Australian X-ray Analytical Association
Newsletter Issue 1 2018

President's Address

Dear AXAA Members and Friends,

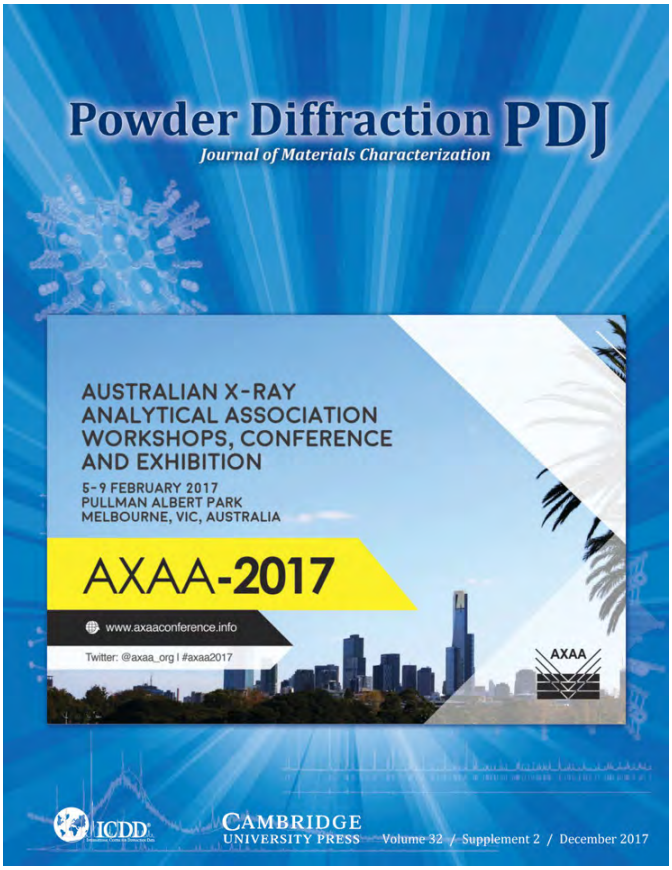
It's hard to believe that more than 12 months has passed since AXAA-2017, and already the National Council are ramping up efforts for the organisation of AXAA-2020. As mentioned in our last two Newsletters, one of the major considerations for future events is cost, and we are looking to scale down the exhibition portion of the event a little in conjunction with a more modest venue to reduce registration fees. We are also considering changes to the conference format, especially regarding the Schools/Workshop component of the programme. We welcome your thoughts on these matters, including possible conference venues, and invite you to contact the National Council with these (see contact details at the end of the Newsletter). We also welcome nominations from the AXAA community for AXAA-2020 Conference Committee members, so if you or someone you know is interested in helping to shape our next conference, please get in touch.

Regarding AXAA-2017, the special AXAA-2017 Conference Proceedings issue of Powder Diffraction journal has recently been published and is available online. The National Council are pleased with the high quality of the publications, and will be endeavouring to make this exciting opportunity to publish your work in this respected materials characterisation journal available again for AXAA-2020. It is worth noting that, in addition to the AXAA-2014 and AXAA-2017 Proceedings, the past three Proceedings of the European Powder Diffraction Conference (EPDIC) have been published in Powder Diffraction, EPDIC being one of the world's leading X-ray conferences (along with AXAA of course!).

Finally, the latter part of 2018 will see AXAA Student Seminar events being held across Australia. These events are an excellent opportunity for students to present their work in a friendly, collegial atmosphere and meet others in the X-ray and neutron scattering community. Prizes will be awarded to the best presentations. Stay tuned for details about these events in your state.

Nathan Webster
AXAA President

Click here to access proceedings online.



Sample Preparation and XRF Analyses (Cement)

Introduction

In cement, as well as other industries, sample preparation is a key step to ensure that XRF analyses are truly effective. To achieve accurate and reliable XRF results that comply with the official standard test methods for analysis of cement and raw materials, it is essential that good quality samples are delivered to the spectrometer for measurement.

Crushing and grinding the sample

The first important point is the sampling. The size, and nature of material chosen should be enough to represent the sample mass of interest. For fine-grained samples, this may be only 200 - 500 g, but for coarse grained samples it is not unusual to begin with 10 - 15 kg.

If the sample grain size is bigger than 12 mm, the whole sample is first crushed in a jaw crusher. During this step, it is essential that the time the material is in the crusher is reduced to a minimum to avoid excessive contamination or alteration of the sample. A jaw crusher, such as the BOYD Elite, is the perfect tool for that. Featuring top and bottom moving jaws, the crusher delivers the maximum amount of crushing, reducing the sample by 35 times its original size in only one pass. For practical reasons, it is also important that the jaws can be readily cleaned.

The crushed sample is then split as many times as necessary to obtain a portion of approximately 250 g. Taking a representative split from a large sample is not as simple as many people believe. Rolling on a mat, multiple grabs, or riffle splitting do not produce accurate results. A classic study conducted at Bradford University demonstrated that a rotating sample divider (RSD) provided the best results. Table 1 shows the standard deviation obtained using different dividing methods.

A combination of the BOYD Elite and RSD (Figure 1), has been designed to work together for a one step crushing and splitting process to minimise operator intervention and achieve perfect division every time

This portion of 250 g is then pulverised to a powder, fine enough to pass through a 100 µm opening size sieve. Theoretically the finer the better, but excessive pulverising can cause serious contamination.

Table 1.

Dividing methods samples %	***Standard deviation***
Cone and quartering	6.810
Scoop sampling	5.140
Riffle splitting	1.010
Table sampling	2.090
Rotating splitting	0.125
Random variation	0.076

Figure 1. BOYD Elite RSD combo.

Figure 2. Pheonix 2.

Source of contamination through grinding

There are two major sources of contamination during the crushing and pulverising process: (1) the previously pulverised sample (cross contamination); and (2) the grinding container.

1. To minimise contamination from the previous sample, the jaws and the grinding head are cleaned after each run by using a portion of the next sample to grind that is discarded after use. Alternatively, a run of pure silica can be used, followed by a quick cleaning to avoid contamination by SiO_2.
2. This source cannot be eliminated, but the appropriate choice of grinding containers can reduce the contamination to elements that are irrelevant. For example: a tungsten carbide head would not be used when W and Co are important to the analyst. Data can be provided on the contamination expected from the various grinding containers.

It is very important at this stage to treat all samples the same way to keep the systematic error to the same level. This makes the results more reproducible, precise, and comparable. Always use the same grinding container, amount of sample, and grinding time.

Method of specimen preparation

Two techniques are used for the preparation of the specimen: (1) confection of compressed powder tablets; and (2) preparation of beads or glass disks by fusion.

1. For the pressed tablet technique, the sample must be ground as finely as possible (to below 75 µm), attempting to limit the particle size distribution, to obtain a fine powder. This powder is pressed with or without adjuvants to obtain a pressed tablet or briquet.
2. The fusion technique consists of the reaction at high temperature between a molten borate flux and the specimen to form a homogeneous molten glass. For XRF work, the molten glass is cooled without crystallising to yield an amorphous homogeneous solid glass disc, with a polyborate glass structure.

The perfect specimen

For XRF analysis, the best preparation method to get a fully homogenous and representative specimen is by the fusion technique. The flat glass disc obtained by fusing the powder samples provides more accurate analytical results compared to the pressed pellet. This technique overcomes all the problems associated with the variation in mineral grains and other mineralogical effects.

In the cement industry, fusion is now often used as the reference method and replaces the traditional wet chemistry techniques for major elements. Using this preparation method, it is possible to build a robust calibration curve covering the whole range of concentrations for the elements of interest in the cement industry, including the raw materials.

Perfect bead repeatability

In order to produce repeatable fused glass discs, it is essential to have perfect control of the fusion conditions. Modern fusion machines are considerably more sophisticated, and the process of the preparation of the fused glass disc is done fully automatically with a perfect control of the conditions and in a safe environment. Current systems use gas or electrical heating; this is the case with Phoenix gas fusion (Figure 2) and the xrFuse electric fusion (Figure 3) machines produced by XRF Scientific.

Both machines allow the operators to load the crucibles containing the sample/flux mixture, start the programme, and return after 12 min. to collect the glass discs, all conducted in a safe, enclosed environment.

Comparison between gas and electrical fusion machines

There is a large difference between a gas and an electric fusion machine, but the most important thing is that they both reach the same performance level and meet the requirements for the actual norms. A good way to evaluate this is using the Rapid Test Method (RTM), as proposed in the ASTM C114-15 norm. The ASTM C-114 norm covers the chemical analysis of hydraulic cement, describing all the test methods used for chemical composition analysis of cement. XRF is mentioned as an example of RTMs. The method needs to be validated with acceptable certified reference materials (CRMs). The standards used for this test are the reference materials 601 B, containing 15 different standards from the Japan Cement Association.

Experimental

For this study, all samples were calcined at 950°C for 2 hours before the fusion. Two sets of glass discs were prepared on two different days using both the Phoenix and the xrFuse fusion machines.

For all the preparations, 1.250 g of sample were mixed with 10.000 g of flux LT66:LM34 + 0.2LiBr (66% lithium tetraborate/34% lithium metaborate containing 0.2% lithium bromide as non-wetting agent) to produce beads of 40 mm. The fusion programme for the Phoenix includes 200 sec. melting and 250 sec. mixing (swirling) at 1100°C. The fusion Program for the xrFuse includes 250 sec. melting and 350 sec. mixing (rocking) at 1100°C.

The XRF analyses were done on a Bruker WD-XRF 58 Tiger spectrometer. This device is equipped with a Rh tube of 4.0 kW, multi-crystals, flow counter (Ar/CH4), and scintillation counter as detectors.

The analytical conditions for the measurements were developed specifically for cement, raw mix, and clinker analysis, and are available as a complete XRF calibration package from Bruker.

All reference materials were duplicated and analysed as unknowns. Table 2 shows all the results and limits in terms of precision (difference between duplicates) and accuracy (difference of the average of duplicates and RM certificate value) for all the elements required to be analysed by XRF.

The results obtained confirmed that both the Phoenix 2 and xrFuse 2 fusion machines comply with the ASTM C-114 requirements and that the choice of either system will depend on other reasons.

Source of error through fusion

There are three major sources of analytical error due to the fusion: (1) incorrect fusion temperature; (2) insufficient homogenisation; and (3) a deformed mould surface.

1. Silicon dioxide (SiO_2), calcium oxide (CaO), sodium oxide (Na_2O), and sulphur trioxide (SO_3) are the most affected by temperature. From experiments conducted, a 50°C variation could cause a 0.5% change in result for CaO in cement.
2. An insufficient homogenisation causes a change in the result for SiO_2 and Cao.
3. If the glass bead is not perfectly flat, this variation causes a change in the result. From experiments conducted, a 0.1 mm variation can cause up to 1 % relative error in the result.

Conclusion

The accuracy and repeatability of XRF analysis depends on the sample preparation. Never neglect this key step. Always check the method repeatability and establish a control chart for the weekly control.

References

• ALLAN, T., and KHAN, A.A., "Critical Evaluation of Powder Sample Procedures", The Chemical Engineer (May 1979), pp. 108 - 112.
• WILLIS, J,. FEATHER, C., and TURNER, K., "Guidelines for XRF Analysis (James Willis Consultants; 2014).

Frederic Davidts and Pascal Deprez - XRF Scientific.

Table 2.

	Norm C114-15		xrFuse 2		Phoenix	
	Precision	Accuracy	Precision	Accuracy	Precision	Accuracy
Na_2O	0.03	0.05	0.01	0.03	0.00	0.03
MgO	0.16	0.20	0.01	0.17	0.01	0.15
Al_2O_3	0.20	0.20	0.13	0.12	0.13	0.12
SiO_2	0.16	0.20	0.15	0.15	0.12	0.16
P_2O_5	0.03	0.03	0.00	0.01	0.00	0.01
SO_3	0.10	0.10	0.05	0.06	0.01	0.05
K_2O	0.03	0.05	0.01	0.03	0.00	0.03
CaO	0.20	0.30	0.18	0.24	0.12	0.19
TiO_2	0.02	0.03	0.00	0.01	0.00	0.01
Cr_2O_3	/	/	0.00	0.01	0.00	0.01
Fe_2O_3	0.10	0.10	0.04	0.05	0.01	0.04
ZnO	0.03	0.03	0.00	0.01	0.00	0.01
SrO	/	/	0.00	0.00	0.00	0.00
Mn_2O_3	0.03	0.03	0.00	0.01	0.00	0.01

Hybrid Pixel Counting Detectors

Hybrid Pixel Counting (HPC) detectors are next generation semiconductor detectors that directly measure all photons generated by your samples. They also enable collection of far more detailed information compared to more conventional 1D data sets. Detectors like the HyPix range from Rigaku also offer the ability to measure in 0D, 1D and 2D modes with the click of a button, i.e. no hardware adjustments.

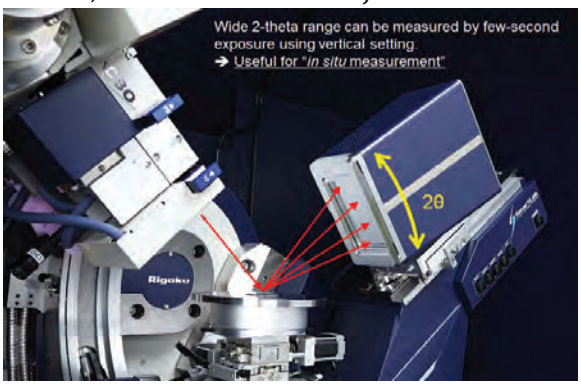


Figure 1. HyPix HPC detector on Rigaku Smartlab XRD.

The ability to switch seamlessly between 0D, 1D and 2D modes offers users the ultimate flexibility. Using instruments like the SmartLab diffractometer, operators can easily switch between various measurement modes such as conventional powder, SAXS, microdiffraction etc. In powder diffraction, uses can use the HyPix detector in 2D mode to carry out very rapid scans and then switch to 0D mode to scan specific areas for optimal resolution. That said, the HyPix 3000 on the SmartLab diffractometer is suitable for performing HRXRD experiements in 2D mode based on its low FWHM.

HPC detectors have also become more common place being coupled with higher flux X-ray sources and computers that are better able to cope with larger data sets and more complex analyses.

These detectors are the benchmark for laboratory systems with the technology even extending into synchrotron applications. Their operation and advantages are discussed herein.

Operation

Most modern 2D detectors to date have been of the CCD (Charge-Coupled Device) or CMOS (Complementary Metal-Oxide Semiconductor) types, also known as Integrating Detectors. These measure photons indirectly by using a scintillator to convert X-ray photons to visible light. The light is then transferred to the CMOS or CCD chip via a fibre optics taper or stub.

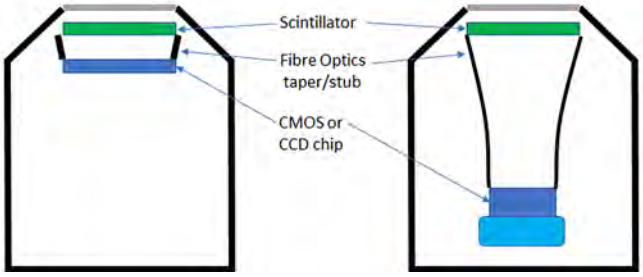


Figure 2. CMOS and CCD 2D detectors.

HPC detectors on the other hand directly convert photons into electric charge and count each one immediately. Furthermore, they are event driven, meaning that they simply count each individual photon and do not accumulate any noise.They also incorporate energy discrimination to prevent any possible charge sharing between adjacent pixels, ensuring maximum accuracy.

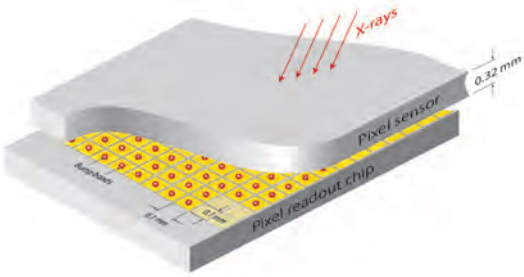


Figure 3. Schematic of a HPC detector.

Advantages of HPC Detectors

With 1D Silicon Strip Detectors (SSD), the detector is restricted in that it can only detect diffracted X-rays in a single plane. For polycrystalline materials, X-rays are diffracted in cones. 2D detectors overcome this problem and are only limited by their size and position which allows them to detect the entire or significant portions of the Debye-Scherrer diffraction cones simultaneously.

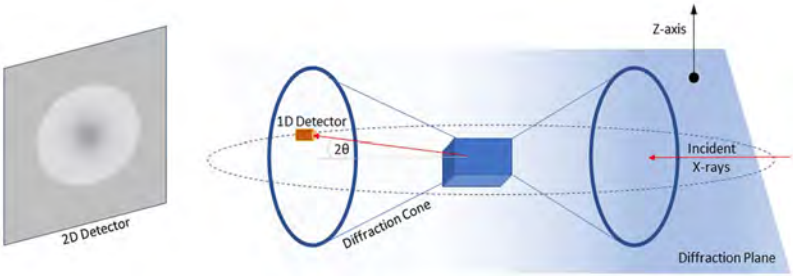


Figure 4. 1D vs 2D detectors.

For powder diffraction is is widely accepted that fine particles randomly oriented will produce the best data. These will generate a pattern like the 30µm silica pattern (figure 5 (top)).

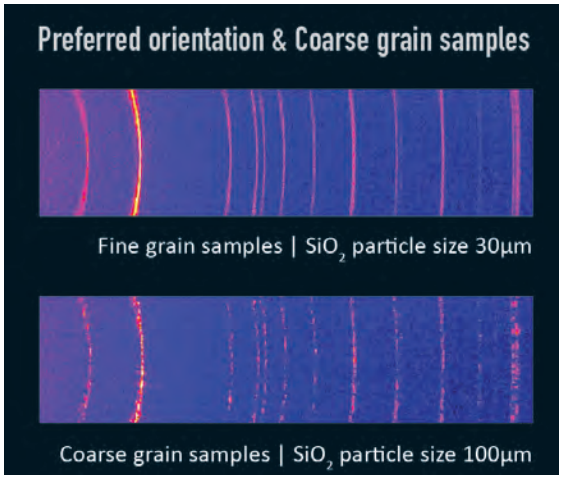


Figure 5. HPC detectors minimise the effect of grain size and orientation

However, this is not always possible, in which case 2D detectors like the Rigaku HyPix range are better able to deal better with a wider range of samples. Samples with coarser particles sizes, with higher degress of orientation or texture can produce broken or spotty Debye-Scherrer rings (figure 5 (bottom)) which can be missed by 1D detectors. 2D detectors look at the whole or significant parts of the ring and integrate intensity data across them reducing the effects of these phenomena.

Also, with larger active detector areas, small pixel sizes (and excellent spatial resolution), higher count rates, fast readout speed, no point spread and zero dead time, these detectors are also able to collect much faster than 1D detectors, with high levels of accuracy. So on top of collecting more accurate data, you can increase your sample throughput, thus fast tracking your research.

From an instrument design perspective, they also have the advantage that they can be air-cooled and hence do not require any water cooling or gas exchange as may be the case for other technology of detectors.

Application Advantages of HPC Detectors

In practice, HPC detectors will benefit users in specific applications such as:

1. Phase identification – Improved intensity statistics are generated for a given 2θ range which also aids in quantitative analysis especially with highly textured or large-grained samples
2. Crystallinity – Faster and more accurate analysis
3. Thin Film – Better able to detect various phases simultaneously for films which may be mixtures of single crystal, polycrystalline and highly textured
4. Texture – Very fast measurements are possible. This allows specific areas of interest to be readily located for pole figure analysis
5. Stress – By measuring larger segments of the Debye ring, higher accuracy data can be collected at higher speeds.
6. SAXS – Data can be collected more quickly and single exposure generates data to be able to map the the sample's structure
7. Microdiffraction – Data captures with high speed and precision, again integrating over portions of the Debye-Scherrer cone minimising the effect of spotty, textured or weakly diffracting rings

Summary

HPC detectors are the the optimal hardware partner for researchers involved in diffraction experiments. By combining ultimate flexibility, with data collection speed and accuracy, they partner with other components like high flux X-ray sources to generate high quality information with next to zero noise. In doing so, you have the ideal configuration to analyse your materials quickly and accurately, providing the next best solution to synchrotrons.

Cameron Chai, AXT.

Upcoming XRF Workshops

- XRF in the Workplace – Sydney, 16th – 20th July 2018
- SuperQ Software Hands On Training – Perth, 1st – 3rd May 2018

The XRF in the Workplace course is a practically oriented training course that is suitable for all users of XRF (not just Malvern Panalytical users). Contact Grace (grace.perrone@panalytical.com) for further details and/or to register for any of the above courses.

Combined Analysis Using X-ray and Neutron Scattering

The 9th session of this workshop will take place in Caen, France from July 2nd to July 6th, 2018.

The training will cover many aspects of "Combined Analysis" by X-ray and neutron scattering, ranging from fundamental requirements to technically relevant industrial and academic applications:

Diffraction technique - an overview, crystallography, Texture Analysis, Residual Stress Analysis, Rietveld analysis, Reflectivity analysis, Phase analysis, Phase and line broadening analysis, The combined solution, XRD and XRF combined analysis, Electron Microscopy, Using MAUD software.

For more information about MAUD software, please visit the website.

If you are interested in this workshop, please email TFS INEL with the subject << MAUD workshop >> at: eric.berthier@thermofisher.com

Registration deadline: **June 29th 2018.**
Attendees are limited to 30. Student rates available.

IMA2018

13 - 17 August 2018
Melbourne, Australia

The International Mineralogical Association is an umbrella organisation uniting professional associations from 38 countries, and its quadrennial meeting is the largest global forum for making contacts and exchanging information with mineral scientists from around the globe. The 22nd meeting in Melbourne, Australia, will be the first such meeting held in Australia and only the second in the southern hemisphere. The meeting will be hosted by the Geological Society of Australia, and held in the Melbourne Convention and Exhibition Centre, between 13–17 of August, 2018.

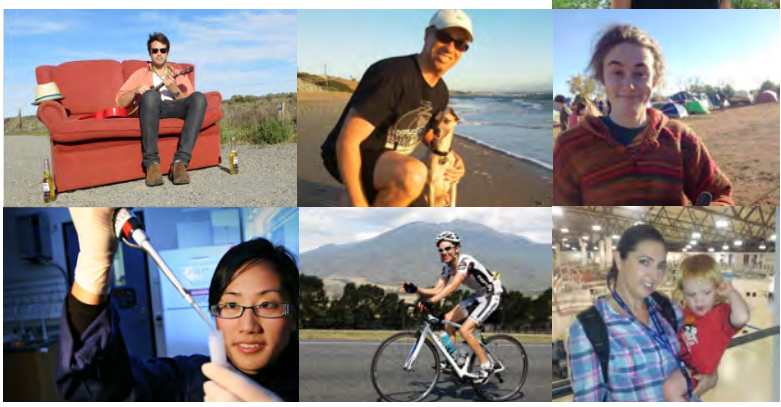
Website: https://www.ima2018.com/

XXII MEETING OF THE INTERNATIONAL MINERALOGICAL ASSOCIATION
13-17 AUGUST 2018 | MELBOURNE

A Day in the Life of an X-ray / Neutron Scientist

In 2016, AXAA started a blog series as a new way to get to know our members. Our 'Day in the Life' posts take a peek behind the scenes of different workplaces to find out the fun bits, the challenging bits, and why you do what you do.

We are currently seeking posts so if you'd like to contribute, or know someone who might be interested, please contact National Council Communications Editor Jessica Hamilton.



W: www.axaa.org/a-day-in-the-life.html

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Please visit our website, www.axaa.org, for further information, or follow us on Twitter @axaa_org.

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AXAA Membership

All registered participants of the AXAA-2017 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 Mark Styles.

AXAA Resource Centre

There are a range of resources available on the AXAA website, including video recordings of the two Public Lectures at AXAA-2017, tips for Rietveld Analysis, Clay Analysis, XRF tips, and more.

We welcome further contributions to our Resource Centre.

Next AXAA Newsletter

The next issue of the AXAA Newsletter will be distributed in August 2018. Please feel free to send contributions for the newsletter to Jessica Hamilton at any time. Any comments or feedback about the Newsletter are welcome.

Malvern Panalytical

TIPS: PROTECT YOUR PLANT BEFORE RELEASING INDUSTRIAL WASTE

In the last two years, companies in our neighbouring countries have been fined for disposing used industrial waste tainted with chemicals and heavy metals into the waters. In Vietnam, fish carcasses washed up on the beaches of Hà Tĩnh, many fishermen lost their livelihoods and needless to mention about water safety. While these companies have received fines, it is fair to say that waste management affects the public and ecosystem.

Malvern Panalytical supports companies with analytical solutions which ensure socially responsible disposal of used industrial water. Here are some tips:

- **Prevent heavy metals from getting into your process:** Careful in-bound QC checking for heavy metals using our latest **Epsilon 4** benchtop EDXRF.
- **Monitor factories' effluent streams:** On-line monitoring of your liquids for heavy metals before disposing into the environment using **Epsilon Xflow**. Hence enabling immediate counteractions to prevent environmental damage.
- **Treating water with contaminants:** Add the right dosage of coagulants by carefully monitoring the zeta potential of particles during your flocculation and sedimentation process. See how our **Zetasizer WT** was used to treat ash from a fire in Canada's waters. Click here

Read more **www.panalytical.com/Xflow**

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- Check before disposing into the environment
- Treat water of contaminants

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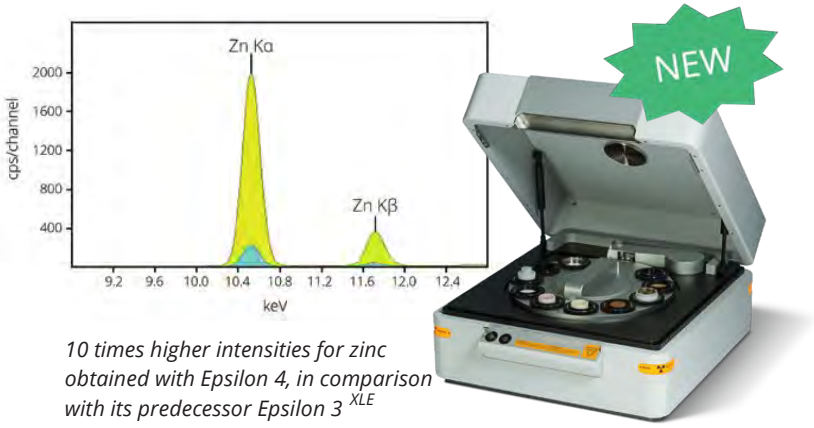

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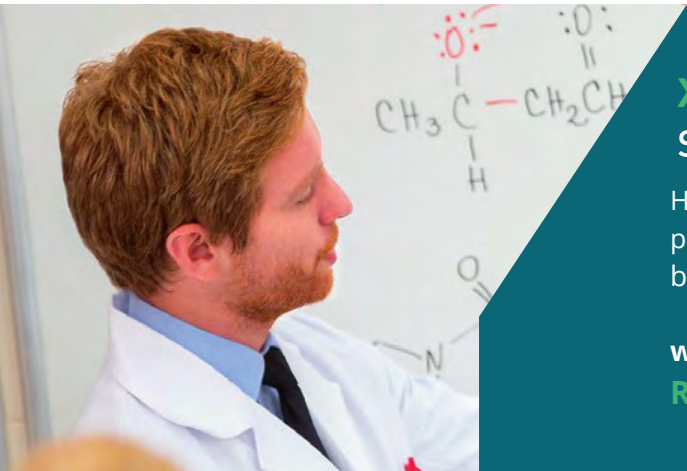

XRF at the workplace training

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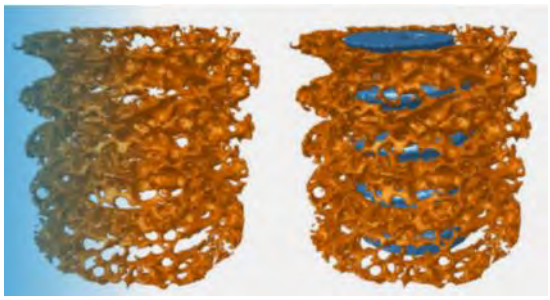
Scanning Trajectories and Reconstruction Algorithms



In this microCT article, we focus on scanning trajectories and the associated topic of reconstruction algorithms. The differences between circular and helical scanning are highlighted. You will see how the quality of reconstruction and reconstruction speed are affected by using different scanning trajectories and reconstruction algorithms.

To get suitable tomographical reconstruction data, movement of the object or source-detector pair can be done in different ways. In the simplest case, the object or source-detector pair is just rotated during the scan, creating a circular trajectory for acquisition geometry. Using more complicated movements, with simultaneous or sequential rotation and translation, creates non-circular acquisition geometries. With special reconstruction algorithms appropriate for a particular acquisition geometry, one can significantly improve the accuracy of the reconstructed results and suppress different artifacts, which may appear on reconstructions from circular scanning. In the SKYSCAN system range, solutions for both circular and non-circular (helical) scanning trajectories exist.

Circular Trajectories

In most situations, the simplest trajectory with fast, but approximate, reconstruction is a circular one. With a relatively small opening angle of the cone beam along the rotation axis, circular trajectories produce reconstruction results very close to the internal structure of scanned objects. The need to scan long objects, which cannot fit in one vertical camera field of view, or to increase the scanning speed by using a short source-detector distance with a big opening angle of the beam, may increase differences between the reconstructed results and the real object structure.

Filtered Back-Projection, the Feldkamp Algorithm

The most popular reconstruction algorithm for cone-beam tomographical systems is a filtered back-projection algorithm originally developed by Feldkamp, Davis and Kress, published in 1984 and later named according to the first letters of the developers' names as an FDK algorithm. NRecon utilizes the FDK algorithm as a standard and performs such reconstruction using CPU with multithreading (NReconServer engine) or acceleration with GPU on graphics cards (GPUReconServer engine). Typically, graphics cards will outperform CPU as can be seen from the table with reconstruction speeds on the website.

Hierarchical Reconstruction

Besides hardware acceleration for reconstruction (by using graphics cards or clusters), another option is to use a more efficient algorithm. The fast, hierarchical back-projection algorithm does precisely this. By dividing the reconstruction volume into smaller ones, requiring fewer projections for reconstruction, significant speed-ups can be achieved. This is especially true for larger datasets, as can be observed from the table. The InstaRecon® together with our partner InstaRecon Inc. For more information on InstaRecon® and interesting further engine for NRecon uses a hierarchical reconstruction algorithm and was developed reading, we kindly refer you to their website.
Additional information can also be found in Method Note MN109 - Reconstruction algorithms and engines for NRecon.

Two vertical slices through a 9-V battery are shown from scans made with the SKYSCAN 1272. For the image on the left, a circular trajectory was used; for the image on the right a helical trajectory was used. An exact reconstruction algorithm was used for the reconstruction of the helical scan data.

Helical Trajectory and Exact Reconstruction

A helical scanning trajectory involves simultaneous object rotation and translation along the rotation axis during data acquisition. In contrast to circular scanning, it respects Tuy's data sufficiency condition, which defines possibilities for exact rather than approximate tomographical reconstruction. This implies that in a number of cases, especially in scanners with wide opening of the X-ray beam, certain reconstruction artefacts, such as blurring at surfaces perpendicular to the rotation axis at high cone angles, can be eliminated (as illustrated in the images of the battery above) and long objects can be investigated without stitching of multiple partial scans. To unlock the full potential of helical scanning, an exact reconstruction is required. Bruker microCT recently added this functionality to the NRecon software together with our partner iTomography™ (https://www.itomography.com).

For more information on this fascinating topic, we refer you to Method Note MN106_Spiral Scanning.

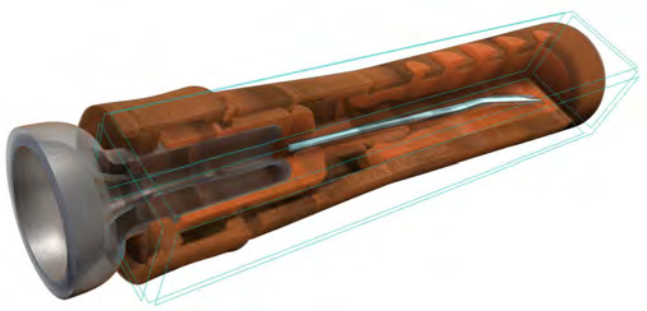


Image of the Month

4 part oversized scan of a syringe, scanned using the SKYSCAN 1275. A quick scan at a voxel size of 10 µm reveals the needle without removing the rubber cap, allowing non-destructive quality control.

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Integration of EIGER2 detector technology into the D8 family of XRD solutions breaks the paradigm that "versatility comes at the cost of performance". In this 15-minute webinar, we discuss the impact that the EIGER2 R 500K has had in the XRD community, with examples across the materials research spectrum. Visit http://bit.ly/EIGER2Web0318 or scan the QR code.

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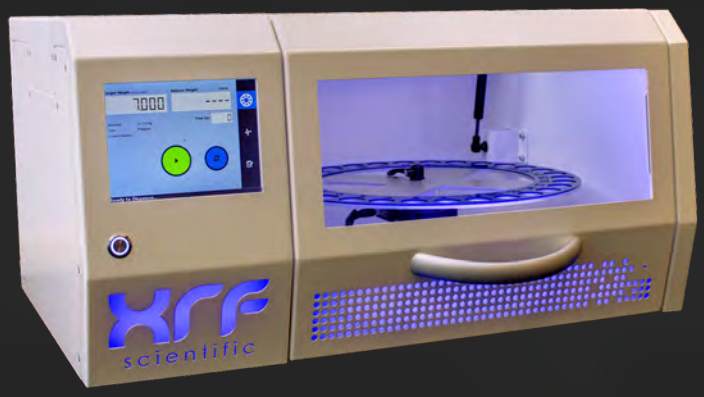

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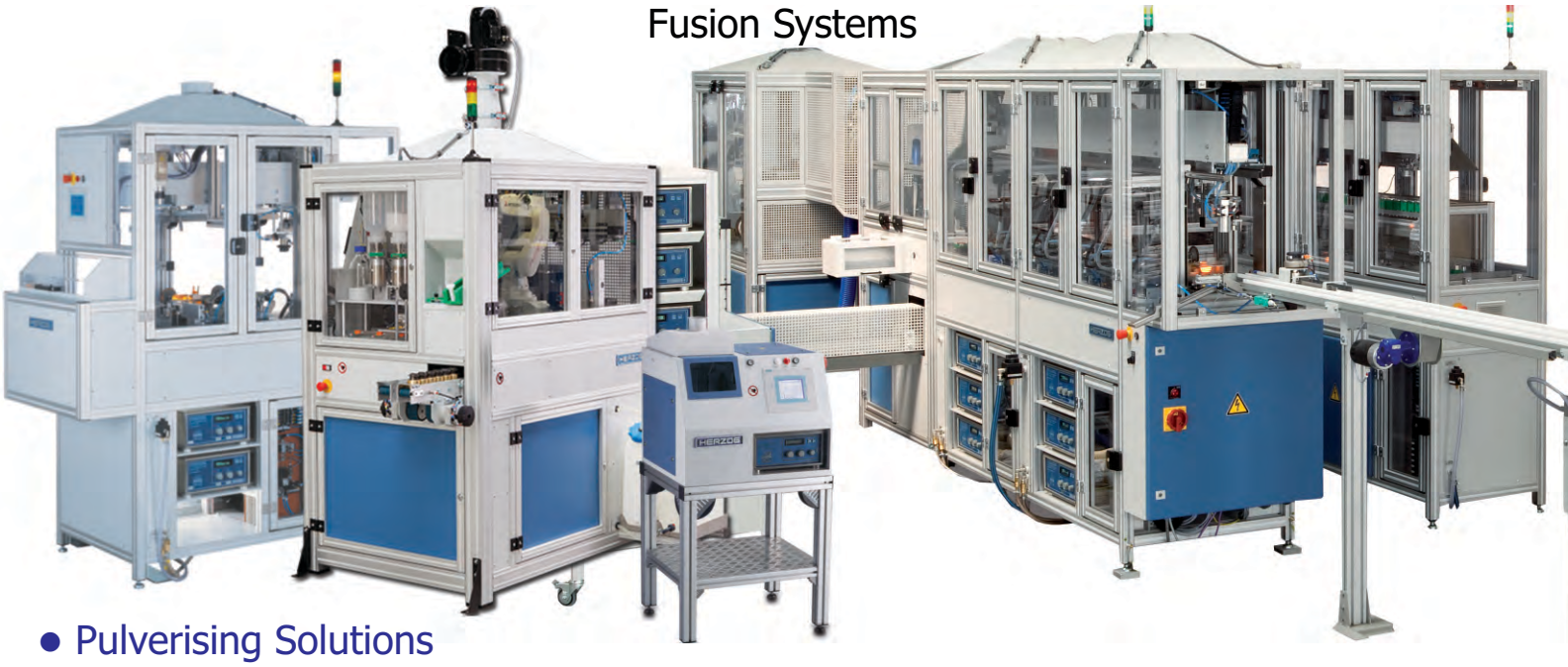

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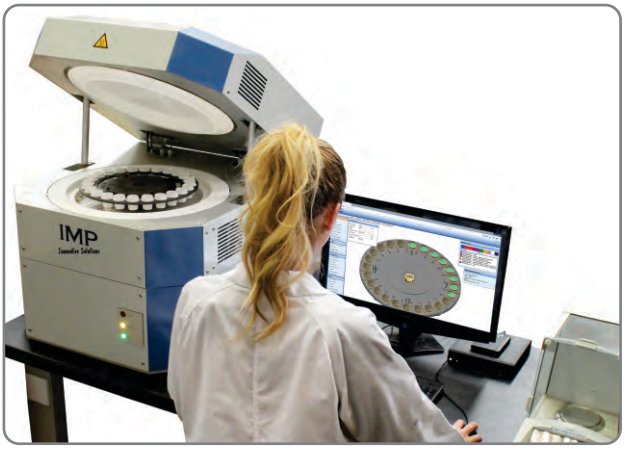

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UWA Researchers Get State-of-the-Art System to Study Protein Crystallography

Sydney, Australia, March 29, 2018 - AXT have recently completed the first Australian installation of a XtaLAB Synergy-S protein X-ray crystallography system from Rigaku Oxford Diffraction in Australia, the world leaders in crystallography. The system was installed at School of Molecular Sciences at the University of Western Australia with funding secured by a team of researchers led by Professor Alice Vrielink via the ARC LIEF funding scheme.

The XtaLAB Synergy-S boasts the latest generation microfocus sealed tube X-ray sources, with high precision optics, all controlled by user-inspired software to form an industry-leading solution. By combining the Cu PhotonJet-S microfocus X-ray sources with the new high-speed kappa goniometer with telescopic two-theta arm, researchers have the ability to tailor their experiments from sample screening to complete in-house data collection and structure solution.

Their system also includes a HyPix-6000HE HPC (Hybrid Photon Counting) detector. With its direct X-ray photon counting design, tiny 100µm pixel size and extremely low noise, researchers at UWA will be able to collect the higher quality data very quickly, allowing them to run more samples and further accelerate their research.

Prof. Vrielink is a leading light in the field of protein crystallography. When asked about the new installation, she replied, “we are carrying out world-class research and needed a system that offered us capabilities to carry out in-house diffraction data collection with speed as well as in plate crystal screening. The Synergy –S system is providing us with all of these features allowing us to remain at the forefront of our crystallographic work on these protein targets.”

Specific areas of interest to Prof. Vrielink and her team include, structure guided drug design aimed at designing new therapeutics in the areas of anticancer drugs as well as new antimicrobials to treat multidrug resistant bacterial infections.

Richard Trett, AXT’s Managing Director, also commented, “in-house protein crystallography has advanced significantly meeting the needs of the next-generation protein-based therapeutics to treat a vast array of ailments suffered by humans. We are pleased and proud that we are helping to put cutting-edge solutions like the XtaLab Synergy-S into the hands of Australian researchers.”

AXT’s life science portfolio includes a complete range of Rigaku Oxford Diffraction diffractometers as well as many other products sourced from multinational and small, innovative suppliers from around the world. More information can be found at www.axt.com.au.

Press Release

Rigaku Introduces Newest SmartLab Intelligent X-ray Diffraction (XRD) System

Rigaku Corporation
3-9-12, Matsubara-cho
Akishima-shi, Tokyo 196-8666, JAPAN

Rigaku introduces new automated multipurpose X-ray diffractometer with intelligent guidance software

April 2, 2018 – Tokyo, Japan. Rigaku Corporation is pleased to announce the release of the *new* next-generation Rigaku SmartLab intelligent multipurpose X-ray diffractometer. A highly versatile automated X-ray diffraction (XRD) system, the newest SmartLab® diffractometer offers continued refinement of the ease-of-use features that enabled the original SmartLab diffractometer to receive the coveted R&D 100 Award, such as automatic alignment, component recognition, Cross Beam Optics and a 2D detector.

SmartLab began as the flagship model from Rigaku in 2006 and new, advanced technologies have been continuously introduced over the years. This newest addition to the SmartLab series of high-resolution X-ray diffraction analyzers is engineered to provide the best performance in all X-ray diffraction or scattering applications by offering not only breakthrough hardware, but also the advanced SmartLab Studio II software with "User Guidance" expert system functionality, to establish a new standard in usability and flexibility for multipurpose X-ray diffractometers.

The new SmartLab system features the brand new PhotonMax high-flux 9 kW rotating anode X-ray source coupled with a HyPix-3000 high-energy-resolution 2D multidimensional semiconductor detector that supports 0D, 1D and 2D measurement modes. This allows all applications to be handled with a single detector, eliminating the inconvenience of preparing and switching individual detectors for different applications. The HyPix-3000 detector can be used to obtain 2D powder diffraction patterns, which can be processed to deliver superior qualitative and quantitative analysis by using all the 2D pattern information.

New Rigaku SmartLab intelligent multipurpose X-ray diffractometer

The system incorporates a high-resolution θ/θ closed loop goniometer drive system with an available in-plane diffraction arm. The system's new Cross Beam Optics (CBO) family feature fully automated switchable reflection and transmission optics (CBO-Auto).

The equipment accepts powder, films, and even textile samples and allows mapping measurements within a sample. *Operando* (a.k.a., real time *in-situ*) measurements can be performed with the new Rigaku SmartLab Studio II software suite, which is an integrated software platform incorporating all functions from measurement to analysis. The system also features robust security and validation protocols to ensure that any technology component - software or hardware - fulfills its purpose within regulatory guidelines, including 21 CFR Part 11, establishing the US EDA regulations governing electronic records and electronic signatures (ER/ES).

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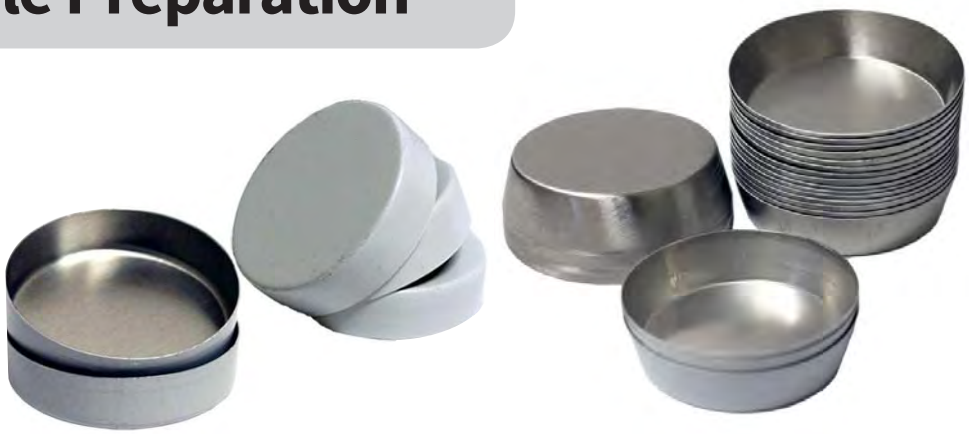

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Xenocs Xeuss 2.0 Q-Xoom: the agile detector for SAXS / WAXS

The Q-Xoom is the latest addition to the Xeuss 2.0 family and a result of the Xenocs-Saxslab collaboration. The Q-Xoom moving detector concept allows the fully automated optimization of instrument configuration to the user's measurement and experiment. It was originally developed on synchrotron beamlines and introduced in the laboratory by Saxslab.

The motorized adjustment of sample to detector distance gives the user the option to optimize detector coverage, resolution and Q-min, effectively moving from WAXS over MAXS to SAXS settings. It is also easy to acquire data with different settings in record time, including through remote operation.

With the detector in vacuum and with no windows in the beam-path, noise is reduced, and the dynamic range of the instrument is increased, covering about 10 orders of magnitude in signal.

The detector position is recorded yielding accurate establishment of the Q-range. With the sample being stationary, the beam-dimensions on the sample are constant and the user is sure to probe the same part of the sample for different detector settings. The stationary sample also preserves the Xeuss 2.0 capability for large sample environments and sample-in-air operation.

The Q-Xoom moving detector option is a milestone in SAXS instrument flexibility, performance and productivity.

For more information contact Rod Clapp on diffraction@bigpond.com.

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Both, Iron concentration as determined quantitively from the measurement process (direct assay method) via fused bead XRF analysis (as per ISO 9516-1) and Calculated Iron derived from the "difference" method (as per ISO procedure 2597-3) have been reported and certified.

To further characterize each material, additional information is provided, which includes the following:

- Particle Size Analysis Data
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- Sorption Testing Data, illustrating the potential effect of hygroscopic moisture on the quality of the chemical analysis results

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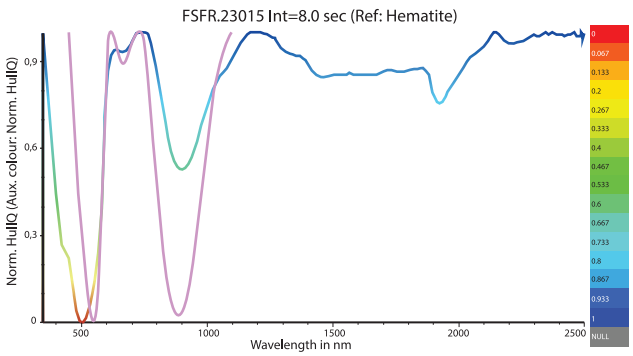

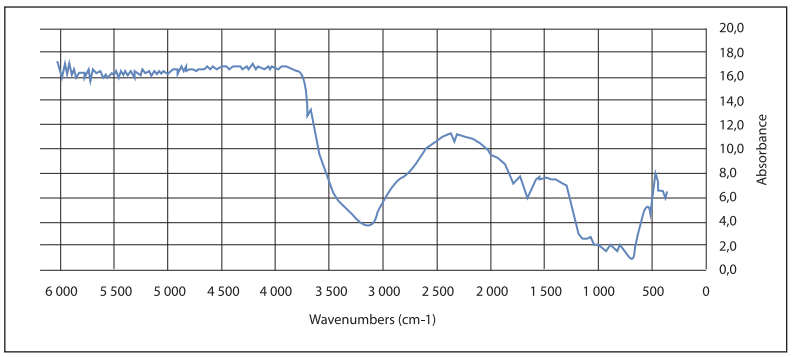

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										371°C	425°C	650°C	Total	
Fe-117	42,02	42,09	16,28	11,39	1,07	0,271	0,447	0,028	0,066	6,95	7,74	9,23	9,62	*
Goethite/Hematite	41,81	41,86	16,17	11,22	1,06	0,274	0,443	0,028	0,063	7,38	8,21	9,65	10,2	**
Fe-115	43,83	43,21	17,29	9,88	0,909	0,239	0,399	0,031	0,064	6,60	7,24	8,68	8,75	*
Goethite/Hematite	43,34	43,06	17,10	9,74	0,900	0,241	0,393	0,032	0,062	7,03	7,66	9,07	9,23	**
Fe-116	45,28	45,37	14,14	10,02	0,948	0,236	0,415	0,031	0,057	6,40	7,05	8,18	8,60	*
Goethite/Hematite	45,01	45,02	14,06	9,88	0,941	0,240	0,411	0,031	0,053	6,93	7,51	8,70	9,13	**
Fe-107	49,43	48,80	22,32	2,06	0,102	0,081	0,080	0,057	0,046	4,45	4,64	4,99	5,05	*
Goethite/Hematite	49,21	48,84	22,11	2,03	0,104	0,077	0,081	0,057	0,043	4,63	4,80	5,22	5,25	**
Fe-108	52,78	52,75	11,99	3,09	0,193	0,175	0,134	0,042	0,021	7,52	7,79	8,38	8,55	*
Goethite/Hematite	52,66	52,63	11,82	3,05	0,194	0,172	0,133	0,043	0,021	7,81	8,14	8,67	8,82	**
Fe-121	54,55	54,61	12,64	2,32	0,075	0,059	0,154	0,056	0,020	5,34	5,56	6,07	6,30	*
Goethite/Hematite	54,41	54,47	12,62	2,30	0,078	0,044	0,152	0,057	0,019	5,50	5,77	6,24	6,56	**
Fe-109	56,38	56,53	8,59	2,30	0,093	0,111	0,734	0,050	0,036	5,66	5,89	6,43	6,69	*
Goethite/Hematite	56,36	56,38	8,55	2,28	0,094	0,105	0,735	0,051	0,034	5,88	6,09	6,65	6,95	**
Fe-105	56,77	56,82	6,39	2,80	0,129	0,094	0,957	0,047	0,039	6,56	6,79	7,33	7,62	*
Goethite/Hematite	56,53	56,61	6,35	2,77	0,130	0,094	0,950	0,047	0,037	6,87	7,11	7,63	7,95	**
Fe-110	56,77	56,73	8,55	2,15	0,086	0,116	0,809	0,053	0,040	5,46	5,67	6,19	6,45	*
Goethite/Hematite	56,56	56,56	8,52	2,14	0,087	0,122	0,802	0,053	0,039	5,69	5,90	6,42	6,68	**
Fe-111	56,91	56,85	8,42	2,20	0,079	0,104	0,816	0,051	0,033	5,50	5,72	6,21	6,45	*
Goethite/Hematite	56,76	56,72	8,30	2,16	0,079	0,096	0,815	0,052	0,032	5,75	5,95	6,47	6,77	**
Fe-113	56,89	56,66	7,12	2,87	0,097	0,108	0,322	0,047	0,032	6,89	7,18	7,71	8,00	*
Goethite/Hematite	56,53	56,55	7,04	2,83	0,097	0,104	0,322	0,048	0,030	7,11	7,38	7,92	8,21	**
Fe-102	57,07	57,11	6,20	2,75	0,101	0,167	0,590	0,048	0,037	6,77	6,98	7,50	7,76	*
Goethite/Hematite	56,91	56,96	6,16	2,74	0,102	0,172	0,588	0,048	0,034	6,95	7,22	7,73	8,05	**
Fe-103	57,19	57,24	5,86	2,76	0,115	0,092	0,863	0,047	0,037	6,67	6,90	7,48	7,77	*
Goethite/Hematite	57,06	56,98	5,83	2,73	0,116	0,086	0,861	0,048	0,036	6,89	7,14	7,70	8,01	**
Fe-120	57,26	57,18	6,20	5,20	0,359	0,038	0,021	0,075	0,022	4,77	5,16	5,91	6,10	*
Hematite/Goethite	56,97	56,95	6,14	5,17	0,361	0,034	0,024	0,077	0,019	5,03	5,45	6,17	6,48	**
Fe-104	57,31	57,39	5,67	2,78	0,113	0,096	0,839	0,040	0,039	6,65	6,89	7,45	7,76	*
Goethite/Hematite	57,12	57,17	5,65	2,75	0,114	0,104	0,835	0,040	0,036	6,89	7,17	7,71	8,03	**
Fe-106	57,33	57,20	5,90	3,08	0,171	0,105	0,572	0,047	0,034	6,54	6,80	7,42	7,69	*
Goethite/Hematite	57,11	57,12	5,86	3,05	0,174	0,103	0,571	0,047	0,032	6,76	7,02	7,64	7,93	**
Fe-114	57,51	57,57	5,17	2,97	0,102	0,087	0,296	0,043	0,033	7,52	7,77	8,34	8,62	*
Goethite/Hematite	57,32	57,41	5,15	2,95	0,101	0,091	0,293	0,044	0,032	7,76	8,06	8,58	8,85	**
Fe-112	57,83	57,86	4,67	2,96	0,099	0,077	0,228	0,043	0,034	7,73	8,02	8,58	8,83	*
Goethite/Hematite	57,64	57,60	4,63	2,93	0,099	0,094	0,229	0,043	0,034	7,97	8,26	8,81	9,18	**
Fe-101	58,18	58,10	5,54	2,45	0,094	0,106	0,495	0,049	0,033	6,66	6,87	7,39	7,66	*
Goethite/Hematite	57,94	57,97	5,51	2,42	0,095	0,101	0,493	0,049	0,031	6,85	7,09	7,62	7,89	**
Fe-119	60,06	60,07	6,23	2,64	0,220	0,045	0,023	0,077	0,021	3,72	3,92	4,35	4,58	*
Hematite/Goethite	59,83	59,95	6,22	2,62	0,219	0,042	0,023	0,077	0,021	3,94	4,10	4,54	4,76	**
Fe-118	61,04	60,89	4,68	4,44	0,577	0,057	0,055	0,049	0,024	1,96	2,17	2,54	2,74	*
Hematite/Goethite	60,71	60,06	4,66	4,40	0,581	0,057	0,055	0,049	0,021	2,20	2,37	2,78	3,01	**
Fe-123	63,22	63,15	5,93	1,77	0,095	0,105	0,037	0,058	0,013	0,270	0,319	0,688	0,816	*
Hematite	63,22	63,12	5,94	1,77	0,097	0,103	0,036	0,059	0,011	0,339	0,384	0,749	0,897	**
Fe-122	63,66	63,50	5,19	2,07	0,113	0,032	0,153	0,042	0,017	0,477	0,556	0,823	0,973	*
Hematite	63,54	63,47	5,19	2,04	0,115	0,030	0,153	0,043	0,015	0,563	0,591	0,885	1,040	**
Fe-124	63,99	63,82	5,18	1,83	0,098	0,036	0,172	0,041	0,012	0,254	0,331	0,561	0,705	*
Hematite	63,98	63,82	5,16	1,81	0,099	0,027	0,174	0,041	0,010	0,336	0,417	0,638	0,804	**

*ISO2596:2006
**ISO7764:2006

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30 April – 4 May 2018

From theory to hands-on exercises, this course offers techniques and skills to improve lab performance. Discover the latest in cutting-edge instruments such as TXRF, hand-held devices, energy dispersive and wavelength dispersive spectrometers through live demonstrations.

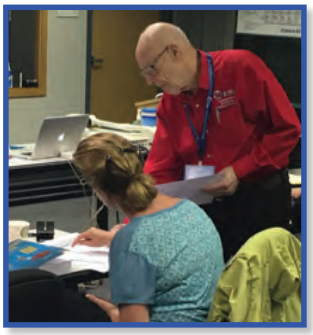
The XRF course covers the basics of X-ray spectra; instrumentation design; methods of qualitative and quantitative analysis; specimen preparation and applications for both wavelength and energy dispersive spectrometry. The course emphasizes quantitative methods, use of automated X-ray spectrometers, review of mathematical matrix correction procedures, and new developments in XRF.

Fundamentals of X-ray Powder Diffraction:

4 – 8 June 2018

For the novice with some XRD knowledge or for the experienced with an interest in the theory behind XRD, this clinic offers a strong base for increased lab performance.

The clinic covers instrumentation, specimen preparation, data acquisition and qualitative phase analysis through live demonstrations. It also covers hands-on use of personal computers for demonstration of the latest software including data mining with the Powder Diffraction File (PDF) and use of the powder diffractometer: optical arrangement, factors affecting instrumentation profile width, choice and function of divergence slit, calibration and alignment, detectors, and X-ray optics.



Advanced Methods in X-ray Powder Diffraction:

11 – 15 June 2018

For the experienced XRD scientist, this session offers enhanced analysis skills through intense problem solving, as well as an introduction to the Rietveld Method. The course emphasizes computer-based methods of data collection and interpretation, both for qualitative and quantitative phase analysis.

The advanced clinic covers factors affecting d-spacings of crystals, as well as factors affecting diffraction-line intensities; structure-sensitive properties (atomic scattering and structure factors), polarization effects, and multiplicity. Additionally, the clinic covers specimen-sensitive effects (orientation, particle size), measurement-sensitive effects (use of peak heights and peak areas), and choice of scanning conditions will also be addressed.

Please visit the ICDD website for more information.

Rietveld Refinement & Indexing Workshop:

Rietveld Refinement & Indexing Workshop I & II: 24 – 28 September 2018
Basic (I) Workshop: 24 – 26 September 2018
*Advanced (II) Workshop: 26 – 28 September 2018

Powder pattern indexing and Rietveld structural refinement techniques are complementary and are often used to completely describe the structure of a material. Successful indexing of a powder pattern is considered strong evidence for phase purity. Indexing is considered a prelude to determining the crystal structure, and permits phase identification by lattice matching techniques. This workshop introduces the theory and formalisms of various indexing methods and structural refinement techniques along with quantitative analysis. One unique aspect of this workshop is the extensive use of computer laboratory problem solving and exercises that teach method development in a hands-on environment.

Take the three-day basic workshop, the three-day advanced workshop or attend both for a full week of hands-on training.

**See the ICDD website for prerequisites for the advanced Rietveld course.*

Register today at WWW.ICDD.COM/EDUCATION

Please note: A minimum of 10 registrants per course is required, otherwise the course will be cancelled and your registration fee will be refunded. You will be notified of a course cancellation no later than two weeks prior to the start of the course.

For More Information Contact

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