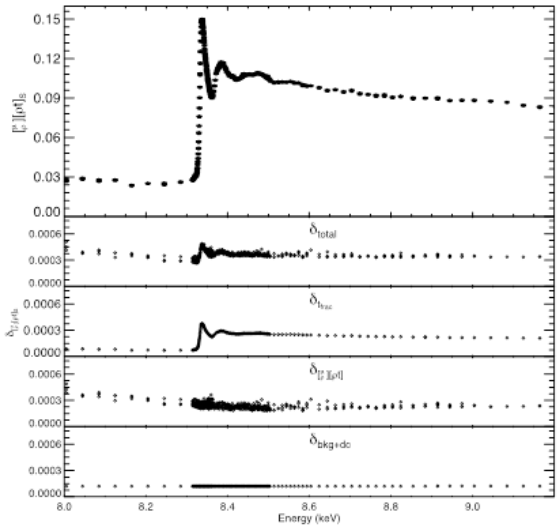

Figure 1. Corrected and normalised XAS of the (i-pr Ni) isomer determined from the attenuations of 15 mM solutions following the solvent subtraction. The modelled background was subtracted from the corrected (dark-current and scatting effect) attenuations of the solutions (shown by the ⋄ symbols; sample detail). The corresponding uncertainty was propagated from the uncertainty contributions of experimental systematics, and from the variance of repeated measurements with both the solution and the solvent. The defined accuracy will allow the reliable structural analysis of (i-pr Ni) using XAFS. At each of the energies, three aperture dependent measurements are in excellent agreement.

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[2] M. T. Islam, C. T. Chantler, L. J. Tantau, C. Q. Tran, M. H. Cheah, A. T. Payne, S. P. Best, *J. Synchr. Rad.* **22** (2015) 1475.

[3] M. T. Islam, J. D. Bourke, S. P. Best, L. J. Tantau, C. Q. Tran, F Wang, C. T. Chantler, *J. Phys. Chem. C,* **120**(17) (2016) 9399–9418.