ISSUE 4 / 2013

PANalytical

X'PRESS

The customers' voice

Easy analysis

The Analytical X-ray Company

Easy analysis – precise and powerful

Welcome to this issue of X'Press, the last one in 2013.

The awareness of our planet's vulnerability and its limited resources is steadily improving. Individuals and companies alike realize that it is essential to exploit nature in an environmentally responsible, effective and efficient manner and to avoid unnecessary waste. Analysis of materials, processes and products is an indispensable requirement for companies in this respect. It is important to analyze as close to the production processes as possible, calling for easy and fast analysis that can be performed by the people working in this production process. In this X'Press we would like to share with you some of our latest examples of such easy and fast analyses that can be executed close to the source.

PANalytical's new Epsilon 1 with its small footprint does not require much dedicated and costly space and can be placed very close to the place where the analysis is really needed. Due to its ease-of-use it can be operated by everyone, enabling dedicated analyses without having to invest in expensively trained operators.

On the other hand, large projects like the Roy Hill mining project (described on page 3), require systems with a high sample throughput for robust and reproducible analysis often operated remotely. This is why IMP Australia chose the collaboration with PANalytical and Herzog to achieve reliable low-cost production of iron ore in this new huge mining project. Analysis even one step closer to the heart of the process is done at LKAB. For online analysis of iron ore this Swedish company is currently placing their new CNA^3 analyzer underground in their Malmberget iron ore mine after having validated the performance of this instrument above the ground first (page 5).

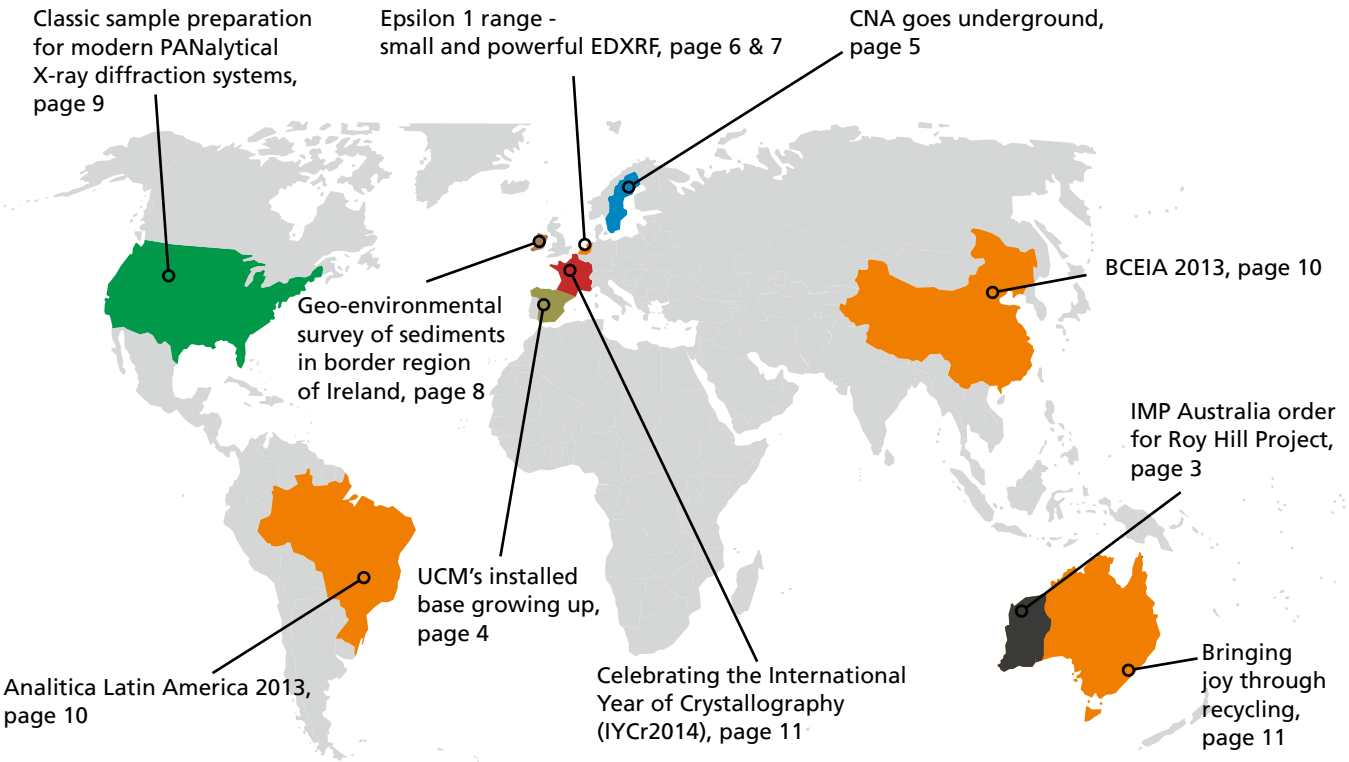
In addition to the instrument performance good sample preparation is also of utmost importance for precise analysis. The US Geological Survey in Denver has adapted a classic method of sample preparation for modern X-ray diffraction systems. When using their modified sample prep method they are able to collect much better data from samples which do not pack well (page 9).

Last but not least I am proud to present PANalytical's participation in the activities for the coming International Year of Crystallography (IYCr2014). As mentioned already in the last issue of X'Press, UNESCO wants to stimulate education and scientific cooperation all over the world, and we are happy to be able to contribute to this goal. We hope to meet many of you at one of the Open Lab events we will organize throughout the year.

I hope you enjoy reading these and more articles in this current X'Press.

I wish you a successful and productive 2014.

Kind regards,
Peter van Velzen



IMP Australia order for Roy Hill Project

When Lang Hancock in the 1950s flew his aircraft over the Pilbara landscape in the north of West Australia, he saw hills that gleamed red, and suspected he'd discovered iron ore. His discovery led to developments that made Australia one of the biggest iron-ore producers in the world. Now his daughter, Gina Rinehart and her family company, Hancock Prospecting are investing around AUD $10 billion in a massive new iron ore project called Roy Hill.

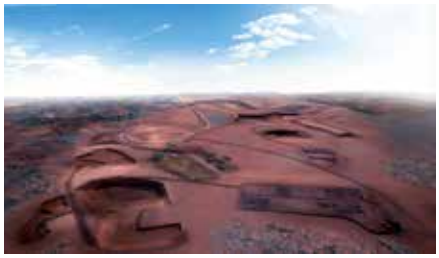
IMP Australia was chosen to provide the laboratory automation (including XRF) for the iron ore analysis at both the new mine and the port facilities. The company works in close cooperation with manufacturers of analytical equipment and system components for logistical interfacing. Over the past few years, IMP has worked closely with PANalytical on many projects to supply XRF analysis systems to interface with their robotic sample crushing and fusion equipment.

This time they decided to use PANalytical Axios systems as these interface well with the IMP and Herzog systems. Additionally PANalytical provides a high level of support, both at installation and throughout the life of the mine.

Following the warranty period of the instruments, PANalytical works directly with the end user in these projects.

PANalytical will supply 3 instruments to Roy Hill: An Axios FAST will be interfaced with the robotic system at the mine lab, with an Axios^{mAX} providing redundancy backup and extra project work. The 2nd Axios^{mAX} will be located at the port facility 344 km away, to ensure the correct grade of ore is loaded onto the ships for export.

All these instruments accomplish a vital task ensuring the integrity of the ore shipped, allowing Roy Hill confidence that their contracts to supply ore of a certain grade are not jeopardized.



Animation of mine in 5 years

The Roy Hill Project in the Chichester Range in the Pilbara region of Western Australia, located 115 km north of Newman and 277 km south of Port Hedland will apply cutting-edge technology, remote operating control and high levels of automation to achieve reliable low-cost production of iron ore. The project is expected to have a mine life of more than 20 years and will consist of:

- 55 Mtpa (million tons per annum) mine, processing plant, airport and permanent accommodation village
- 344 kilometer heavy haul railway linking the mine and port
- Port stockyard and two-berth export facility in Port Hedland
- Corporate headquarters and remote operations center based in Perth (1,272 km away)

Supplying XRF systems to projects like this continue to cement PANalytical as the preferred supplier of analytical instruments to the (Australian) mining industry. Close cooperation of IMP and PANalytical provides systems that are customized to optimally meet the specific needs and preferences of each customer.

This ensures precise, easy and reproducible analysis of the vital properties of the mined ore.

UCM's installed base growing up

The Universidad Complutense de Madrid (UCM) is one of the oldest universities in the world and the top public university in Spain. One of their research centers has recently expanded their fleet of PANalytical systems with 2 new X-ray diffraction (XRD) systems.

The UCM has a number of 'Centros de Asistencia a la Investigación' (CAI), which provide support for researchers; one of them is the XRD/XRF unit. This CAI had already eight PANalytical systems of different generations installed at two sites. The Faculty of Pharmacy owns two diffraction systems and the Faculty of Chemistry works with five different diffraction systems (X'Pert MPD, X'Pert MRD, X'Pert PRO Alpha-1, X'Pert PRO MPD, and a stand-alone generator with a SAXS camera) and one XRF spectrometer (Axios 4 kW). In October 2013 two new diffractometers were installed: an X'Pert Powder and an Empyrean system.

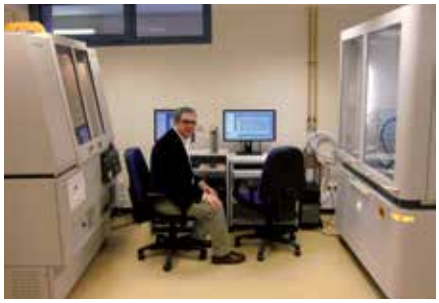
These new acquisitions are part of the project Campus of International Excellence in the 'Materials for the Future' cluster. The cluster aims to develop a network of research groups in the fields of structural and advanced functional materials from both the UCM and UPM (Universidad Politécnica de Madrid). These groups are experts for synthesis and structural and physicochemical characterization of these 'materials for the future'. Their research covers the determination of structure-property relations of some of the hottest topics in materials science and technology, like metallic, ceramic, magnetic and conducting materials, biomaterials and cement-based materials.

The university's XRD/XRF unit can now offer two brand-new applications to their customers: pair distribution function analysis (PDF) and 2D microdiffraction. The pair distribution function analysis can provide very useful nanostructural information about the short- and long-range ordering of the atoms in various material types (crystalline, nanocrystalline, amorphous or liquid). Microdiffraction delivers not only qualitative information of the phases present in small samples or areas, but also micro-structural information regarding preferred orientation of crystallites and the measurement of local stress.

The university's origins lie in the Middle Ages, when King Sancho IV of Castile created the Studium Generale on May 20, 1293.
In 1499 it was converted into a full university by Pope Alexander VI who renamed the institution Universitas Complutensis (after Complutum, the Latin name of Alcalá de Henares, where the University was originally located).

In 1836 the university was moved to Madrid and named Universidad Central de Madrid (Central University of Madrid). It is now located on a sprawling campus in the Ciudad Universitaria district of Madrid.

Currently, the UCM is the largest university in Spain and the second largest one in Europe with about 85,000 students and a staff of 9,500, of which over 6000 are directly involved in teaching duties.



Emilio Matesanz accompanied by X'Pert Powder and Empyrean

XRD specialists Julián Velázquez, Ignacio Carabias and Emilio Matesanz (from right to left) at work in the new lab

The XRD/XRF CAI provides services and technology to other public institutions and private firms, in nearly all experimental scientific areas. The highly qualified CAI technical staff and a modern and high-tech instrumental infrastructure offer an outstanding scientific know-how adapted to the specific needs of their clients.

CNA goes underground

The Swedish company LKAB (Luossavaara-Kiirunavaara AB) is a high-tech international minerals group, world-leading producer of processed iron ore products for steelmaking, and a growing supplier of mineral products for other industrial sectors. The company mines iron ore at Kiruna and Malmberget, in Lapland, Northern Sweden. Their Malmberget mine is one of the largest underground iron ore mines in the world. LKAB has selected PANalytical and its newest CNA development - the CNA³ analyzer - for their online analysis requirements.

PANalytical CNA³ is the latest generation of online bulk material analyzers featuring Sodern neutron technology. This new version has been specifically designed for mining and related industries including iron ore, copper and coal. At first glance, it appears to be a simple black box mounted on a rail, connected to an electric panel via a thick cable. However, this simplicity hides the proven state-of-the-art Sodern neutron technology and PFTNA (pulsed fast and thermal neutron analysis).

PANalytical CNA³ featuring Sodern neutron technology

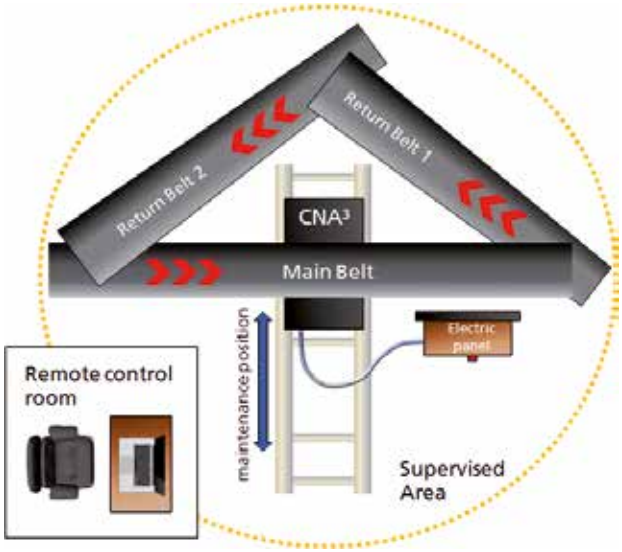
Compared to its larger predecessors common in the cement industry, the CNA³ is more compact and more rugged to handle tougher environments such as underground or open-pit mines.

One of the most notable features of the CNA³ is the 'open top' configuration – there is no upper section of the analyzer mounted above the belt. This feature makes the CNA³ suitable for practically all belt widths, belt depths, speeds or trough angles.

LKAB has completed testing their new system on a commercial scale test platform above the ground. They are now in the process of moving the CNA³ underground at nearly 800 m below surface.

The analyzer will directly measure key elements in the ore to determine the quality of the magnetite on the belt to the processing plant. This information will provide feedback to the mining operation and feed forward information for the processing plant.

A new rail extraction system can extract the CNA³ box from under the belt, even while the belt is running and is fully loaded. This allows maintenance or adjustments to be performed at any time without disturbing the production on the belt. Finally, with no direct belt contact, the CNA³ has no wear parts and minimal maintenance, making it an easy-to-use and very powerful instrument for online analysis.



Test platform with the CNA³ installed (above)

Schematic view of the LKAB test bench setup (left)

Epsilon 1 range – small and powerful XRF

For four weeks in a row the newly launched Epsilon 1 systems were shown at the Gulf Coast Conference in Texas (USA), the BCEIA in Beijing (China), the Fennoscandian Exploration and Mining Conference (FEM) in Finland and at the China Mining Conference in Tianjin. Visitors were attracted by the compact design and superior performance.

PANalytical further extends its benchtop portfolio with Epsilon 1, a low-cost fully integrated XRF analyzer consisting of a spectrometer, built-in computer, touch screen and analysis software. The well designed optical path, a wide range of excitation capabilities and a highly sensitive SDD detector make it a star performer in the low-cost benchtop instrument class.

When higher performance and sample throughput is required, other benchtop XRF analyzers like Epsilon 3 and Epsilon 3 XL are available that operate at higher power and are equipped with a sample changer.

The Epsilon 1 range complements PANalytical's energy dispersive X-ray fluorescence series with four application-specific benchtop solutions. These are cost-effective, factory pre-calibrated packages for a range of key analytical processes. They consist of an Epsilon 1 spectrometer with a validation sample and a starting kit for preparing the first 100 liquid/powder cups for analysis in the fuels and lubricating oils market, mining industry and research and education.

Epsilon 1 for determination of additives in lubricating oils: Compliant analysis

Producers of lubrication oils must also comply with increasingly stringent environmental requirements, like ASTM D6481. Measurement precision is a key requirement of this test method.

Epsilon 1 is the ideal analysis solution for the determination of phosphorus, sulfur, calcium and zinc in unused lubricating oils. Total costs of ownership are typically less than for techniques such as AAS, ICP and wet-chemical methods that additionally require a dedicated skilled operator.

Epsilon 1 for research and education: Flexible elemental analysis

If you want to liven up your college and university classes or enhance your serious cutting-edge research, the Epsilon 1 is an easy-to-operate, compact and X-ray safe instrument.

It provides pre-programmed 'out-of-the-box' XRF analysis from simple element identification and quantification to more sophisticated analysis, like fingerprinting, standardless, and layer analysis, illustrating the ease of X-ray fluorescence analysis.

Self-contained system
Built-in computer with powerful CPU and 120 GB hard drive

Repeatability for years
A low-drift X-ray tube

Maximum sensitivity
50 kV thin window
Ag anode X-ray tube

Focus on easy analysis

Epsilon 1 for determination of sulfur in fuel: Guarantee your fuel production quality

Levels of sulfur in fuels are heavily regulated because of the environmental impact and their influence on engine lifetime. This is reflected in several test methods and standards, like ASTM D4294, ISO 20847, ISO 8754 and IP 496 where energy dispersive X-ray fluorescence (EDXRF) is the specified analytical technique.

On top of the generic EDXRF benefits, such as low operating costs, Epsilon 1 offers out-of-the box norm-compliant analysis. ASTM D4294 is currently the most stringent test method for the determination of sulfur in fuels and Epsilon 1 easily complies with it.

Epsilon 1 for the mining industry: Accurate and safe quantification of rocks and ores

X-ray fluorescence spectrometry is widely used in the mining industry during exploration, mine resource planning and mineral beneficiation. Epsilon 1 easily and precisely determines elemental concentrations of rocks, soils and a wide range of ore types.

The instrument is calibrated with Omnian, PANalytical's market-leading standardless analysis package. Omnian can be used to analyze a wide variety of sample compositions from sodium to americium across the periodic table.

Epsilon 1 can be placed very close to where the analysis is needed, for example in the quarry or mine-site office.

Spillage protection
Shields the heart of the system from spillage

Economical footprint
Compact design requires less than 0.15 m^2 of valuable lab space

Easy operation
LCD touch screen for easy walk-up operation

Geo-environmental survey of sediments in border region of Ireland

The Tellus Border project is an EU-funded mapping project that involves the collection of scientific data on soils, sediments, waters and rocks in the border region of Ireland and the integration of these with existing data from the Tellus project in Northern Ireland. The results provide the region's first cross-border baseline geo-environmental dataset which will support long-term environmental management and mineral exploration activity in an area encompassing 12,300 km². The Geological Survey of Ireland is a partner in this cross-border initiative, which is led by the Geological Survey of Northern Ireland. PANalytical Nottingham Analytical Services was awarded a contract for the multi-element trace-level analysis of 4,400 sediment samples by X-ray fluorescence spectrometry.

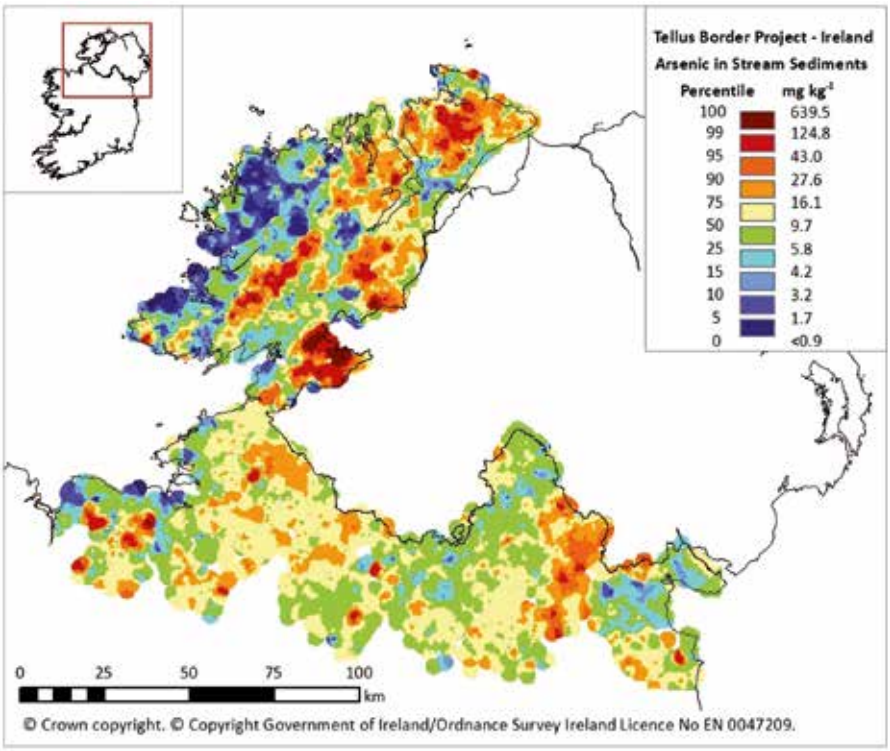
Tellus Border is funded by the INTERREG IVA development programme of the European Regional Development Fund, which is managed by the Special EU Programmes Body (SEUPB) in Northern Ireland, the border region of Ireland and western Scotland.

Using their Axios^{mAX} and Axios^{mAX}-Advanced wavelength dispersive X-ray fluorescence (WDXRF) systems together with their Epsilon 5 energy dispersive X-ray fluorescence (EDXRF) spectrometers PANalytical achieved trace-level determinations for 52 elements in three analytical suites. The implementation of strict quality control procedures to ensure confidence in the results and to prevent cross-contamination resulted in a high-quality dataset suitable for determining a regional geochemical baseline for inorganic elements in sediments.

Between August 2011 and June 2012 field teams working on behalf of the Geological Survey of Ireland collected stream sediment samples from streams using a wet sieving method to obtain a fine fraction sediment sample (<150 µm particle size). Samples were freeze-dried, disaggregated, milled to <53 µm particle size and combined with a wax binder into pressed powder pellets incorporating 12 g of sample material.

At the Geological Survey of Ireland, analytical data has been mapped using sampling location coordinates and an inverse distance weighted interpolation method in ArcGIS, an interactive mapping platform. Resulting maps reveal the influences of regional geology, hydrology and human activities on the chemistry of sediments in small streams.

This data will be joined with that already collected for Northern Ireland and is now available online free-of-charge at www.tellusborder.eu. The data will be of interest to environmental managers and policy makers, mineral exploration companies, agricultural scientists and geological researchers.



Mapped XRF data for arsenic in stream sediments, border region of Ireland

PANalytical

Classic sample preparation for modern PANalytical X-ray diffraction systems

Geological samples can often be challenging to present to the diffractometer in a uniform and randomly oriented fashion – just ask resident geologist Bill Betterton of the U.S. Geological Survey in Denver, CO (USA). Good sample preparation is critical to the results; this is why at his lab, nearly all samples are prepared for XRD using the classic side loading or side-drifting method. This technique has been around for many years, used by National Institute for Science and Technology (NIST) for the preparation of standard reference patterns[1,2].

USGS science for a changing world

This sample preparation method is also required when producing diffraction patterns to be analyzed using RockJock – a free program for calculating the quantitative clay mineralogy from XRD patterns of randomly oriented powder samples[3]. Lab members have adapted this sample preparation method to work with certain sample holders and the spinner stage with sample changer on their X'Pert PRO MPD XRD system.

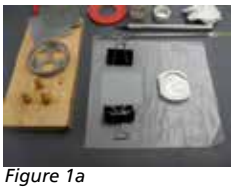


Figure 1a

Figure 1b

Figure 1c

Using the materials shown in Figures 1a&b, USGS places the frosted microscope slide against the cell with clamps to create a cavity to fill from the side opening. When full, they seal this side hole with drafting tape. Average sample preparation time is 4-5 minutes with this method.

A good example to demonstrate the benefit of side loading versus standard back loading (shown in Figure 2) is a geological sample of a soil with high asbestos content. Bill says "Asbestos does not like to pack due to its fibrous nature." By visual inspection, you can see that the (110) peak near 10 degrees and the (310) peak near 28 degrees dominate the back-loaded sample (pattern in blue). The same sample prepared by side loading (red pattern, top) exhibits a pattern with intensities closer to the stick pattern for the asbestos phase.

Other materials well suited to this sample preparation method include coal, clays, micas, feldspars, pyroxenes, amphiboles, carbonates, and gypsum. According to Bill, "high organic content coal (90%) is difficult to pack – the sample is fluffy and tends to heave out of or settle in a back loading holder – but is well suited for preparation by side loading. Many pulverized materials with rod or flake shaped crystals/particles due to cleavage, can benefit from this sample preparation method."

"Minimizing preferred orientation makes a more definitive search/match identification, because more peaks that should be present are there at expected intensities. In quantitative analyses such as Rietveld, fitting will go more smoothly with better goodness of fit," explains Bill Betterton.

In addition, this side-loading holder is a universal mount which can be used to hold a 32 mm round glass slide, for an oriented clay mount or a PW1817/32 round Si zero-background plate. This holder also allows measurements to as low as 0.7 degrees 2θ.

For information on obtaining a USGS side loading holder for your lab, please inquire at info@panalytical.com.

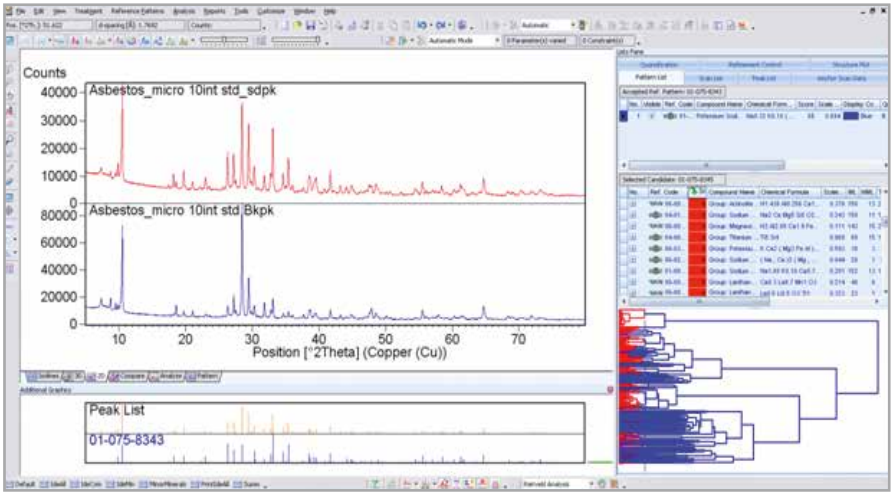


Figure 2

1. **Preparation of Specimens for X-ray Fluorescence and X-ray Diffraction Analysis**, edited by Buhrke, Victor E., Jenkins, Ron, and Smith, Deane K., 1998, John Wiley & Sons, Inc., New York, NY, page 148
2. Jenkins, Ron, and Snyder, Robert L., **Introduction to X-ray Powder Diffractometry, Volume 138 in Chemical Analysis**, 1996, John Wiley & Sons, Inc., New York, NY, pages 247-249
3. The Mineralogical Society of America's web site: www.minsocam.org/MSA/Software

Show time

Analitica Latin America, 24 - 26 September 2013

The exhibition was held in São Paulo at the Transamerica Expo Center. It is the biggest show for analytical instruments in Latin America and is organized every two years.

PANalytical hosted a 64 m² booth with a small conference room. Visitors could get acquainted with the Axios^{mAX} WDXRF system, the XRD Empyrean platform, the new PIXcel^{3D} 2×2 detector, the EDXRF benchtop Epsilon 3 XL system and the Eagon 2 fusion machine. The sales and service team was present and demonstrated the Omnian and HighScore (Plus) software.

A big "thank you" to our guests for their visit. We hope to see you and many more again in 2015.

BCEIA, 23 - 26 October 2013

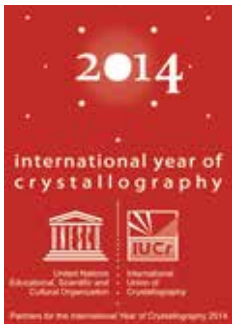
The Beijing Conference and Exhibition on Instrumental Analysis (BCEIA), sponsored by the China Association for Instrumental Analysis and supported by the Ministry of Science and Technology of China was successfully organised for the 15th time. It has been a highly regarded biennial event in academia, similar in magnitude and status as Pittcon in the US.

BCEIA 2013 with the theme 'Analytical Science creates Future' featured a plenary session with lectures delivered by world renowned scientists and 8 sub-sessions for oral and poster presentations. In the exhibition area, PANalytical showed the brand-new systems Epsilon 1 for research and education and the X'Pert^3.

A Chinese version of the 'PANalytical beer challenge' was staged at the booth for the first time. In addition to cans of popular local beer we also displayed a variety of milk powder, toothpaste, and coins to demonstrate the power of Epsilon 3 and the FingerPrint software.

The PANalytical booth attracted a fair bit of media attention as two media companies approached us for interviews and video shoots. Product manager Dr. Harald van Weeren and China country manager Xue Shilei fielded questions from the media and introduced the new products as well as the applications of ScatterX^{78} and Epsilon 3.

Celebrating the International Year of Crystallography (IYCr2014)



As already reported in X'Press 3/2013, the General Assembly of the United Nations has declared the year 2014 to be the International Year of Crystallography (IYCr2014). The IYCr2014 commemorates the award of the Nobel Prize to Max von Laue for the discovery of X-ray diffraction by crystals that marked the beginning of modern crystallography.

X-ray diffraction is a very powerful technique for materials analysis, from applications like geological investigations (mineralogical exploration), to research into novel materials for renewable energy (including batteries, fuel cells and solar cells), pharmaceuticals development, residual stress analysis in engineered components and epitaxially grown thin films for solid-state electronics.

However, the importance of this this powerful technique is not commonly known all over the world yet. Therefore the aim of IYCr2014 is to raise awareness, inspire and attract the interest of young people and promote education and research in crystallography.

PANalytical will contribute by organizing a number of Open Lab events throughout the world. The map shows an overview of these locations.

For more information on the Open Labs, please visit our website www.panalytical.com/IYCr2014.

Some achievements of crystallography

- Structure determination of DNA
- Understanding of computer memories
- Unraveling protein functions
- Helping with design of powerful new materials
- Identification of drugs
- Investigation of cultural heritage artifacts

Bringing joy through recycling

For almost 5 years PANalytical Australia has been donating wood from the packing crates to 'Men's Sheds' - a local non-profit organisation made up of elderly men, mostly retirees who enjoy building and construction.

Originated from a shed in a backyard scenario, one of the aims of Men's Sheds is to improve the overall health and quality of life of elderly males, while helping the local community. Since its inception in 1996, there are now over 850 Men's Sheds across Australia.

Every few months, PANalytical sends a large amount of wood to Men's Sheds or sometimes they would drive a trailer to our warehouse in Sydney to pick it up. With the wood supplied, the men create toys such as doll houses, doll beds and rockers to give to poor children who are not as fortunate as many of us. Thanks to the great work by Men's Sheds they can now own and enjoy handmade toys.

PANalytical Australia is glad to support an organisation that not only enables us to be friendly to the environment but also brings joy to many needy children.

Australian Men's Shed Association

Events calendar 2014, Q1

The list below shows a selection of events where you will find us during the upcoming period. If you attend any of these events please come and visit us.

Date	Event	Location
28-30 January	Thermo Plastics Concentrates	Coral Springs, FL, USA
27-31 January	Southern African Powder Diffraction Conference and Workshop	Johannesburg, South Africa
7-9 February	IFEX 2014	Ahmedabad, India
9-13 February	AXAA 2014	Perth, Australia
23-26 February	SME meeting	Salt Lake City, UT, USA
2-6 March	Pittcon	Chicago, IL, USA
17-20 March	ArabLab 2014	Dubai, U.A.E.
26-28 March	VII Expominas	Quito, Ecuador
21-25 April	Expomin	Santiago, Chile

Please visit www.panalytical.com/events for more information.

The PANalytical webinars

PANalytical has a long tradition of educating and teaching people in the use of our systems and the respective techniques. Alongside with our user courses we have a series of live webinars.

The first one in this series dealt with 'High-pressure and high-temperature XRD studies of hydrogen storage materials' and was presented by PANalytical's Marco Sommariva, application specialist XRD.

Other live webinars in 2013 discussed GISAXS, the new Highscore (Plus), the basics of texture analysis, X-ray reflectivity mesurements, spark emission spectroscopy, non-destructive and easy elemental impurities testing for USP <232>, X-ray diffraction techniques in transmission geometry, and introducing ScatterX^{78} –high-performance SAXS/WAXS.

All these webinars can now be found in the 'Webinar on demand' section on our website and can be watched at any time.

Please visit www.panalytical.com/webinars for more details and for registration.

Colophon

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