AXAA e-newsletter

Issue 2, 2013 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Friends and Members,

We hope this newsletter finds you all well. AXAA central is alive and kicking with our preparations for the upcoming AXAA-2014 event. We have a lot of special events in store for AXAA-2014, and the organising committee is excited to announce that in 2014 we will offer the opportunity to publish full papers in the *Powder Diffraction* Journal as part of the conference. Our list of plenary speakers is growing, with confirmations received from high-profile attendees including Prof. David Bish (Indiana University, USA), Mr John Fowler (Intertek, WA), Prof. Bill David (ISIS, UK) and Dr Robert von Dreele (Argonne National Laboratory, USA).

As AXAA-2014 approaches, we have some fantastic opportunities for students planned at our upcoming student seminar days, including bursary prizes to attend AXAA-2014 offered at both our NSW and Vic events. Once again, read on for further details.

Finally, we remind our membership that now is the time to consider nominations for the Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis and the Bob Cheary AXAA Award for Excellence in X-ray Diffraction Analysis. The deadline for nominations is 25th October and fast approaching

Vanessa Peterson
National Council President

AXAA 2014 Workshops, Conference and Exhibition – **CALL FOR ABSTRACTS**

Confirmed plenary speakers
Professor David Bish (Indiana University, USA)
Mr John Fowler (Intertek, WA)
Professor Bill David (ISIS, UK)
Dr Robert von Dreele (Argonne National Laboratory, USA)

Registration and abstracts for the AXAA 2014 Workshops, Conference and Exhibition are now open! Please register and submit your abstracts through: www.axaaconference.info

To submit an abstract you will need to submit your contact details on the abstract submission site, you will then receive instructions about the abstract preparation and submission by email.

For AXAA 2014 there is also the opportunity to submit a full paper for publishing in *Powder Diffraction* Journal. Select the relevant option during registration if you intend to submit a full paper for publication in *Powder Diffraction*. Papers will be a minimum of 4 and maximum of 6 printed journal pages. Full papers will be submitted ~1 month after the conclusion of AXAA 2014 (full details regarding paper preparation and submission will be provided at a later date).

Abstract submission closes Monday 14th October 2013

A very special thank you to our Platinum Sponsors:

BRUKER PANalytical

Matters for Scatterers

Coal, with a global annual consumption currently reaching 7 billion tons, is a major source of energy accounting for 40% of world electricity supply. Even in Australia, 66% of our electricity is supplied by coal-fired plants. This scenario is expected to continue stably in the foreseeable future. However, coal combustion has huge environmental implications as the vast total amounts of coal combusted release undesirable quantities of hazardous elements, even those present in trace levels.

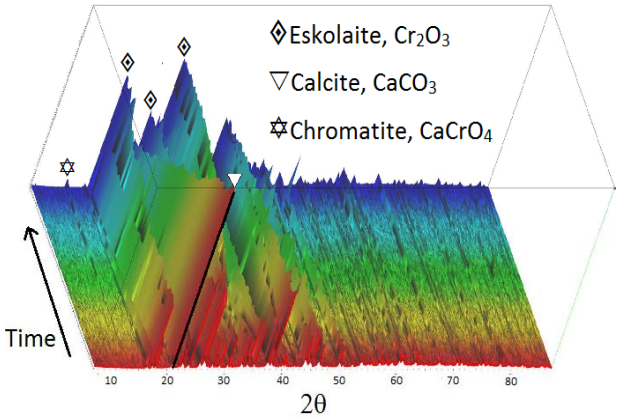
One of these elements of concern is hexavalent chromium. In contrast to its non-toxic trivalent state, Cr(VI) is highly mobile and have been identified to be a human carcinogen. Therefore, it is essential to understand the conditions affecting and contributing to its formation.

From *ex-situ* XRD and XANES, there is a strong evidence of a complex interaction between Cr(III) oxide and CaO (an abundant component in coal) upon heating in the presence of O_2 to 600°C onwards, resulting in the formation of $CaCrO_4$ and $Ca_3(CrO_4)_2$. For other oxides including Fe_2O_3 and MgO which are also abundant in coal, the presence of Cr(VI) was not observed by their heating with Cr_2O_3. Therein lies questions on what are the pathways for the transition of Cr(III), and the evolution of different oxidation states for Cr under the assistance of CaO.

Based on these considerations, *in-situ* high-temperature XRD (HT-XRD) experiments using the Australian Synchrotron Powder Diffraction beamline were performed on Cr_2O_3 + CaO mixtures using different heat settings and gas environments.

Sample mounted on platinum strip in furnace for in-situ HT-XRD experiments.

Compared to *ex-situ* analytical methods, real-time monitoring enables identification of intermediate products and their kinetics, and hence, provide key information necessary for the formulation of detailed reaction mechanisms. With these, new fundamental knowledge on the detailed phase transition of Cr is gained, thereby improving the ability to anticipate and mitigate the production of Cr(VI) during coal combustion.



3D view of the progression of in-situ XRD spectra used for tracking the reactant's kinetic development.

Fiona Low
Monash University
fiona.low@monash.edu

XRF Operations Technical Note

Towards Better XRF Results: Maximising Wavelength Dispersive (WD) XRF Instrument Stability

For good quality XRF analysis results it is essential that the spectrometer is operating in a stable mode. One of the factors which will affect the precision and accuracy of the results is thermal stability. If the cabinet temperature fluctuates then this may influence the crystals which by a change in crystal D-spacing will alter the position of the respective element line to be measured. Changes in cabinet temperature will also cause changes in the density of the P10 gas in the flow counter, which will in turn, change the gain of the flow detector. This temperature change may also have an influence on the various and many electronic components which could affect the signals and levels which control the spectrometer and the output of the detectors. While WD XRF spectrometers have sophisticated in built cabinet temperature control, providing a stable temperature environment for the XRF spectrometer (i.e. well designed air conditioning) greatly assists in achieving optimum cabinet temperature stability.

Another of the factors that affects XRF performance is the temperature of the x-ray tube anode. If the tube target temperature changes then the x-ray emission intensity of the tube will vary. Varying the power on the x-ray tube will cause the above effects as well as having a serious destabilising effect on the attached chiller unit which may start oscillating over a large temperature range.

In modern XRF spectrometers the tube target temperature is controlled, within tight limits, by the cooling water temperature controller. The precise constant temperature of the tube target and hence, maximum stability, can only be maintained when the tube is operated under constant power (isowatt) conditions.

That is, the product of the tube voltage (kV) and the tube current (mA) must be a constant and this must be maintained when changing excitation conditions for different analytes. For example, heavy elements like Fe are normally measured at high kV (typically 60 kV) whereas light elements like S at low kV (typically 30 kV). Assuming a target power of 3 kW for the purposes of this example, an operating current of 50 mA would be selected for Fe and 100 mA for S. This isowatt switching maintains the same total power at the tube target for both measuring conditions and hence minimises temperature changes during switching, resulting in optimum stability and minimal switching times.

In some instances, default operating conditions suggested by the instrument software (crystal, collimator, detector, etc.), may result in excessively high count rates. It is sometimes laboratory practice to reduce the tube power, by as much as 75%, to bring the count rates within the operating range of the detector in the instrument. **This is not good practice** since it will take considerable time for the tube anode to re-equilibrate to a stable operating temperature and during this time the XRF will not be operating at optimum stability. It is far better practice to reduce intensity by other means such as: alternative crystal or collimator combinations, using an alternative characteristic line or inserting an appropriate beam filter. Attenuators are often available in simultaneous instruments. It is possible to program in a time delay to allow the tube to regain stability and this is applied automatically by some manufacturers. However, this approach leads to longer analysis times, hence lower throughput and may still causes the other effects mentioned above.

Apart from the impact on instrument stability there is anecdotal evidence to suggest that non isowatt switching is detrimental to x-ray tube life. This is likely as rapid changes in tube power may cause significant thermal stress within the tube which could, in turn, lead to premature tube failure.

Of course, the above also assumes that the electrical power supplied to the spectrometer and ancillary equipment is to the quality and standard as suggested by the suppliers. Poor quality electrical power will cause instrument instability. Hence, fitting a good quality UPS/Power Conditioner is strongly recommended.

Ken Turner (Ken Turner Consulting)
Gary Pritchard (Gary Pritchard Consulting)

Upcoming events

1. AXAA Student Seminar Days

NSW

19th September 2013

Chemistry Seminar Room, GO5, Dalton Building, University of NSW

Contact Vanessa Peterson (vep@ansto.gov.au)

VIC

9th October 2013

Seminar Room, National Centre for Synchrotron Science, Australian Synchrotron

Contact Nathan Webster (nathan.webster@csiro.au)

Prizes for the best presentations will be bursaries to attend AXAA-2014

With special thanks to UNSW and the Australian Synchrotron for providing the venues

2. **National XRD Course**

X-Ray Powder Diffraction Analytical Methods

Curtin University, Perth, Sat 9 Nov – Tue 12 Nov 2013

Venue: Department of Imaging and Applied Physics, Curtin University, Bentley (Perth), Western Australia

[Client-specific version of the course can be presented at the customer's site]

- Duration of Curtin course: 4 days
- Dates: 9 -12 November, 2013
- Course Director: Professor Brian O'Connor
- Course presenters: Professor Brian O'Connor and Dr Catherine Kealley
- Further information and application form: B.O'Connor@curtin.edu.au
- Cost: $2,695 including GST

Availability of places strictly limited.

Overview: The course has been designed to give participants a theoretical and practical grounding in the principal characterisation methods which make use of x-ray powder diffractometry data. Approximately 60% ofthe course involves hands-on instruction. Participants personally collect diffractometry data sets and then process these, both manually and with PC computers, in

exercises on various analytical methods, including Rietveld analysis. Public domain software will be used, including *WINPLOTR* and *Rietica*. The course also includes overviews and demonstrations of the commercial software packages *X'Pert HighScore Plus* and *Diffracplus Topas*. While the course is relevant to the analysis of all classes of crystalline materials, attention is devoted mainly to materials relevant to the mining and mineral processing sector.

3. Internet XRD (Basic) Course

Available from April 2013

This course has been developed primarily in response to industry requests. It provides XRD analysts, particularly those new to the field, with on-site instruction on the practical principles of powder XRD for materials analysis.

Features of the course include –

- 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 9 modules, with an assignment being set for each module.
- Feedback on the assignments provides excellent mentoring.
- May be used as a vehicle for in-house XRD training.

Overview: The course has been designed to give new XRD practitioners a grounding in the use of powder XRD for materials analysis, with particular reference to data measurement, phase identification and quantitative phase composition analysis. It provides an excellent grounding for those wishing to proceed to more advanced XRD characterisation methods using techniques such as Rietveld analysis, indexing and structure solution.

Course availability: 5 places currently available
Course Director: Dr Brian O'Connor.
Course fee: $2,695 including GST.
Further information and enrolment:
brian_oconnor@iprimus.com.au (Tel 08 9291 7067)

4. Internet XRF Course: Series 6, 2013

The course provides XRF analysts, particularly those new to the field, with on-site instruction in the practical principles of wavelength dispersive XRF. Features of the course include –

- 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 11 modules; with an assignment being set for each module.
- Feedback on the assignments provides excellent mentoring.

The course now has a substantial number of international participants, as well as Australians, and is being used by companies as a vehicle for in-house XRF training.

Course availability: There are **4 places available** as of the end of June 2013.

Course director: Dr Brian O'Connor.
Course fee: $2,695 including GST.
Further information and enrolment:
brian_oconnor@iprimus.com.au (Tel 08 9291 7067)

5. TOPAS Beginner Course

Course 1: September 12th and 13th, 9am-5pm
Bruker Office, Preston, VIC

Course 2: November 14th and 15th, 9am-5pm
Curtin University, Bentley Campus, Perth, WA

Course fee: $185 + GST; this includes course notes and lunch and tea/coffee on both days.

Place are filling fast and registrations close 23rd of August (for the Melbourne course).

The program for the Topas Beginner Course is now available here:

www.bruker-axs.com.au/topasbc_prog.pdf

The registration form can be downloaded here:

www.bruker-axs.com.au/topasbc.pdf

Attendees are required to supply laptops and Topas pre-installed.

Loan licence dongles may be supplied for the two days, however it is strongly suggested attendees bring their own licence dongle.

For more information contact Martin Duriska
md@bruker.net.au

AXAA Awards for Excellence in X-ray Analysis: Call for Nominations

Closing Date for Submission of Nominations
25th October 2013

The Awards
Two awards have been established, one for XRF and one for XRD. These will be for "significant long term

contributions" to x-ray analysis rather than say a single paper, and will perpetuate the contribution of the person after whom the award is named.

XRF – Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis

XRD – Bob Cheary AXAA Award for Excellence in X-ray Diffraction Analysis

Form of Awards
Each award will comprise an engraved medal.

Selection Criteria

1. The principal criterion will be the excellence of the applicant's development of high-impact, innovative x-ray analysis methods and their take-up by the x-ray analysis community. Work in which XRF or XRD has been a peripheral tool will not be considered.
2. The period over which the contribution is to be considered will be at least 5 years.
3. All or most of the cited work will have been conducted in Australia.(cont'd)
4. The recipient will have been a member of AXAA for at least 5 years prior to the application being submitted.
5. It is desirable, but not essential, that the applicant has contributed to AXAA in a substantial way, for example through quality presentations at AXAA national conferences and/or administrative service for AXAA.
6. Past recipients of an AXAA XRD or XRD award will not be considered for a second award in the same category.

Applications
Applications will be submitted by a nominator on behalf of the applicant. The documentation will comprise:

- CV
- Publication list. This may include items protected by confidentiality if the applicant can pre-arrange an appropriate confidentiality agreement.
- Advocacy statement highlighting the application's alignment with the selection criteria.
- Names and contacts for three technical referees, one being the nominator.
- Applications are to be submitted in electronic form to the AXAA President – Vanessa Peterson. Please send to Vanessa as an attachment: vanessa.peterson@ansto.gov.au

Selection Process

1. The National Council will appoint a selection panel of three persons for each award. The selection panels will be allowed approximately two months to review the applications and make recommendations.
2. The recommended recipients will be considered by the AXAA Council which will then make a formal decision on the recommendations. Decisions by the Council will be final, and there will be no appeal process.
3. The Council reserves the right not to make an award if the standard of applications is deemed to fall below the expected standard.

Membership Matters

AXAA-Inc membership is for the 3-year period starting from the 2011 AXAA National Conference (existing membership will be re-approved in 2014). Membership is free. Candidates should provide a brief CV and a short statement of intention about what they intend to do in the organisation. Please send these to National Council Secretary Natasha Wright. The council votes on membership applications at least once every 6 months.

AXAA Contacts

http://www.axaa.org/

NATIONAL COUNCIL PRESIDENT
Vanessa Peterson, Bragg Institute, ANSTO
Telephone: (02) 9717 9401
e-mail: vanessa.peterson@ansto.gov.au

NATIONAL COUNCIL SECRETARY
Natasha Wright, CSIRO Materials Science and Engineering
Telephone: (03) 9545 2041
e-mail: natasha.wright@csiro.au

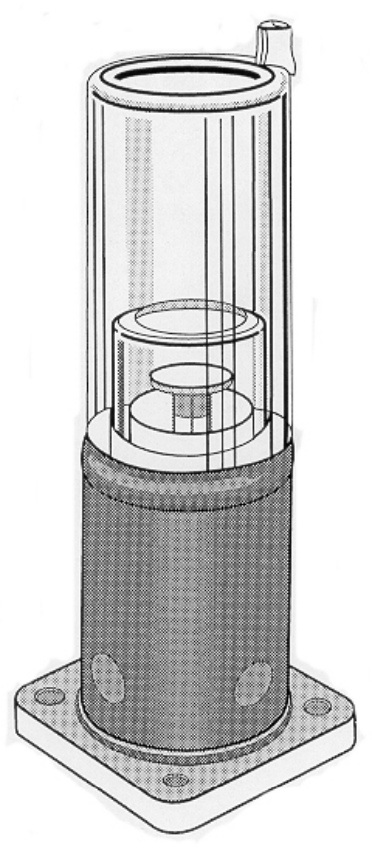
NATIONAL COUNCIL TREASURER
Gordon Thorogood, Institute of Materials and Engineering, ANSTO
Telephone: (02) 9717 3183
e-mail: gordon.thorogood@ansto.gov.au

NEWSLETTER EDITOR
Nathan Webster, CSIRO Process Science and Engineering
Telephone: (03) 9545 8635
e-mail: nathan.webster@csiro.au

Diffraction Technology

Instruments and accessories for X-ray analysis

Your source of X-ray Diffraction Tubes



The XRD 1000 series of glass insulated tubes All feature high primary intensity, a homogeneous intensity distribution, spectral purity and long lifetime.

- Direct replacements for:
  glass insulated “European Standard” Tubes

The XRD 2000 series of ceramic insulated tubes are constructed with a steel body which incorporates the latest ceramic insulation. This provides for enhanced thermal conductivity and a much longer life span. All feature high primary intensity, a homogeneous intensity distribution, spectral purity and long lifetime.

Full line of Tubes
2 anode lengths
4 different focal spots
7 different target materials

- Large and Thin Be-window
- Sharp focal spots
- Manufactured under ISO 9000
- Direct replacements for all common “European Standard” XRD tubes:

All available on short delivery, please ask for a quote.

Diffraction Technology e-mail diffraction@bigpond.com phone 03 9787 3801 Web www.diffraction.com.au

PANalytical

PANalytical X-ray solutions: from mining to quality control

Exploration, grade control, process monitoring and quality assurance benefit from robust and flexible analysis of raw materials, feed, concentrate and tailings.

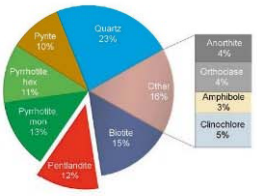
PANalytical systems are ideal for the high throughput analysis requirements of the mining industry. Supported by the unique service program PANassist and a global sales and service team, PANalytical gives you the expertise you need for precise and accurate control of mining, beneficiation and ore grade. Experience PANalytical's innovative and flexible analysis solutions especially designed for the minerals and mining industry.
Besides the elemental content determined with X-ray fluorescence (XRF), the knowledge of the minerals determined with X-ray diffraction (XRD) can predict process behavior and recovery rates of the beneficiation process (flotation, magnetic separation, etc) of the ore. The examples shown below demonstrate the enormous potential of XRD as an inexpensive, reliable tool, useful in the characterization of ore materials from any geological environment.

Process control for copper/lead/zinc

- Avoid tube blockings with frequent control of clay minerals present in the ore and concentrate
- Increase ore recoveries by adjusting the chemicals used for flotation based on the exact minerals composition of the feed materials
  - Chalcopyrite, bornite, chalcosite, covellite, malachite…
  - Galenite
  - Sphalerite
- Improve geometallurgical model by combining geological and metallurgical information to create spatially-based predictive model for mineral processing

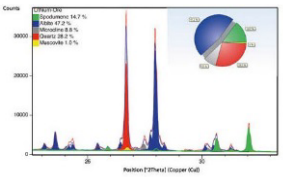
Quantification of nickel ores

- More detailed study of genesis, mineral distribution and subsequent beneficiation of magmatic nickel ore deposits
- Time and labor efficient identification and quantification of the mineralogy, specially of hexagonal pyrrhotite and monoclinic
  - Better recoveries during magnetic separation process
  - Optimized flotation due to different oxidation behavior of pyrrhotite polymorphs
  - More precise estimation of nickel recoveries due to different nickel content of pyrrhotite polymorphs
  - Better accuracy compared to traditional method



Analysis of lithium ores and processed concentrates

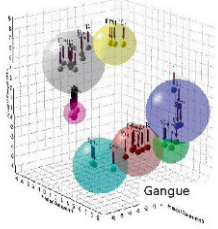
- Direct analysis of lithium containing minerals in raw ores from hard rock deposits or brines
- Quantitative phase analysis of all crystalline minerals present (Li phases and gangue minerals) without dissolving and further extensive sample preparation
- Cost and time effective due to no chemicals involved
- Optimal recoveries with monitoring the complete process
  - a. Ore (feed)
  - b. α-Spodumen
  - c. Residue
  - d. β-Spodumen
  - e. Na-zeolite



Exploration and mine planning of gold deposits

- Fast identification of gold bearing mineralogical provinces of a deposit using statistical cluster analysis
- Improved prediction of leaching rates
- Optimized recoveries during flotation due to:
  - Identification and quantification of mineral phase with similar chemical composition (pyrite, arsenopyrite or sinnerite)
- Better understanding of metallurgical processes such as autoclave
  - Analysis of secondary phases like arsenates or sulfates

Samples with gold-bearing sulfide phases



Tailor made solutions for mining industries:

Axios^{mAX}-Minerals spectrometer for routine analysis of elemental composition

CubiX^3 Minerals diffractometer for exploration and process control of mineralogy

Epsilon 3 bench-top spectrometer for cost effective elemental analysis

CNA cross-belt analyzer for real time elemental analysis

X-rays help to see !!!

PANalytical
Spectris Australia Pty Ltd
Unit 24/31 Governor Macquarie Drive
Chipping Norton
Sydney NSW 2170
Tel.: +61 2 8700 2700
Fax: +61 2 8700 2710
scott.gilroy@panalytical.com
www.panalytical.com

PANalytical launches new XRF analysis software

New software offers convenience, speed and accuracy

PANalytical, the world's leading supplier of analytical X-ray equipment and software, has launched its new software for X-ray fluorescence (XRF) systems. PANalytical developed Stratos, a brand new software package, for both the Epsilon 3 and Axios spectrometer ranges. The company has also released an upgrade of the FingerPrint software for the Epsilon 3 range.

Stratos – sophisticated analysis made easy

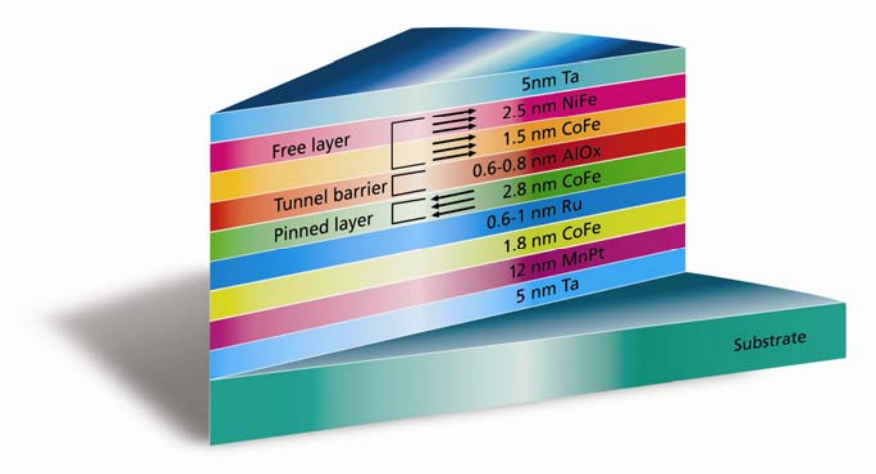
The Stratos XRF analysis software features built-in intelligence and can quickly and accurately analyze the thickness and composition of coatings, surface layers and layered structures. Multi-layer samples can be analyzed with bulk standards, without the need for in-type standards that are sometimes hard to source. Another significant advantage of the software is its flexibility and the ease of use provided by the 'Virtual Analyst'. This unique tool is a consultant for advanced method development and provides optimum measurement settings for analysis, which can be time-consuming for complex stack structures. Stratos is able to analyze more than 16 layers, depending on their thickness and composition. It is also easily combined with PANalytical's Omnian semi-quantitative software.

The Stratos software developed by PANalytical makes daily operation easier, with a simple interface that helps new users. Stratos is available for both the Epsilon 3 range (energy dispersive, EDXRF) and the Axios range (wavelength dispersive, WDXRF). Applications of Stratos include the automotive industry, wafers and solar cells and the coating and packaging industry.

FingerPrint software upgrade for more convenience

FingerPrint software is the second generation of PANalytical's successful material identification software for the benchtop Epsilon 3 range of spectrometers. The unique combination of FingerPrint software with Epsilon 3 sets new standards for industrial and process control applications. The upgraded FingerPrint software makes it even easier for material testing where the actual composition is not of interest and analysis speed is important. The addition of simple pass/fail reporting means that expert knowledge is no longer required to interpret the results and application of PCA (principal components analysis) allows cluster analysis in 3D visualization. FingerPrint software is used in materials identification, comparison and quality control and can be used in a wide range of applications, for example in the pharmaceutical, cosmetic, food and petrochemical industries.

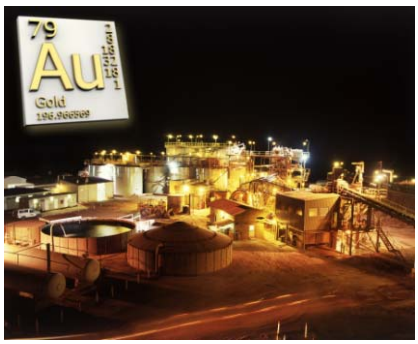
For further information, visit: www.panalytical.com or contact your local PANalytical representative.



Caption: Stratos enables accurate thickness and composition determination for coating and packaging industries. The key advantages include maximized multi-layer capability, minimized calibration costs, full flexibility and ease of use.

New Developments in XRF for Gold Processing

Applied Rigaku Technologies, Inc. (Austin, Texas USA), announces new developments in EDXRF systems for use in gold processing. Gold processing commonly utilizes methods to recover trace gold content by using cyanide leaching techniques. After leaching, the pregnant leachate is typically absorbed onto activated carbon in processes such as Carbon-in-Pulp (CIP), Carbon-in-Leach (CIL), Carbon Column (CC) and other processes. Simple, quick and accurate quantification of the gold content in the stripping solution is critical for optimum control of electrowinning extraction processes where gold is recovered by electrolysis.



By routinely measuring the gold in the stripping solution, operators can know when the solution is barren of gold content. The measurement may traditionally be carried out using Atomic Absorption (AA) analysis, which can be time consuming and somewhat prone to errors in sample handling and preparation techniques. Energy Dispersive X-ray Fluorescence (EDXRF) offers a faster and simpler analysis of the gold in the stripping solution, for both at-line measurements and on-line real time process control. Having a fast and simple technique for making the measurement adds significant value by allowing more frequent measurements to be made by skilled and unskilled operators alike.

EDXRF INSTRUMENTATION

For at-line analysis the Rigaku NEX QC⁺ offers the operator a self-contained benchtop system with modern touch screen and simple intuitive icon-driven software flow. With no external gasses, no special sample preparation and short measurement times, the NEX QC⁺ is an ideal tool for monitoring at the processing site. For continuous real-time monitoring of gold stripping solution, the technology is also available as the on-line model NEX OL. Both systems make use of the same proprietary tube filter set for excellent removal of background signal, as well as SDD detection (Silicon Drift Detector) to achieve detection levels of 1 ppm or less.

At-line

NEX QC+

On-line

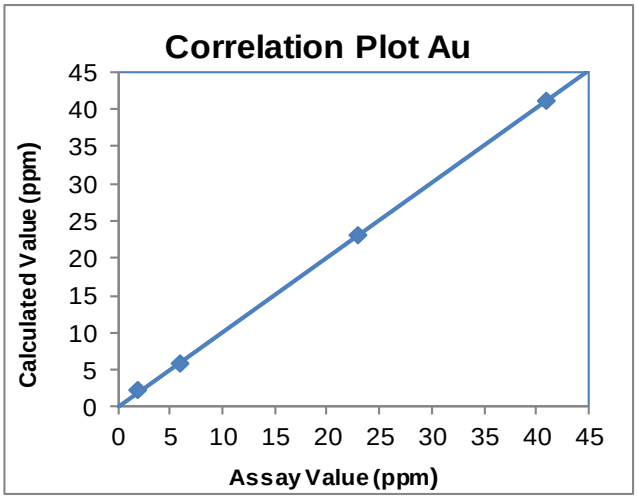
NEX OL

RIGAKU PERFORMANCE

The at-line and on-line performance is comparable. Demonstrated here is the performance of NEX QC+ using 100 sec measurement time.

Element: Au	SEE: 0.16 ppm
Units: ppm	Correlation: 0.9999

Sample ID	Assay Value	Calculated Value
2	2.0	2.2
6	6.0	5.8
23	23.0	23.0
41	41.0	41.0



One-time calibration is achieved using a simple empirical linear regression based on four or more assayed samples.

Element: Au	Units: ppm		
10 repeat measurements @ 100 sec per analysis			
Sample	**Standard Value**	**Average Value**	**Std Dev**
2	2.0	2.4	0.4
6	6.0	5.8	0.6
23	23.0	22.8	0.7
41	41.0	40.5	0.7

Repeatability of the measurement is excellent, giving the operator <1 ppm precision in 100 sec analysis time.

Element	Estimated LLD	Measurement Time
Au	1.3 ppm	100 sec
	0.9 ppm	200 sec
	0.8 ppm	300 sec
	0.7 ppm	400 sec

Excellent sensitivity for monitoring gold instrip solution.

The Rigaku NEX QC+ and NEX OL EDXRF analyzers achieve excellent results in monitoring the concentration of gold in strip solution during the processing of ores, tailings and slags, and monitoring other strip solutions for silver, copper or other metals can also be served by Rigaku EDXRF. For more information, contact Applied Rigaku Technologies info@rigakuedxrf.com or your local Applied Rigaku Technologies distributor. www.RigakuEDXRF.com

Benchtop X-ray diffractometer

MiniFlex300
MiniFlex600

1. Introduction

In January 2012, Rigaku released the MiniFlex300 and the MiniFlex600, the latest models in the MiniFlex benchtop XRD series. The numerical designations indicate the generator performance of these systems, 300 W and 600 W respectively.

MiniFlex diffractometers are widely used in a variety of fields, such as ceramics, minerals, inorganic materials and pharmaceutical ingredients. They are small compared with conventional X-ray diffractometers—about one-twentieth the volume and one-tenth the weight.

2. New features

2.1. High power

Approximately 1.5 times higher intensity is obtained with the MiniFlex600 compared to the MiniFlexII (450 W).

2.2. High intensity and high resolution mode

Two modes can be selected according to the application: intensity priority or resolution priority. In intensity priority mode, a continuously variable divergence slit is used in addition to the slit correction mode (variable divergence slit+fixed divergence slit) featured on the MiniFlex Ⅱ to obtain high intensity measurement data. In resolution priority mode, high resolution soller slits of 0.5° and 2.5° are provided in addition to the standard soller slit of 5.0°. A fine focus X-ray tube with 0.4×0.8 mm focus is used for higher resolution measurement than is possible with a normal focus X-ray tube.

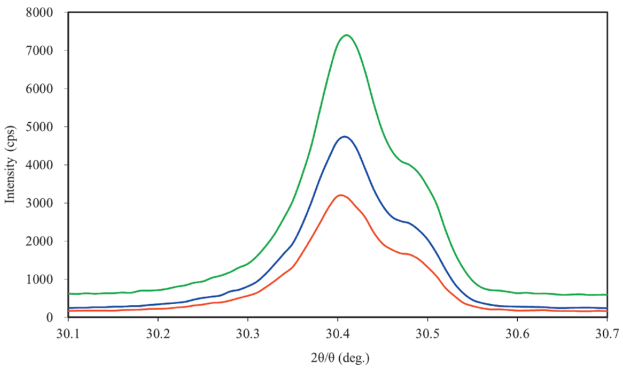


Fig. 1. Comparison of X-ray diffraction intensity between MiniFlex 300 (red line)/600 (green line), and MiniFlexII (blue line).

2.3. Real-time angle correction system

In real-time angle correction, angular precision is increased by applying a correction based on the difference between the theoretical and observed angle values. Once the correction value has been determined, it can be applied to every measurement under computer-control. The best angle precision is achieved by the combination of the high precision mechanical goniometer ring and real-time angle correction.

2.4. High intensity measurement using high-speed one dimensional detector D/teX Ultra

Using the D/teX Ultra detector, intensity data from 10 to 100 times higher can be obtained compared with a scintillation counter. The D/tex Ultra has high energy resolution, and the user can change the energy detection range via software. XRF reduction mode enables users to perfume low background measurements without a graphite monochromator.

3. Other features

Because the MiniFlex300 has an internal chiller, it does not require an external chiller unit and therefore requires a smaller installation space.

An automatic sample changer for six samples, the ASC6, which has a sample in-plane rotation mechanism, can be used for sample measurement.

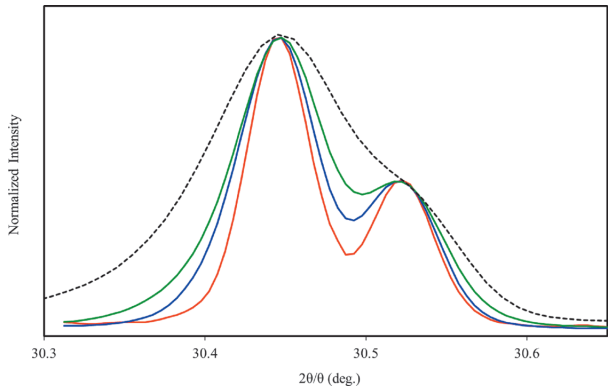


Fig. 2. Comparison of X-ray diffraction profile in high resolution measurement mode. Dotted-line: Standard, Green line: High resolution 1, Blue line: High resolution 2 Red line: High resolution 3

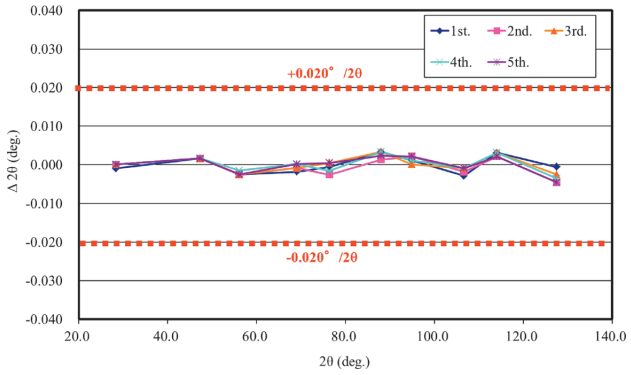


Fig. 3. Angular precision of Si standard reference material using real-time angle correction system. (*Y*-axis ($\Delta 2\theta$) is the difference between the theoretical and the observed angle value. *X*-axis (2θ) is the diffraction angle value of Si.)

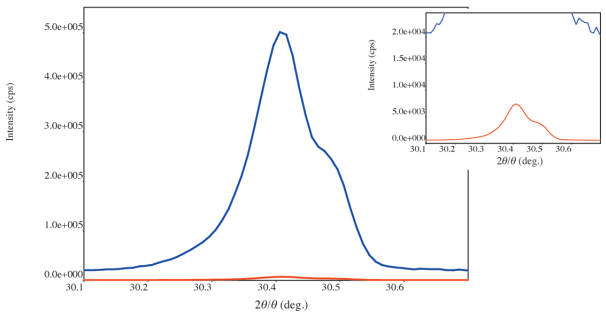


Fig. 4. Comparison of X-ray diffraction intensity between scintillation counter (red line) and D/teX Ultra (blue line).

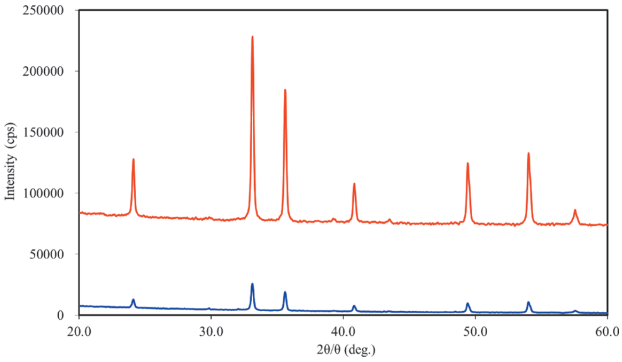


Fig. 5. Effect of high energy resolution with D/tex Ultra. Red line: Window width 17, Blue line: Window width 3.