AXAA e-newsletter

Issue 3, 2014 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Members and Friends,

As the end of the year approaches it is worthwhile reflecting on 2014 which has been a busy year for AXAA. Of course, the main event was the 2014 AXAA Workshops, Conference and Exhibition (AXAA-2014) in February, where some 200 participants from industry, academia and research institutions from across Australia and around the globe converged on Perth for a week of learning, dissemination, discussion and networking centred on X-ray and associated techniques of analysis. Photos from AXAA-2014 can still be found online.

We are extremely excited that the special AXAA-2014 conference proceedings issue of Powder Diffraction Journal will be released very shortly, with most of the contributions already appearing as *FirstView* Articles on the journal website. We are delighted with the quality of all of the papers, and thank all of the authors, reviewers and editors for their efforts. At this stage we anticipate that this exciting publishing opportunity will also be offered for participants at AXAA-2017.

The Australian Synchrotron User Meeting was held recently at the National Centre for Synchrotron Science, and AXAA was a proud Bronze Sponsor of this annual event which showcases recent beamline developments, as well as their application in materials, earth, environmental and biological sciences to name a few. As a regular synchrotron user and participant in the user meeting, I have witnessed the evolution of the facility from its early days in 2007, to the vibrant research institution that it is now with beamlines competing strongly with those at the large European and US facilities in terms of scientific output and impact. For the first time in 2014, the New User Symposium was held immediately before the User Meeting, and I encourage all interested AXAA members to head along to future events and get involved with the synchrotron community.

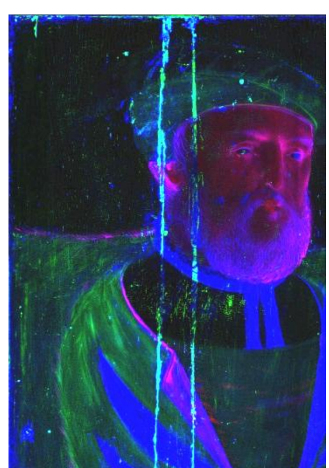
Finally, the National Council met recently and decided that AXAA-2017 will be held in Melbourne! In the coming months we will decide on a conference venue, so stay tuned for further details in coming Newsletters. For now, on behalf of the National Council I'd like to wish all of the AXAA community an enjoyable festive season, and a happy and healthy 2015.

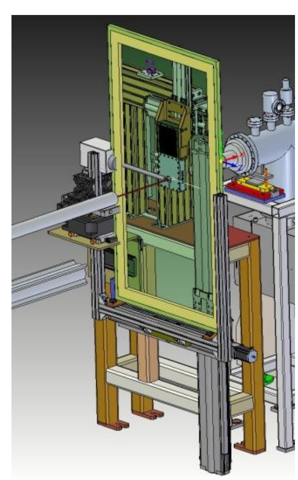
Nathan Webster
AXAA President

X-ray Fluorescence Microscopy

Vital statistics and new capabilities, enabling science across many disciplines

The X-ray Fluorescence Microscopy (XFM) beamline [1] at the Australian Synchrotron has hosted over 250 user groups since operations began in 2008, and sustains high levels of oversubscription. XFM can be used for elemental and chemical microanalysis across many length scales and is a powerful tool for quantitatively mapping trace elements within whole biological specimens. Armed with the world-leading Maia detector [2,3], and ongoing collaboration with CSIRO, almost 100,000 scans have been performed, representing an estimated 100 Gpixels of data and a stage transit of around 200 km with a positioning accuracy of 2 µm. The advances in X-ray fluorescence detection now enable routine acquisition at mega-pixel per hour rates which in turn allows collection of 3D information such as tomography in realistic times (hours rather than weeks to complete a tomographic data set). Chemical Speciation Imaging (CSI) or X-ray Absorption Near Edge Structure (XANES) imaging results in a stack of 2D images with the third dimension containing a XANES spectra in each pixel [4]. CSI has been demonstrated with moderate definition (10,000s of pixels/image) across a diverse range of applications [5]. Recent studies have improved the efficiency and sensitivity of CSI to environmentally relevant concentrations [6].





Left) RGB composite image of Henry VIII portrait showing mercury (red), calcium (green), and iron (blue) collected on the prototype milliprobe. Right) Mockup of milliprobe large area (0.6 m X 1.2 m) scanning stages with ~50 µm resolution.

In order to fully harness the power of the Maia detector, we have undertaken an ambitious series of upgrades to microprobe and nanoprobe support tables, scanning stages, tomography, and large-area scanning stages - the milliprobe. We have also developed cryogenics for the microprobe instrument giving temperature control for the specimen environment.

Finally, there is a range of beamline ancillaries including equipment for specimen pre-alignment, cryogenic specimen preparation and a portable XRF analyser for measurement of trace and major element concentrations in samples before XFM.

For more detailed information on the XFM beamline capabilities see our webpage, or contact the XFM staff at XFM@synchrotron.org.au. The breadth of science conducted at XFM is also highlighted here.

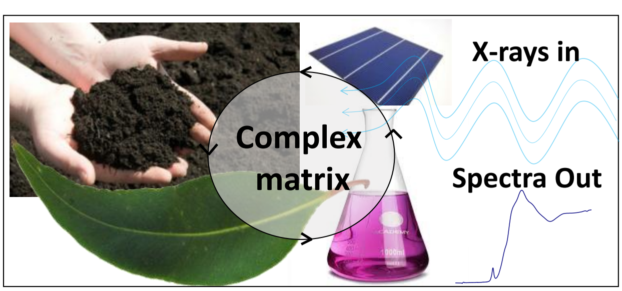
David Paterson
XFM Beamline, Australian Synchrotron

[1] Paterson D. *et al.*, *AIP Conference Proceedings*, 2011;**1365**;219.
[2] Siddons DP *et al.*, *AIP Conference Proceedings*, 2004;**705**;953.
[3] Kirkham R *et al.*, *AIP Conference Proceedings*, 2010;**1234**;240.
[4] Etschmann BE *et al.*, *American Mineralogist*, 2010;**95**;884.
[5] Kopittke PM *et al.*, *New Phytologist*, 2014;**201**;1251.
[6] Etschmann BE *et al.*, *Environmental Chemistry*, 2014;**11**;341.

X-ray Absorption Spectroscopy

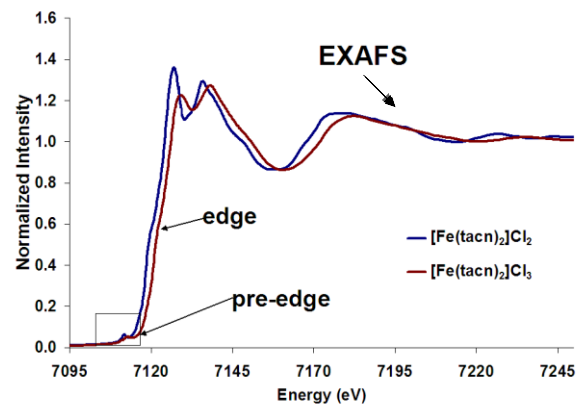
From Soils to Solar Fuels, and All That Lies Between

Members of AXAA are undoubtedly aware of the importance of X-rays to our understanding of molecular-level structure. But I would like you, as analytical scientists, to reflect on the challenge of studying a material which is not crystalline, where you need to know something that is happening at a molecular-level. It may be the fate of a trace metal in a soil, a contaminant in a production process, or the fate of starting material. You may ask, can this problem be solved with traditional spectroscopic techniques? Can magnetic or electronic spectroscopies help me? In some cases the answer may be yes, but in many others like soils, production processes and solar fuels, you have a complicated matrix which interferes with most analytical techniques, and what you need to know can remain a mystery.



Herein lies the advantage of X-ray Absorption Spectroscopy (XAS). Using this technique we can, in principle, "home in on an element of interest" and take information rich spectra against what can be an incredibly complex background matrix, even *in situ*.

An XAS experiment fundamentally probes absorption as a function of incoming X-ray wavelength. You could describe it as a synchrotron based cousin of UV-Vis. An example of a two Fe K-edge spectra are given below. The spectra obtained are typically information rich, containing formation about bonding (pre-edge), oxidation state (edge) and electron diffraction type information (EXAFS). The technique has contributed substantially to our understanding of molecular-level processes in systems as diverse as soils and solar cells.[1-3]



The XAS community in Australia were quite excited to have Dr Bruce Ravel [4] visit from the NIST Materials Measurement Science Division, Synchrotron Methods Group, where he gave a plenary at the Australian Synchrotron Users Meeting "X-ray Absorption Spectroscopy in the Age of Insertion Devices" on a range of different analytical techniques. We were also lucky to have Bruce stay a few extra days to give an advanced XAS course on the use Artermis for EXAFS fitting.

Finally, I wanted to conclude this piece with a brief mention about the future of XAS in Australia. While XAS can in prinicpal be done on any element from boron onwards, in Australia we currently do not quite cover the critical range of energies where XAS has contributed a lot, that being the mid-energy XAS region from about 1000 eV- 4500 eV which includes S and Cl, important to many industrial and academic processes. Going forward I would love to see a mid-energy XAS beamline at the Australian synchrotron. I believe it could contribute substantially to a number of major industrial and academic programs, not limited to things such as trace metals in coal, the study of acid sulfate soils and new organic solar cells. Beamlines need investment and support and we all need to think about how to make this happen going forward for both XAS and beyond.

Rosalie Hocking
James Cook University

[1] Analysis of Soils and Minerals Using X-ray Absorption Spectroscopy, Kelly SD, Hesterberg D, Ravel B, Soil Science Society of America, Methods of Soil Analysis Part 5, Mineralogical Methods SSSA Book Series No. 5. 387-464.
[2] van Kuiken BE *et al.*, *J. Phys. Chem. Lett.*, 2012;**3**;1695–1700.
[3] Hocking RK *et al.*, *Aus. J. Chem*, 2012;**65**;608–614.
[4] Ravel B and Newville M, *J. Sync. Rad.*, 2015;**12**;537–541.

Applications of Synchrotron-Based X-ray Tomography

X-ray Computed Tomography (CT) is one of the most important techniques in X-ray analysis, with applications ranging from diagnostic medicine to fundamental materials science. Synchrotron based X-ray tomography offers a range of advantages over laboratory based systems, including improved image resolution, high-energy monochromatic beams and high beam intensities which can enable time-resolved measurements. This article highlights some recent applications of synchrotron-based X-ray tomography performed since the completion of the Imaging and Medical Beamline (IMBL) at the Australian Synchrotron.

Additive Manufacturing

Additive manufacturing (AM), or 3D printing, makes it possible to fabricate parts with highly complex geometries that could not be produced using traditional manufacturing methods. It also enables rapid prototyping, thus streamlining the process of design optimisation. CSIRO is including AM in the initiative to facilitate development of an Australian titanium manufacturing industry. X-ray tomography is being used in this environment to gain a fundamental understanding of the process. Due to the highly absorbing nature of titanium for low energy X-rays, this work has been carried out at the IMBL of the Australian Synchrotron.

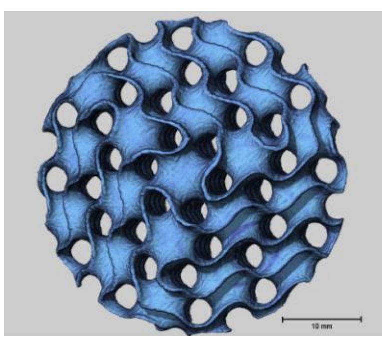


Figure 1: Tomographic image of a titanium gyroid produced via additive manufacturing.

A range of simple and complex shapes have been considered in order to examine relationships between design, build parameters, product integrity and surface condition. Figure 1 shows the relatively smooth surface finish associated with broadly curved structures. A more typical AM surface is shown in the hand skeleton model in Figure 2. The morphology of the original powder is retained to some extent and the layering of the build is more apparent. Slicing through the volume also reveals that some of the powder has been entrained within a hole in the model. Observations such as these, in combination with findings from other characterisation techniques are helping to produce a better picture of AM with a view to improving building algorithms and process modelling.

Nicola Scarlett
CSIRO Mineral Resources Flagship

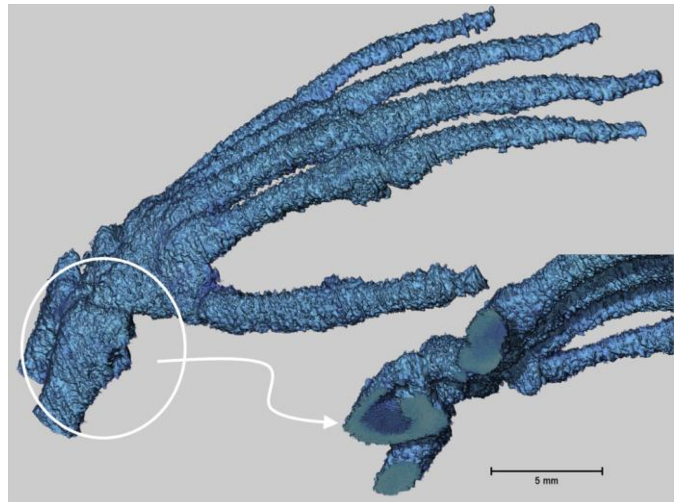


Figure 2: Tomographic images of a titanium hand skeleton showing the rough surface finish of a complex shape and entrained powder within a hole in the model.

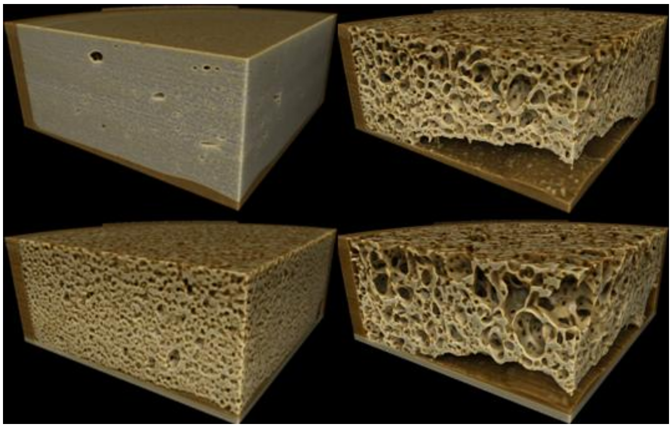
Synchrotron Micro-CT of Rising and Baking Dough

We recently undertook a micro-CT study on the IMBL at the Australian Synchrotron to investigate the effect of different salt additives (NaCl, KCl, NaBr, and no salt added) on the 3D structure of dough made from high and low protein flour. The properties of wheat flour vary depending on the variety and region where it is grown, and this, together with the salt composition, can have a significant effect on the suitability of different flours for wheat products such as bread, pasta and breakfast cereals.

A specially designed oven was used to prove and bake 'mini loaves' about 25 mm in diameter, while enabling CT scans to be performed *in situ* at 5 minute intervals. The powerful X-ray beam meant that each scan took only 16 seconds and provided high-resolution 3D 'snapshots' of the dough at each time-point. With 29 time-points for each of 16 samples this resulted in 464 datasets for reconstruction and analysis; a real Big Data challenge! We handled this using tools on the MASSIVE supercomputer cluster. We used X-TRACT, a CSIRO-developed software package, for rapid tomographic reconstruction of our datasets – this transforms the raw datasets into 3D 'images' of the doughs. These were then analysed using the commercial package Avizo to determine the evolving structural parameters of the dough.

Through the analysis we were able to compare the differences in structural parameters such as porosity and wall-thickness over time for each of the different samples. This revealed that as expected the high-protein flour produced a better-rising more porous dough. It also showed that for both types of flour NaCl and KCl had only a modest effect on dough structure compared to no salt, but NaBr resulted in a much denser, less porous dough.

Sheridan Mayo
CSIRO Manufacturing Flagship



Time sequence of dough rising (3D rendered view)

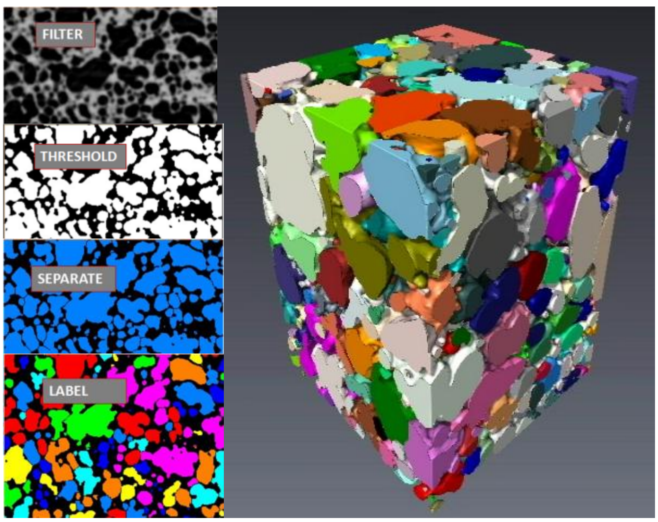


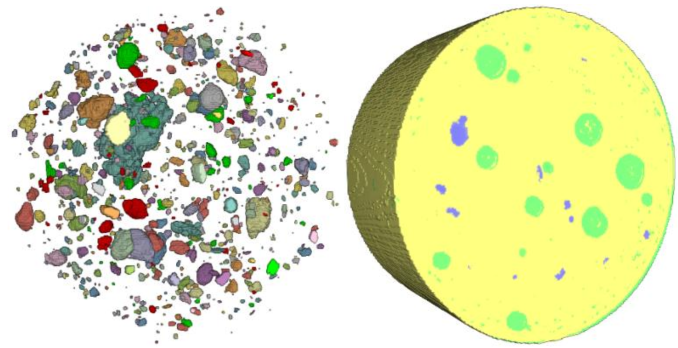
Image analysis steps used to segment and analyse dough samples (left) and 3D rendered view of segmented dataset (right).

DCM characterisation of 3D microstructures

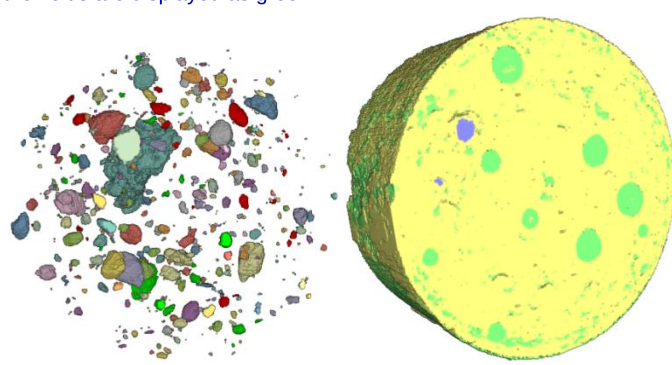
A data-constrained modelling (DCM) approach has been developed at CSIRO which enables 3D characterization of compositional material phase distributions using quantitative, multi-energy synchrotron X-ray CT data. DCM employs CT datasets collected using two or more X-ray energies to extract additional information regarding the composition of each volume element in the 3D reconstruction (such as phase/pore fractions), and can be used to address issues such as phase mixing and segmentation. Using this information, material properties at the macroscopic length scale can be modelled quantitatively.

Prevention of corrosion of metal parts is important for airplanes. The parts are coated with a paint primer with corrosion inhibitor and filler particles. The performance of such aerospace paint primer is dictated by the microscopic distributions of such corrosion inhibitor particles. The CSIRO Manufacturing Flagship has been collaborating with Shanghai Synchrotron Radiation Facility (SSRF), Chinese Academy of Sciences, Manchester University and Boeing. A laboratory paint primer material sample has been used for this investigation. The sample contains 3 vol.% of strontium chromate particles and 3 vol.% of rutile particles in an epoxy resin matrix. The sample is cast as a 1.5 mm diameter cylinder. The 3D distribution of strontium chromate particles and other material phases have been characterized with the CSIRO DCM software using X-ray CT data taken at 15 keV and 17 keV beam energies at SSRF. The 3D microstructure has been characterized before and after the sample being immersed in a mild acid simulating the environment exposure. Some hidden leaching paths for the strontium chromate particles in the epoxy resin have been observed.

Sam Yang
CSIRO Manufacturing Flagship



Left) 3D distribution of strontium chromate particles before leaching in a mild acid solution. Different colours represent different strontium chromate particle clusters. Right) strontium chromate particles displayed as blue, the epoxy resin matrix is displayed as yellow, and the voids are displayed as green.



Left) 3D distribution of strontium chromate particles after leaching in a mild acid solution for 20 minutes. Different colours represent different strontium chromate particle clusters. Right) strontium chromate particles displayed as blue, the epoxy resin matrix is displayed as yellow, and the voids are displayed as green.

AXAA Membership

All registered participants of the AXAA-2014 conference are automatically granted AXAA membership for 3 years. Alternatively, new memberships can be obtained free of charge, by making an application to the National Council. Candidates should provide their CV and a short statement about how they intend to contribute to the organisation. Please send these to the National Council Secretary Natasha Wright (see AXAA contacts) if you would like to apply.

AXAA Website and Contacts

AXAA has a new-look website! Please come and have a look at: www.axaa.org

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Please email contributions for Issue 1 of the 2015 AXAA Newsletter to Mark Styles by Friday the 20th of March. Any comments or feedback about the Newsletter are welcome.

Upcoming Events

2nd Asia-Oceania Conference on Neutron Scattering

19-23 July 2015
Novotel Sydney Manly Pacific

This conference is aimed at those who use (or would like to use) neutron scattering facilities in the Asia-Oceania region. AOCNS provides an opportunity for researchers to share and discuss their findings as well as the latest developments in neutron scattering techniques. Abstract submissions close on 10 March 2015. More information can be found on the conference website.

Computational and Simulation Sciences and eResearch, Annual Conference 2015

13 February 2015
Melbourne Convention and Exhibition Centre

CSIRO is holding a one-day multidisciplinary workshop on advanced 3D quantitative microstructure characterisation and microstructure-based materials properties modelling. For more information, please visit the website, or contact sam.yang@csiro.au.

X-ray Materials Analysis Internet Courses – Wavelength Dispersive XRF and Powder XRD

Mode of Instruction for XRF and XRD Courses

These internet-delivered courses provide XRF and XRD analysts, particularly those new to x-ray analysis, with on-site and/or at-home instruction on the underlying principles and principal analytical methods. Features of the two courses -

- Start at any time
- Self-paced instruction to accommodate the needs of busy people
- Study materials transmitted as e-mail attachments in the form of a set of modules; with an assignment being set for each module.
- Feedback on the assignments provides excellent mentoring.

The courses have a substantial cohort of international and local participants, and are being used by companies as vehicles for in-house XRF and XRD training.

Courses Director: Dr Brian O'Connor

Internet XRF Course: Series 8, 2015

The Internet XRF Course comprises modules on - XRF Overview; X-ray Excitation of the Specimen; X-ray Dispersion and Detection; XRF Data Measurement; Data Analysis Basics; Methods of Quantitative Analysis; Absorption-Enhancement Corrections; Specimen Preparation; Major Component Analysis Using Fusion Buttons; Trace Element Analysis Using Powders; and Analysis of Sub-Milligram Environmental Samples.

Course fee: $2,850 including GST

Internet XRD Course: Series 3, 2014

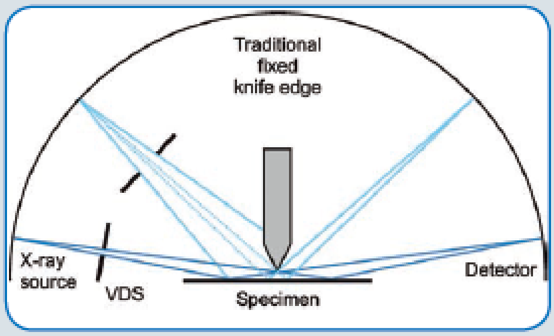
The internet XRD Course comprises modules on - XRD Overview; Essential XRD Fundamentals; XRD Measurement Strategies (I); XRD Measurement Strategies (II); Search/Match Identification Analysis (I); Search/Match Identification Analysis (II); Case Studies in Search/Match identification Analysis; Phase Composition Analysis Using Line Intensities; and Introduction to Advanced Methods (indexing, Rietveld phase analysis, structure solution, etc.)

Course fee: $2,850 including GST

Further Information and Enrolment Procedure:

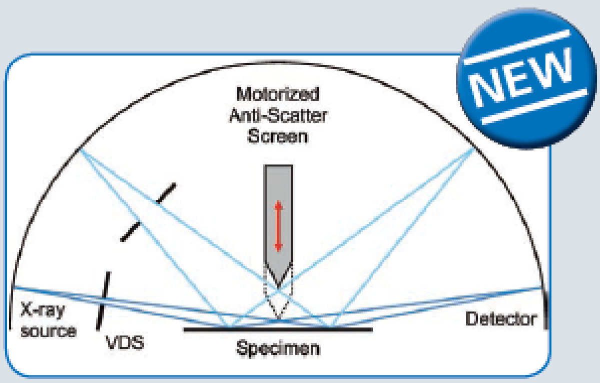
brian_oconnor@iprimus.com.au (Tel 08 9291 7067)

Company News and Advertising:



VDS: Variable Divergence Slit

A traditional fixed knife edge allows suppression of parasitic scatter at low angles 2θ, but also crops the beam at higher angles 2θ. This affects both the Bragg-Brentano and the parallel beam geometry, and is particularly pronounced for the Bragg-Brentano geometry if using variable slits (constant illuminated sample length). As a result, intensities are inceasingly cropped as 2θ increases.



With the unique Motorized Anti-Scatter Screen, the knife edge position is automatically optimized to achieve maximum suppression of parasitic scatter while not cropping the beam at any angles 2θ. As a result, highest-quality data with accurate intensities and minimum instrument background will be obtained.

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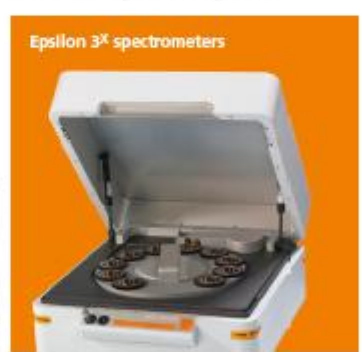

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PANalytical launches new compact benchtop XRF system for pharmaceutical catalysts analysis

PANalytical has just launched a new Epsilon 1 XRF (X-ray fluorescence) analyzer at this year's meeting of the American Association of Pharmaceutical Sciences (AAPS), November 2-6, in San Diego, California. Designed as the most powerful benchtop spectrometer in its class, the new Epsilon 1 provides a compact and cost-effective 'out-of-the-box' pre-calibrated solution for the analysis of residual catalysts in pharmaceutical materials.

Metal catalysts are used as part of the pharmaceutical development and manufacturing processes. Post-process efforts are made to remove the undesired catalyst from the material but there are often residues remaining. These residues require comprehensive quantification to meet stringent industry permissible limits, such as those specified in pharmacopoeias including the United States pharmacopoeia and the European pharmacopoeia. The Epsilon 1 will set the new benchmark for analytical performance and ease of use in this class of industry-dedicated XRF instrumentation.

"The need for pharmaceutical catalyst analysis is an industry-wide concern," commented Pieter de Groot, PANalytical Corporate Marketing Director. "The Epsilon 1 offers a pre-calibrated total solution package, including installation qualification and operation qualification procedures. Designed with simplicity in mind, for the first time PANalytical's high standards of analysis are now available in this class of desktop instruments."

For more information on PANalytical's innovative range of X-ray based analytical techniques and additive analysis solutions visit the PANalytical website at www.panalytical.com or contact your local PANalytical representative.

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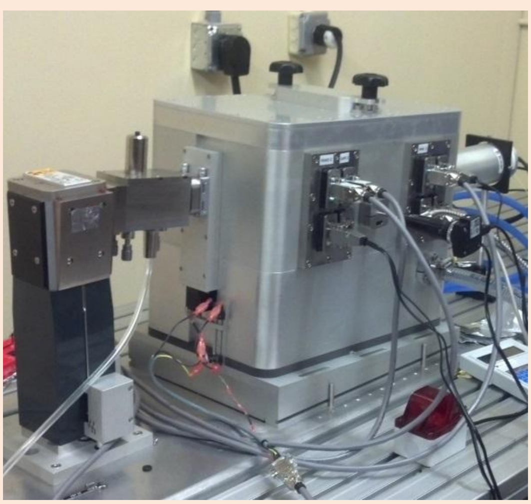

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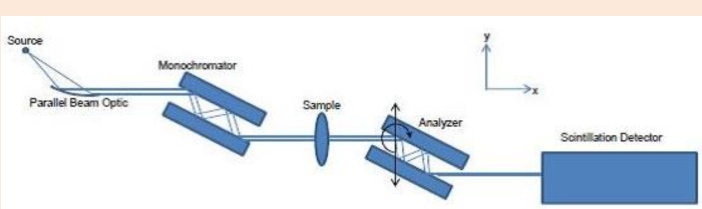
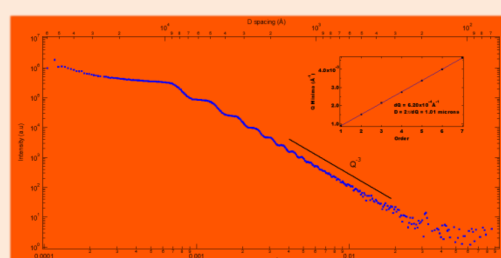
For isotropic samples, Choice of detector, Flexible sample holding, All -Vacuum path. Q range ~ 0.0001 to 0.1 A^{-1} (Features 1 nm to 2.5 μm).





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