

# **RSC Analytical Division Awards Symposium and AGM 2009**

**Manchester Metropolitan University, 18th May 2009**

**Venue: Lecture Theatre T0.03 in John Dalton Building MMU**

[http://www.rsc.org/images/Maps\\_tcm18-151771.pdf](http://www.rsc.org/images/Maps_tcm18-151771.pdf)

12.30 Registration

13.00 Buffet Lunch

13.45 Opening Remarks

14.00 **Theophilus Redwood Lectureship Winner 2009**  
**Frank Marken, University of Bath, UK**

14.45 **L S Theobald Lectureship Winner 2008**  
**Steve Ellison, LGC Ltd, UK**

15.30 Tea/Coffee

16.00 **Robert Boyle Medal Winner 2008**  
**R Graham Cooks, Purdue University, USA**

17.00 Meeting Ends

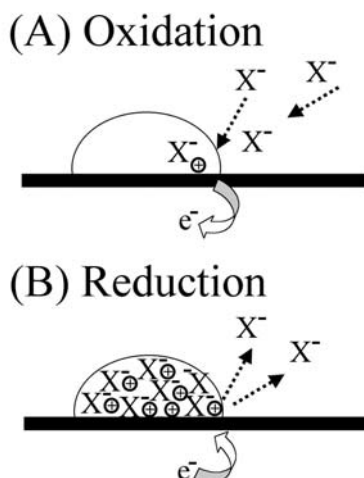
17.15 **RSC Analytical Division AGM**

# Ion Transfer Voltammetry: From Femtoliter Droplets to Flowing Solutions

Frank Marken

Department of Chemistry, University of Bath, Claverton Down,  
Bath BA2 7AY, UK.

This lecture aims at introducing the audience to electrochemical processes involving ion transfer at liquid | liquid | solid triple phase boundaries. Electrochemically driven ion transfer processes from aqueous to organic media are readily observed at electrodes modified with an immobilised water immiscible redox liquid [1]. The transfer of ions occurs at a potential determined by the aqueous concentration and aqueous hydration energy and has recently been investigated for example for the transfer sulphite [2] and for carboxylates [3].



Anion transfer processes are reported for immobilised droplets or films, and for flowing 4-(3-phenylpropyl)pyridine | aqueous electrolyte or acetonitrile | aqueous electrolyte interfaces. The application and benefits of this methodology in flow microreactor electroanalysis, for ion separation, or for sensors will be discussed.

- [1] The electrochemical ion-transfer reactivity of porphyrinato metal complexes in 4-(3-phenylpropyl)pyridine | water systems. M.J. Bonné, C. Reynolds, S. Yates, G. Shul, J. Niedziolka, M. Opallo, F. Marken, *New J. Chem.* 30 (2006) 327.
- [2] Electrocatalytic determination of sulfite at immobilized microdroplet liquid - liquid interfaces: The EIC' mechanism. N. Katif, S.M. MacDonald, A.M. Kelly, E. Galbraith, T.D. James, A.T. Lubben, M. Opallo, F. Marken, *Electroanalysis* 20 (2008) 469.
- [3] Probing Carboxylate Gibbs Transfer Energies via Liquid | Liquid Transfer at Triple Phase Boundary Electrodes: Ion-Transfer Voltammetry versus COSMO-RS Predictions. S.M. MacDonald, M. Opallo, A. Klamt, F. Eckert, F. Marken, *PCCP*, 10 (2008) 3925.

# Measurements for Regulation: The Roles of Analytical Quality and Measurement Uncertainty

Stephen L R Ellison

LGC Limited, Queens Road, Teddington, England

Analytical measurement supports the regulation of trade, transport of goods, health services, and environmental protection. These activities have immense economic significance; for example, world exports of food, chemicals and mineral products alone in 2002 were valued at over two trillion US dollars, and all are subject to regulation that depend on analytical measurement. The analytical measurements themselves also represent substantial costs; in the UK alone, it has been estimated that £7.4bn (over 10m US dollars) is spent annually on analytical measurement, and the principal reason for analysis given by companies is "Compliance with regulation". The commercial reason for this expenditure is simple; the cost of non-compliance is usually arranged to exceed the cost of compliance. Nor is the consequence of non-compliance purely economic; regulation is often put in place to protect the public, and a substantial failure has potentially serious health and welfare implications. These factors make it vitally important to understand and manage the quality of analytical measurement.

Among the most important indicators of analytical measurement quality is the uncertainty associated with a measurement result, and analysts worldwide are increasingly required to assess and (where appropriate) to report the uncertainty associated with their analytical results. Though the existence of experimental uncertainty has been understood perhaps as long as analytical science has existed, the new requirements have generated an apparently new question for many working analysts and regulators; how should uncertainty information be used in regulation?

This presentation will discuss the issues associated with this question. The presentation will consider the importance of measurement in regulation, and discuss the regulatory and accreditation context in which limits are defined and analytical measurement data used. The differing needs of regulators, analysts and enforcement officers will be considered. The paper will briefly introduce the concept of measurement uncertainty and describe current approaches to the assessment of uncertainty for analytical work, with attention to the relative advantages and disadvantages of each. The impact of uncertainty on regulatory decisions will be discussed, with reference to recent studies of regulatory practice in the EU and examples from regulatory enforcement in and outside the UK. Finally, the respective responsibilities of regulators, accreditation agencies, laboratories and other organisations will be considered.

In summary, it will be shown that the widespread implementation of measurement uncertainty assessment and reporting in laboratories has generated, or perhaps re-generated, a range of questions for analytical laboratories and regulators alike; that these questions have practical implications in the field; and that regulators, international standardisation bodies and laboratories all have a part to play in setting appropriate policies for current and future regulation

# **Ion Chemistry Outside the Mass Spectrometer: Ambient Ionization, Dissociation and Reaction**

R. Graham Cooks  
Department of Chemistry, Purdue University, USA

This presentation attempts a reconceptualization of chemical analysis by mass spectrometry. This venerable method can give instantaneous results, on samples that have not been prepared for analysis, in experiments done in the field. To achieve these aims the instrumentation must be miniaturized, chromatography must be abandoned in favor of tandem mass spectrometry (MS/MS) and the new ambient ionization methods must be deployed. This presentation describes how these aims have been achieved. We describe (1) a miniature hand-held MS/MS instrument, capable of unit mass resolution to 500 Th but also capable of protein analysis. (2) Two ambient ionization methods – by definition methods in which the sample is not introduced into the vacuum system – that allow instantaneous MS analysis of complex samples which have not undergone prior preparation. One method is based on ionization using solvent droplets, the other on plasma ionization. Specificity is achieved by MS/MS and by chemical reactions associated with ionization. Imaging of latent fingerprints, determination of tumor margins in tissue sections, contaminants in foodstuffs and other applications are described. (3) A successful interface between the Mini MS and the ambient ion sources is described too.

Processes in which ions are reacted, dissociated and soft-landed onto surfaces – all in air – are implemented. These experiments take the standard unit operations of mass spectrometry and perform them on ions of measured mass outside the mass spectrometer. These developments increase the flexibility of mass spectrometry as an analytical method; they also extend its role as a preparative method in which material is selected on the basis of mass and then collected for independent experiments.