

PANalytical PANalytical PANalytical PANalytical

PUSHING THE BOUNDARIES

extending knowledge





The Analytical X-ray Company

Pushing the boundaries, extending knowledge

Welcome to this latest issue of X'Press – the first one of 2013.

At the beginning of a new year it is customary to look ahead at what the year may bring. For PANalytical 2013 marks the beginning of a broadened horizon. As already mentioned in our last X'Press we concluded the acquisition of ASD, a leader in near-infrared solutions. On page 3 we present a short introduction to the technique and how ASD's products serve customers with high-performance analytical instrumentation. ASD's position and reputation in remote sensing, mining and other industrial markets complement PANalytical's offering in scientific and industrial markets by adding a contiguous product line with portable, handheld, benchtop and online products to our portfolio. I am excited to welcome the ASD crew to our team.

One of the areas where ASD's expertise will add to our offering is beneficial in the mining industry. This edition of X'Press features recent developments in mining in Scandinavia and Poland. At CBJ Poland, a service lab uses 12 PANalytical XRF systems for analysis of samples derived from copper production processes carried out at the mines and foundries of KGHM Polska Miedź S.A., as well as for the analysis of a broad range of geological samples.

In Finland, Talvivaara Mining struggled with uranium levels that were too low for detection using the established fused bead procedure. The answer came in a specially tailored version of PANalytical's Pro-Trace analytical package. The Oulu Mining School employs Axios^{mAX} in their unique MiniPilot plant offering an innovative environment for education and research of minerals processing.

Many of our industries are undergoing substantial and rapid change. New talent will be a main driver when



applying their newly found knowledge in ever more creative ways. Because we at PANalytical believe that we have a responsibility to support these future thought leaders, we created the PANalytical Award. I am happy to announce that the first winner of this Award is Thomas Bennett, an extraordinary young man who has made significant advances in the analysis of amorphous materials by a combination of analytical techniques and methods. I had the pleasure of personally informing Dr. Bennett that he had been selected as the Award winner. A great achievement given the strong field of competition in this year's PANalytical Award.

Please enjoy this issue of our X'Press magazine and let me once again thank you profoundly for your continued confidence in PANalytical. For more information about our products and services please visit www.panalytical.com and I look forward to your suggestions at info@panalytical.com



With best regards, Peter van Velzen



Welcome to ASD and NIR

ASD Inc. is excited to join the PANalytical team. Founded in 1990, ASD designs, manufactures, and sells sophisticated near-infrared (NIR) instrumentation solutions and applications expertise for materials measurement and research. ASD is recognized worldwide for providing high-performance analytical instrumentation solutions to industrial and scientific markets.

Our portable, accurate and rugged instruments convert measured data into actionable information to help optimize processes or improve research.

In the mining industry, ASD's NIR instrumentation solutions are a natural pair for XRF and XRD analyzers. Mining customers often desire both the molecular and elemental information. ASD's instruments provide customers with insights into the minerals, which indicate the presence of copper or gold, or minerals such as talc or clay that could slow the metal recovery process.

The instruments are handheld and can easily be used on-site for fast testing. In this way customers profit from faster exploration of economic deposits, more visibility into the mining processes and increased mine productivity and ore yield. In addition to providing topof-the-line NIR spectroscopy instrumentation, ASD also offers full service solutions with software packages and chemometric modeling services.

ASD's legacy is one of innovation and leadership in addressing customers' needs – providing the latest technologies combined with the highest standards of quality and service. We continue to provide customers with valueenhancing solutions to their materials measurement needs and look forward to building on this rich history as part of PANalytical.







IS NOW





A user ground truthing a field using a handheld instrument.

NIR and its benefits to PANalytical customers

Simply put, NIR bounces light off a surface, and the spectral response is the measurement. NIR examines the molecular properties of a material such as moisture content, fat, protein, or types of minerals. This is different from XRD or XRF instruments that detect elemental components like calcium, gold or copper.

Benefits of near-infrared (NIR) technology

- Non-destructive
- Measure materials in a wide range of forms
- Requires little or no sample preparation
- Can be used to analyze multiple constituents in a single scan

NIR spectroscopy is applicable in a wide range of industries including:

- Raw materials analysis
- Pharmaceutical
- Food

ASD's two core markets are comprised of remote sensing and mining exploration and production.

Proven quality for Centrum Badań Jakości Ltd.



Centrum Badań Jakości Ltd. (CBJ sp. z o.o.) in Lubin, Poland, provides comprehensive services for various physico-chemical and microbiological tests, analyses and measurements, including sample collection and preparation, along with evaluation of the obtained results.

CBJ sp. z o.o. is one of the largest testing laboratories in Poland with approximately 500 employees in total and laboratories in the cities of Głogów, Legnica, Lubin and Polkowice. For years the company has been using and perfecting a certified Integrated Management System that conforms to the requirements of the PN-EN ISO 9001 standard.

Over 1000 analytical methods have been accredited by the Polish Centre for Accreditation, including analyses of materials derived from copper mines and foundries, solid fuels, food, water, waste, sediments, soil and emission measurements. Microbiological tests and analyses (e.g. of food and water) are carried out as well as sample collection for environmental tests and copper concentrates.

One of the most common analytical techniques used by CBJ sp. z o.o. is X-ray fluorescence spectrometry, which allows a quick, non-destructive analysis of multiple elements in solid and powdered materials. In its laboratories CBJ houses a large number of sequential WDXRF and EDXRF spectrometers from PANalytical. At present, CBJ sp. z o.o. utilizes 12 XRF spectrometers for its measurements e.g. PW2400, MagiX, Axios^{mAX}, Axios 3 kW, MiniPal 4 and Epsilon 3 XL. The systems are used for the analysis of samples derived from copper production processes carried out at the mines and foundries belonging to KGHM Polska Miedź S.A., as well as for the analysis of geological samples.



The results from these analyses are used to optimize technological processes and to evaluate materials obtained at every stage of production.

Additionally samples are analyzed using Omnian, PANalytical's program for semi-quantitative standardless analysis, allowing a preliminary determination of the chemical composition of unknown samples. In order to provide optimal results, the company regularly participates in proficiency testing and inter-laboratory comparison programs.

Besides expanding the range of services offered, CBJ sp. z o.o. is constantly collaborating with both domestic and foreign scientific research institutions in order to exchange knowledge and to improve reference materials and measurement techniques.



Axios goes for gold at the Perth Mint

Recently the Perth Mint invited PANalytical to demonstrate the capability of the Axios XRF (X-ray fluorescence) system for the analysis of fine gold. The Perth Mint dates back to 1899 and operates one of the largest refineries in the world, refining gold mined in Australia, New Zealand and Papua New Guinea.

Besides ICP-OES and AAS, gravimetric fire assay has been the industry method of choice for gold analysis for more than a century. Today however, customers are seriously considering X-ray spectrometry in an attempt to maximize analysis efficiency while maintaining the tight quality and high accuracy of the results. Could the PANalytical team persuade one of the world's most reputable mints to convert to XRF?

XRF analyses fine gold by the difference method. This measures major and minor impurities, such as silver, copper, nickel and iron. It achieves faster analysis time with lower labor costs and can be run unattended at night. The risk of exposure of staff to lead and lead oxide waste, a poisonous by-product of the fire assay method is greatly reduced as well as the need to recover precious metals which may have been dissolved for ICP analysis.

The bullion coins and bars produced at the mint come from 99.99% pure gold. The coins themselves are analysed at a minimum purity of 99.5%, setting the bar high for the Axios system. It performed as expected, offering within 0.006% accuracy to the gravimetric fire assay method. Consequently, these results went on to mean that the Perth Mint commissioned an Axios^{mAX}-Advanced system to be installed with a range of additional operational extras.

"The XRF results are in very close agreement with the fire assay values," remarked Analytical Services Manager, Michael Koch. "We made the right decision to go for the PANalytical Axios^{mAX}-Advanced system." Michael Koch, Manager Analytical Services and Tracy McNab, Senior Scientist

Although conventional fire assay is still the industry standard, XRF use in refineries is predicted to expand significantly. It is estimated that eventually 75% of fine gold analysis will be performed with X-ray spectrometry. The Axios system will also be set up for analysis of silver in doré bars, refinery recoveries from scrubber residues, and analysis of platinum group elements.



Calibration for silver shows excellent accuracy.

A visit to The Perth Mint refinery is a rare privilege. Following a tour of the facility visitors are instructed to empty their pockets before walking through a high security area - complete with X-ray scanners and metal detectors.

Even more stimulating are the guided tours offered to visitors at the city heritage premises. You can watch the amazing spectacle of a traditional gold pour, see the world's largest gold bar exhibition, the world's largest gold coin (1 tonne) and the second largest gold nugget still in existence.



A fruitful partnership at Talvivaara Nickel Mine

Collaboration of Talvivaara Mining Company and PANalytical began in 2008 with the purchase of an Axios WDXRF system. The partnership between the companies moved to a new level in 2012 as innovative methods for uranium analysis were developed and installed.

Initially, the Axios spectrometer was used to analyse geological samples (black schist) from exploration. Due to strong mineralogical effects found during the evaluation period, a fused bead process was chosen as the main sample preparation method. This guaranteed repeatable results and was applicable for all geological samples. At a later stage the analysis of end products was included and a measuring program for liquid analysis (bioleaching solutions) was calibrated and successfully introduced.

In 2010 the company started to investigate the possibility of uranium production as a by-product of its other operations and subsequently, they began a project to extract uranium from the bioheapleaching solution, in which it leaches from the ore like other metals. As Talvivaara's interest grew it was natural for them to look to PANalytical for help developing appropriate analytical methods.

The problem was that uranium levels were too low for detection using the established fused bead procedure. The answer came in a specially tailored version of PANalytical's Pro-Trace analytical package.

PANalytical's Application Competence Center in Almelo together with the team in Finland agreed on specifications for the setup. The average metal contents in Talvivaara ore are 0.22 % Ni, 0.50 % Zn, 0.02 % Co, 0.13 % Cu and 0.0017% U. Large deposits with open pit mining and bioheapleaching make it possible to extract nickel profitably, demanding four additional jump-edge calibration sets as well as measuring condition fine tuning, and special attention to the choice of the background position and adapted selection of the MAC (mass attenuation coefficient) calculations. Usually installed for high-power 4 kW spectrometers, using Pro-Trace with the 2.4 kW Axios at Talvivaara meant additional changes were also needed.

In the Spring of 2012, a large number of samples were sent from Talvivaara for the Pro-Trace calibration trial. This setup was transferred to the Talvivaara site in June 2012. A two-week visit from PANalytical experts ensured the new uranium analysis process was working well. The customer's Axios software was upgraded to SuperQ 5 with complete calibration for the Omnian standardless program and Pro-Trace application.

At the same time, Talvivaara also invested in an Epsilon 3 XL EDXRF spectrometer for the analysis of uranium content in organic liquids (from the uranium recovery). As part of the installation visit standards were prepared, the application was set up, and users at Talvivaara were trained. They are now able to execute fast, easy and reliable measurements of uraniumcontaining solutions (both aqueous and organic) and of pressed pellets.



From left to right: Mr. Hannu Lahtinen, Mrs. Jonna Jokiniemi, Violeta Uricanu (PANalytical) and Mia Nygård

Mining in Finland



Bioheapleaching

Bioleaching is the process of leaching metals from ores through exploiting a naturally occurring bacterial reaction. By optimizing the physicochemical and microbiological process parameters such as temperature, pH, oxygen and nutrition levels, metals can be leached at an increased optimum rate.

The ore is ground into particles less than 8 mm and piled into large heaps. Leaching fluid laden with high levels of the naturally occurring bacteria is trickled into the heap and oxygen is pumped through. In these conditions the bacteria eat the ore thus separating the metals which are then leached into the liquid run-off.

The bacteria used in the Talvivaara bioheapleaching process are naturally growing in the ore and, endemic to the area, are well adjusted to the prevailing environmental conditions.





Oulu Mining School is the network which takes care of the education and research in the mining and mineral field at the University of Oulu, Finland.

Core partners are the Department of Process and Environmental Engineering, the Department of Geosciences and the Extension School. Active co-operation is done with Aalto University (formerly Helsinki University of Technology) and Luleå University of Technology, Sweden, within the Nordic Mining School. It also works closely together with research institutes (Geological Survey of Finland and the Finnish Institute of Occupational Health (FIOH)) and mining companies such as Pyhäsalmi Mine, Agnico-Eagle Kittilä Mine, Talvivaara Mine and Outokumpu Chrome Mine.

PANalytical is the main supplier of analytical equipment for the MiniPilot plant.



Fast analyses for the Oulu Mining School

The University of Oulu (Finland) is an international science university founded in 1958, with a research and education community of 16,000 students and 3000 employees, and one of the biggest and most multidisciplinary universities in Finland. It houses the Oulu Mining School who have recently invested in PANalytical X-ray fluorescence (XRF) equipment and purchased an Axios^{mAX} WDXRF spectrometer and an Eagon 2 automatic bead machine.

PANalytical already has a large installed base in the mining field of the Nordic area and this was one of the selection criteria to become a supplier of XRF equipment. The spectrometer is connected to the process management system of the MiniPilot plant of the Oulu Mining School assaying the feed ore, concentrates and tails of the beneficiation process and enabling the process control and grade and recovery reporting via a Schneider Ampla[™] manufacturing execution system.

The MiniPilot plant of the Oulu Mining School is the world's first continuous university-based pilot-scale concentrator and is installed at the Department of Process and Environmental Engineering of the university. The small-scale research and minerals processing plant is designed for a learning environment. It represents the concentrating process of the Pyhäsalmi mine in Finland on the scale of 1:5000 offering an innovative environment for education and research of minerals processing unit operations.

With this continuous plant up and running the need for getting analysis results within a short period of time increased considerably. Typically a result from a commercial laboratory can only be expected within a few days or even weeks. Thus, the department felt it would recquire a XRF spectrometer of its own including effective sample preparation and trained operators. This allows them to shorten the time needed from sampling to an accurate analysis report to a quarter of an hour making research work much more efficient with fewer experiments based on gut feeling only.

The PANalytical Axios^{mAX} is now used for the analysis of a wide range of environmental engineering and mineral processing sample matrices. Sample preparation facilities are equipped for most material types: rocks, ashes, refractories, metal specimens, loose powders and liquids.

An important part of the investment was personnel training carried out by qualified instructors from PANalytical on spectrometer use, application development and sample preparations for different applications.



ScatterX78 – the new high-performance SAXS/WAXS attachment

The new PANalytical ScatterX⁷⁸ attachment was launched last November at the 15th International Small-Angle Scattering Conference (SAS-2012) in Sydney, Australia. Held once every three years in different places worldwide, the event attracted about 300 attendees from more than 20 countries. All aspects of small-angle X-ray and neutron scattering were discussed.

ScatterX⁷⁸ is the latest add-on for PANalytical's Empyrean multipurpose X-ray diffraction platform. It allows for high-performance small-angle X-ray scattering (SAXS) measurements with fast data acquisition, and it can be used even for highly diluted and low-contrast samples. ScatterX⁷⁸ consists of a compact and easy-to-use vacuum chamber that houses a variety of sample holders and advanced modules for conditioning the X-ray beam.

SAXS is a versatile tool that is used for the dimensional and structural analysis of virtually all types of nanomaterials. ScatterX⁷⁸ also allows for basic 2D SAXS measurements which is of particular interest for the characterization of anisotropic samples.

"Advanced SAXS measurements normally require dedicated and often large instruments which tend to be difficult to use and are thus only accessible to real experts in the field," remarked PANalytical product manager Jörg Bolze. "With the unique, alignment-free ScatterX⁷⁸ attachment this technique is now available as an advanced, yet cost-effective research tool for nanomaterial research laboratories equipped with the multipurpose Empyrean diffractometer."

With ScatterX⁷⁸ SAXS measurements can be extended to higher scattering angles up to 78 degrees. These wideangle scattering (WAXS) data provide complementary information about the crystalline phases and the size of nanocrytals.



As an application example, the figure shows SAXS data that were acquired with ScatterX⁷⁸ from aqueous surfactant systems at different low concentrations. The characteristic SAXS profiles allow to easily and quickly differentiate different aggregation states of the surfactant molecules, such as micelles and liquid crystalline phases.



Association behavior of amphiphilic molecules





Winner of the first PANalytical Award: Thomas Bennett

The inaugural PANalytical Award of 2012 has been won by Dr. Thomas Bennett, affiliated to the Department of Materials Science at the University of Cambridge, UK. The prize was based on a research article published in Physical Review Letters (104, 115503, (2010)), entitled: "Structure and Properties of an Amorphous Metal-Organic Framework". As entrant and first author on the publication Dr. Bennett is to receive the PANalytical Award trophy, a certificate and € 5,000.

The PANalytical Award initiative was launched to reward and encourage exceptional young researchers who are most affected by the reduced government research spending in many regions. PANalytical has sought to provide a substantial, but more importantly a meaningful injection to the career of such an outstanding scientist.

Researchers that have never held a professorship and who use laboratoryscale X-ray equipment as their primary analytical technique were eligible to apply for the award. Entries for the award could be submitted during the course of 2012 and had to have been published in the period between 1 January 2010 and 30 September 2012. The winning article was chosen by a committee composed primarily of independent researchers, unaffiliated to PANalytical and was selected from a strong field of entries submitted from across the globe.

A key feature of Dr. Bennett's work is the interpretation of X-ray total scattering data (PDF). In conjunction with a variety of other techniques such as neutron scattering, electron diffraction, optical microscopy and computer modelling the authors provide a cohesive picture of the metal-organic framework under investigation. The selection committee was impressed by the level of understanding that Dr. Bennett and his co-workers were able to extract from combination of analytical techniques.

The study represents a significant step towards understanding this kind of amorphous materials, a realm that workers in the field of X-ray diffraction have found discouraging since the technique was established a century ago. Dr. Bennett comments on his research: "The real promise here is that we can introduce chemical functionality into the crystalline material, before amorphization into a glass-like one. This is most likely the route to functional amorphous materials such as electroluminescent and optically active glasses. Part of the battle however will be getting people to look past the traditional boundaries of needing a crystalline material to work with."



Thomas Bennett studied Natural Sciences (Chemistry) at Trinity Hall, University of Cambridge, graduating with MSci (Hons) in 2008. He continued his studies at the Department of Materials Science and Metallurgy in the University, researching the thermo-mechanical properties of porous framework materials.

The characterization, properties and applications of amorphous metal-organic frameworks featured prominently in his PhD, awarded in 2012.

XRD at the museum (3)

The National Museum of Wales in Cardiff (UK) is third in a series in X'Press about museums in the United Kingdom who have recently invested in PANalytical X-ray diffraction systems to advance their research programs.

The National Museum of Wales was established by Royal Charter in 1907 and now has 8 sites (7 museums and a collection center) throughout Wales. Now known as Amgueddfa Cymru – National Museum Wales, a shorthand version of its mission statement is 'showing more treasures to more people': an expression of a real desire to give greater access to the museum's collections and to use them to the full as invaluable resources for lifelong learning.

Wales has a long history of mineral extraction from the beginnings of metalworking by humans until the present day. The mineralogy behind the metal ores is an important area of scientific investigation undertaken by Amgueddfa Cymru.

One of the public services in the museum is to help to identify mineral and artefact specimens found by members of the public in Wales. As Wales is such a geologically varied and interesting country, the number and range of finds is served well by the use of X-ray diffraction.

Tom Cotterell, a mineralogist in the department, has used PANalytical's X'Pert MPD to help identify a number of new occurrences of rare mineral species in Wales. His current research topic is to study the mineralogy and provenance of lapis lazuli specimens in the museum's collection (as well as samples from other collections). This striking blue mineral was commonly used in paint pigments and each worldwide occurrence has a typical mineral assemblage which can, it is hoped, indicate its provenance. Even the final paint pigment from the canvas of an art work can carry this unique signature – so indicating the source of the pigment and in some cases perhaps even the authenticity of the painting. Tom has already identified some fake specimens of lapis during his investigations!

Another very topical topic investigation involves the detailed mineralogical analysis of the bluestones of Stonehenge. It is well known that some of the huge monoliths at Stonehenge originated in the Preseli Mountain area of Wales. Using modern techniques including XRD, it has been possible to pinpoint very narrow geographic areas from whence the stones most likely originated by matching their mineralogy exactly.

Work is still ongoing to try to establish if field investigations can identify possible quarrying activities in the areas with the same petrology.



X-ray diffraction equipment at Amgueddfa Cymru - National Museum Wales. This enables rapid, and non-destructive analysis of a wide variety of specimens.





Empyrean for Vale

Vale is one of the world's top mining companies headquarted in Brazil and maintaining operations in 37 countries. Their Centro de Tecnologia de Ferrosos (CTF) promotes the technological development of iron ore products for the steel industry. The center is located in Nova Lima in the Minas Gerais State, a huge mining area in Brazil.

At the end of 2012 a PANalytical Empyrean system was installed at a new laboratory that was built to support the minerals research for all mines of Vale. In January 2013 the users were trained on-site in this new lab.

The laboratory has more modern equipment for chemical analysis, sample preparation, metallurgy, and materials characterization, focusing on the measurement and characterization of iron ores. The Empyrean diffractometer together with a Mössbauer spectrometer will help Vale in the advanced research of compounds characterization. X-ray diffraction and the HighScore Plus software will allow analysis of a large number of samples with the Rietveld method and also cluster analysis.



From left to the right: Training attendees Elen Eller, Valdirene Resende, Alei Domingues and Márcia Blandina; Dr. Luciano Gobbo (PANalytical XRD Senior Application Specialist)

As the user Valdirene Resende said: "The Empyrean has an excellent performance with a high-level automation, providing X-ray diffraction data of excellent quality." She added that "HighScore Plus is a very 'friendly' software for the users of X-ray diffraction."

PANalytical takes a week of workshops to Turkey

November 2012 saw PANalytical and their Turkish representatives Atomika Teknik take a tour of Turkey with a week's worth of workshops and seminars introducing X-ray analytical techniques to those not familiar with X-ray diffraction (XRD) and X-ray fluorescence (XRF).



ÜNİVERSİTESİ

Eczacibaşı The week began in Istanbul with a seminar on the basics of XRD and XRF presented to representatives from more than a dozen cement factories. A visit at Eczacıbaşı Esan, a long-standing mining customer of PANalytical, ended this exciting day.

> The next day students and academics from across Turkey's 220 universities attended a basic introduction to XRD at Istanbul's Bilecik University. The seminar was well received and ended with an introduction to the Rietveld quantification method. Subsequently approximately 30 participants enjoyed the next day's 'hands-on' Rietveld workshop which was judged a resounding success.



After a well-earned day of rest the team travelled to Bilkent University, in Turkey's historic capital Ankara, where the final event of the week took place at the National Nanotechnology Research Center (UNAM). PANalytical's Martin Tremblay introduced the application of thin film analysis and gave demonstrations of hardware and software. The enthusiasm from the seminar spilled over into the lunch break with students engaging the team in questions and discussions.

The levels of enthusiasm and appreciation observed at the workshops contributed greatly to the week's success. With plans to build on and expand this success in 2013, PANalytical's Turkish trip was really just the start...



Events calendar 2013 - Q1/Q2

You will find us at the following events during the upcoming period. If you attend any of these events, please pass by and visit us!

Date	Event	Location
March 17-21	Pittcon	Philadelphia, PA, USA
March 18-20	Building Materials Conference 2013	Halle, Germany
April 1-5	MRS Spring	San Francisco, CA, USA
April 14-18	IEEE-IAS / PCA Cement Industry Technical Conference	Orlando, FL, USA
April 16-19	Analitika Expo 2013 / A-TESTEX 2013	Moscow, Russia
April 24	BCA IG Meeting	Leicester, UK
April 24-26	CEMENTTECH 2013	Beijing, China
May 20 - 24	PPXRD 2013	Beijing, China
June 4-7	Forum Labo & Biotech	Paris, France
July 1-5	ICMAT 2013	Suntec, Singapore

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

Pittcon 2013: March 17 - 21, Philadelphia

Pittcon is the largest and most comprehensive annual conference and exposition on laboratory science and instrumentation in the world. PANalytical will be introducing several new products and services at this event, and will have on display many of our products for attendees to experience first hand. In addition, a live Epsilon 3 XL benchtop XRF system will be running samples in real time during the exhibition.





Colophon

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Data subject to alteration without notice. This customer magazine is printed in the Netherlands on 50% recycled, chlorine free paper and is published three times per year.