



CENTRE FOR PROCESS ANALYTICS AND CONTROL TECHNOLOGIES

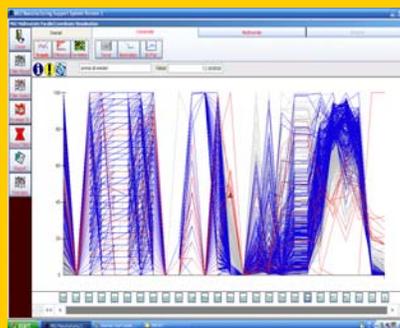
## CPACT NEWSLETTER

DECEMBER 2008

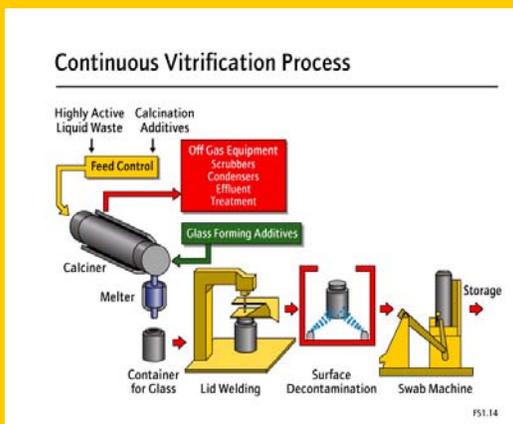


Sellafield is the largest and most complex nuclear site in the UK. The site contains over 1,000 facilities which perform a wide range of tasks including nuclear fuel reprocessing, MOX fuel manufacture, major decommissioning projects and radioactive waste management. The Sellafield site is operated by Sellafield Ltd, on behalf of the Nuclear Decommissioning Agency (NDA) and employs over 10,000 people.

A key technology used at Sellafield is vitrification, which is a process where Highly radioActive Liquid (HAL) waste is processed into calcine then immobilised by chemically bonding it with glass to produce vitrified waste.



Parallel Co-ordinate Visualisation from the MS2 Process Analysis



Vitrification Process Overview

Future investigations involving the MS2™ system will focus on ways to target the main causes of premature failure and other challenging areas, investigate ways to improve equipment performance, reduce container processing residence time and wash cycles in decontamination and identify operational parameters which prolong torch welding life”

Using the MS2™ system means there is an opportunity to reduce the time taken to study process data and to draw conclusions leading to potential efficiency savings. There are also opportunities to improve the safety culture and performance. Finally the team are looking at applying data analysis to a range of different data sets to gain a better understanding of key plant areas to improve the process control performance.

Carl Steele, Sellafield Ltd

Process improvement is an important objective at Sellafield. In partnership with the National Nuclear Laboratory (NNL) a full scale non-radioactive test rig of the vitrification facility has been developed. This is leading to a number of key process improvements including increased waste incorporation, which could lead to a reduction in the volume of waste that will need to be disposed of. The test rig has also helped widen the process envelope, increase mass throughput and optimised the use of sugar to reduce the impact of dust carried over into the off-gas system for recycling.

Sellafield has also applied AJM Consulting Ltd’s MS2™ Process Diagnostics system to several investigations at the vitrification facility. Dr Carl Steele, part of Sellafield Ltd High Level Waste (HLW) technical support team is using MS2 with positive results:

“MS2’s data filtering function, particularly its applicability to non-numeric data, has proved most useful in enabling investigations into robot arm fault causes. For welding operations data analysis, MS2 has identified several multivariate regions of operation of the weld torch. It has also been applied to interpreting databases and factors affecting melter performance and longevity.



### Process Analysis and Data Analysis Support Lean Manufacturing at Syngenta

The measurement and analysis of process data are key capabilities for enhancing process understanding, and reducing variability and waste in lean manufacturing processes. The application of process analysis and data analysis tools reduces waste in Syngenta’s lean manufacturing processes. The implementation of Pfaudler maintenance free pH measurement technology at the Huddersfield and Grangemouth Manufacturing Centres resulted in reduced cycle time and reduced operator exposure. The Kaiku Intelligent Pipe, which uses impedance spectroscopy to detect small process changes, has been evaluated for detection of wash water cleanliness to reduce cycle time and wash water usage during product changeover in multiproduct plant. Data analysis tools are used for enhancing process understanding, running efficient plant trials, and rapidly identifying and rectifying process problems. In one example a process problem was identified using a Shewart control chart and then the cause of the problem was identified using principal components analysis. The rapid resolution of the processing problem minimised process downtime and maintained product quality.

Valerie Wheeler, Syngenta

## CPACT Academics in demand!

David Littlejohn (CPACT Strathclyde), Paul Watts (CPACT Hull) and Julian Morris (CPACT Newcastle) were all invited to talk at the CPAC Satellite Workshop in Rome on 26-28 March 2008. David gave a talk entitled 'Non-invasive and On-Line Techniques for Monitoring Fermentation Processes at Development Scale'. Paul Watts presented on 'High Throughput Synthesis in Flow Reactors' and Julian Morris talked about 'Process Performance Monitoring'.

### David Littlejohn had also been invited to talk this year at the following events:

- Launch of the Chemical Sciences Scotland Breakfast meeting on Wednesday 14th May. 'On-line Process Analysis and Control—Examples, benefits, barriers and how to increase up-take'

- Particulate Systems Analysis PSA 2008 Conference in Stratford upon Avon on 2-4th September 2008.

- 'PAT—What it is and why it is relevant to all'

- 187th meeting of the Infrared and Raman Discussion Group which took place on 10-11th September 2008 at Glasgow University. 'The application of vibrational spectroscopy as a process analytical probe'



### Julian Morris had also been invited to talk this year at the following events:

- Invited Keynote Speaker, "PAT and its Implications for Robust Process Monitoring and Closed loop Control", EuPAT, Scientific Progress Underpinning Innovative Manufacturing Control and Quality by Design, Göteborg Sweden, 28-29 October 2008

- Invited Speaker, "Process Analytical Technologies and Quality by Design – a Process Systems Engineering View", ISPE Conference: PAT Data Management, September, Manchester, 2008

- Invited Speaker, "Process Analytical Technologies and Quality by Design – a Process Systems Engineering View", 7<sup>th</sup> European Symposium on Biochemical Engineering (ESBES 7), Faro Portugal, September 2008

- Invited Speaker, "Process Analytical Technologies and Quality by Design – a Process Systems Engineering View", Royal Statistical Society Conference, Nottingham, September 2008

- Invited Keynote Speaker, "Chemometrics and its Relation to PAT", IBC Euroforum Meeting on PAT- Process Analytical Technology, 1-2 April 2008, Copenhagen

- Invited Keynote Speaker, 'PAT and its Implications for Robust Process Monitoring and Closed loop Control', Pharma IQ Annual PAT and Quality by Design Meeting, 28-29 February 2008, London



- Invited PAT Workshop (1 Day Workshop), "PAT, Lean Six Sigma and Multivariate Data Analysis", Pharma IQ Annual PAT and Quality by Design Meeting, 28-29 February 2008, London

- Invited Opening Plenary Speaker, "PAT – the impact for Process Systems Engineering", IFPAC 2007, Baltimore USA, January 2008

### Paul Watts had also been invited to talk this year at the following events:

- Invited Lecture, 'Continuous flow synthesis in micro reactors', Chemische und biologische mikrolabortechnik, Ilmenau, Germany, February 26-28 2008.

- Lecture, 'The use of immobilised crown ethers as in-situ protecting groups in organic synthesis', ACS National Conference, New Orleans, April 6-10 2008.

- Invited Lecture, 'Flow reactors for chemical synthesis: An opportunity for process control', IMRET 10, New Orleans, April 6-10 2008.

- Lecture, 'Immobilisation of thermophilic enzymes within miniaturised flow reactors for biotransformations', IMRET 10, New Orleans, April 6-10 2008.

- Keynote Lecture, 'Organic synthesis in continuous flow reactors', SynTOP 2008: Smart synthesis and technologies for organic processes bridging chemistry and engineering, Berlin, Germany, June 11-13 2008.

- Invited Lecture, 'Complex chemistries within micro flow reactors', CPAC Summer Workshop and Conference, Seattle, USA, July 15-17 2008.

- Plenary Lecture, 'Enhanced chemical synthesis using flow reactors', Second Biotage Summer Program – Modern Tools in Organic Synthesis, Edinburgh, July 29-31 2008.

- Invited Lecture, 'Optimised high throughput chemical synthesis using flow reactors', Gordon Conference, Combinatorial Chemistry: High throughput chemistry and chemical biology, Oxford, August 31 - September 5 2008.

- Invited Lecture, 'Chemical synthesis in flow reactors', The Siena conference on product and process optimization, Siena, Italy, October 5-8 2008.

- Invited Lecture, 'Organic synthesis in micro reactors', EANM08: Annual congress of the European Association of Nuclear Medicine, Munich, October 11-15 2008.

- Invited Lecture, 'Chemical synthesis in flow reactors', Petrochem Business Links, Rotterdam, Netherlands, November 6 2008.

- Invited Lecture, 'Chemical synthesis in continuous flow reactors', VITO, Mol, Belgium, November 14 2008.



*If you would like more details about any of the above presentations, please e-mail [natalie@cpact.com](mailto:natalie@cpact.com)*

## Knowledge Transfer Partnerships

**KTP's are extremely effective!**

I hate to put the money side of things first, but KTPs are extremely effective. Selling the idea of a high level graduate for a three year period for the price of a graduate trainee for one year is not the hardest sell. When you are combining this with technology transfer from a top university, the projects almost sell themselves.

The application process for many grants is quite daunting for those of us who aren't involved in their preparation on a routine basis. Happily, the KTP process is fairly straight forward and there is plenty of assistance from the dedicated KTP support offices in the universities to help you through the complicated parts. An experienced KTP adviser familiar with your region and your industry, was also on hand, throughout the application process. So, whilst I wouldn't say it was a breeze, it did pass without loss of too much hair.

There is a very strong focus on delivery of the technology, as real business applications, in a KTP project. Importantly, the application process defines the work program to a very detailed level and sets up a local management committee to oversee the project. This ensures understanding and commitment to the programme before commencement and avoids the possibility of a constantly evolving project ultimately under achieving.

*Colin Clarke, National Nuclear Laboratory*



## Transmission Raman Studies

Nicci Townshend, a CPACT PhD student, has been carrying out a series of experiments using the Kaiser Optical Systems Raman Workstation, on loan to CPACT from Clairet Scientific Ltd. Nicci's work has involved looking at pharmaceutical tablets with a low concentration of the active ingredient (<3% w/w). As expected, Raman spectroscopy has proved useful for these samples as the technique allows the relatively strong fundamental vibrations of the molecules to be studied. The Raman Workstation uses Kaiser's PhAT probe technology to obtain spectra from a large area of the tablets in reflection and also to apply the new technique called transmission Raman where the exciting laser is focussed on one side of the tablet, while the Raman spectrum is collected from the opposite side. Transmission Raman therefore collects Raman scattering data from the entire thickness of the tablet.

Nicci has measured Raman spectra from literally hundreds of samples. She has also produced "calibration tablets" which contain known concentrations of the same active ingredient, but held together in a different matrix of excipients. The calibration tablets have then been used to predict the concentration of the active in the real tablets. The reflection Raman data gives a highly accurate quantitative prediction of active ingredient content. Nicci is now focussing her attention on analysing the real and calibration tablets by HPLC in order to give a direct comparison between the results obtained with Raman and liquid chromatography. Nicci is also further investigating the effect of particle size on the results produced by transmission Raman.

*John Andrews, Clairet Scientific*

