NORWEGIAN CHEMICAL SOCIETY

DIVISION ANALYTICAL SCIENCES

PROGRAM
THE 21st NORWEGIAN X-RAY CONFERENCE

5-7 SEPTEMBER 2022
Strand Hotel Fevik, Grimstad

Monday 5 September

0800 REGISTRATION opens in front of the conference hall at Fevik Strand Hotel
Distribution of program

0900 Training session HH-XRF Maggi Loubser **Chair: Alf**
Short course title: How to Safely, get the best data out of your HH-XRF

1100 Exhibition Opens

1200 LUNCH

1300 Opening: Elke Thisted, Leader of the Norwegian X-ray Conference Committee

Session: HH-XRF **Chair: Susanne**

1315 **Handheld XRF opening a new world in Heritage Conservation.**

Maggi Loubser - *Tangible Heritage Conservation Program, School of the Arts, University of Pretoria, South Africa* – Invited speaker

After specialising in X-ray Fluorescence spectroscopy, I thought I had the technique pretty much mastered, and then I got drawn into the world of Heritage Conservation and using handheld XRF as tool in this field. I quickly learned a whole new set of problems and limitations associated with this new field and had to engage on a steep learning curve to learn how to optimally analyse material that cannot be sampled representatively and homogenously and quantified in the traditional sense.
I also learned that this did not make the data any less valuable, I just needed a paradigm shift from quantitative to qualitative analyses. Apart from the obvious application of researching the materiality of specifically African Artists (of which almost no such data is available), and which is not just crucial when trying to conserving an artwork, but also plays a major role when trying to determine the authenticity of an artwork, many new applications revealed themselves to me on this new adventure.
It started with a Rembrandt that turned out not a Rembrandt, then moved onto bronze sculptures by Dutch born South African artist Anton van Wouw, Alexis Preller's paintings and Lucky Sibiya's polychrome wooden sculptures. As students came along with new projects many other practical applications saw the light; technical questions like the specific tanning processes for leather articles, glass colourants like Uranium glass or Paris green and even an in-depth study of the composition of the pewterware on the oldest shipwreck along the African coast.
One of my current projects is a study with a PhD candidate on toxic insecticide residues on the ornithology and mammalogy collection of the Zimbabwe Museum of Natural History. This is old-hat for the Northern Hemisphere, but the first study of its kind in Africa.

In this presentation I will tell the story of many case studies to demonstrate the diversity of using handheld XRF in Conservation Science, but also discuss the challenges and difficulties in interpretation of the data.

1400 **Why you should look at your spectra, and why you should want to**
Michelle Taube - *National Museum of Denmark*

When the National Museum of Denmark bought a handheld XRF in 2008, we were one of the first cultural heritage institutions in the country to have one. At the time, it was expected that professionals in cultural heritage would run the instrument from the computer and interpret the raw spectra to determine which elements were present in a given sample. We bought a second instrument in 2019. In the intervening time, the handheld market had expanded, and the manufacturers had moved more to a point-and-shoot approach. Although manufacturers still advertise handheld instruments for cultural heritage, the control software is harder to use and the raw spectra are difficult to access, which leads users to believe that it is enough to purchase a number of calibrations and then accept the numbers shown on the screen. This talk will present a number of case studies that show how the calibration methods work and why it is almost always necessary to take even a quick look at the spectra themselves to fully understand the results and the material investigated. It will also present cases that highlight the additional information that can be gleaned by taking a closer look at the spectra.

1420 **XRF-analysis of pigments, inks and fillers in hand colored incunable**
Karen Mengshoel - *Norwegian Institute for Cultural Heritage Research (NIKU)*

A hand coloured incunable (1493), now in Christiania Kathedralskoles Bibliothek was analysed using HH/XRF. The use of pigments, dyes and fillers were documented and compared to those on a hand coloured print, also in the same collection. The aim was to support the theory that they came from the same publishing house in Northern Germany. The investigations also included analysis of the leather binding. The talk will present the case study and discuss its results.

1440 **Elemental Mapping and Multivariate Data Analysis using Portable XRF Data: Applications to the Study of Baroque Epitaphs in Stavanger Cathedral**
Kidane Fanta Gebremariam - *Arkeologisk museum, Universitetet i Stavanger (UiS)*

Portable X-ray fluorescence spectroscopy (pXRF) is a powerful multi-elemental analytical technique well suited for fast, non-destructive, non-invasive, in-situ and on-site analyses. In the case of paintings, such instruments are routinely applied for the qualitative identifications, usually in tandem with complementary analytical techniques. However, the data gathered can be used even in a more informative manner considering its quantitative features. These are often overlooked in the investigation of paintings using pXRF instruments. They can be used for mapping of the spatial distribution of the chemical elements of interest from systematically selected spots of the paintings.
More recently, macro-XRF instruments are being increasingly applied for high resolution mapping of diverse paintings. However, such instruments are very costly and are not easily accessible to many museums and heritage institutions engaged in the study and conservation of paintings. In that context and view of diverse paintings in different settings and sizes, the benefits of using pXRF for elemental mapping purposes are of paramount importance. Moreover, the quantitative data acquired can be subjected to statistical multivariate data analyses, extracting very useful information about the correlation between the chemical elements and the spots of analyses from same and different paintings. These results can be displayed in a graphical manner for simplified presentation of the large multi-elemental data and enhanced interpretations. Besides being crucial in documentation of the alteration products, painting materials used originally and later, such approach can shed light in many aspects of the paintings. It facilitates art historical investigations and conservation interventions among other applications. In this contribution, some of the results of the XRF analyses on the Baroque epitaphs from 1660-1675 located in the Stavanger Cathedral will be presented. The cases illustrate what can be acquired from the spatial distributions of specific chemical elements on the paintings and the statistical multivariate data analyses.

1500 Coffee, poster session and exhibition

1630 Departure for social trip to Næs Ironworks Museum www.jernverksmuseet.no
Næs Ironworks Museum in Tvedestrand is the site of a unique premodern iron works dated back to 1665. Most of the industrial buildings, including machinery and equipment, and some of the workers' houses are preserved or have been reconstructed. Today the old ironworks is a unique museum.

2030 DINNER (Fevik Strand Hotel)

Tuesday 6 September

Session: XRD **Chair: Petter**

0830 **University Center for X-Ray Diffraction Analysis, RECX - Examples of Services for Industry and Research**
Helmer Fjellvåg - *Universitetet i Oslo* - Invited speaker

Professor Helmer Fjellvåg will give examples of the use of XRD methods for analyzing and quantifying industrial materials. Helmer Fjellvåg is in charge of RECX, the Norwegian national center for x-ray diffraction infrastructure. This is a service institution that receives samples for analysis, supports analysis and interpretation and also allows users to have access to the facility instrumentation for running on-site studies. RECX has a wide range of instruments for recording diffractograms at ambient, at low or high temperatures, in operando, on thin films, single crystals and SAXS. Required sample material can be in the range of milligrams to grams depending on material type, analysis method and sample presentation.
RECX is located at the University in Oslo and at the Norwegian Institute of Technology in Trondheim. For Hydro Aluminium an example of use is the analysis service, sending pre-prepared samples and getting the recorded diffractograms in a format of choice back. Throughput has been good.
X-ray diffraction is an under-used technology in Norwegian and Nordic industry and the NKSR Conference recommends this as an opportunity to meet and learn about a central institution that can be your key to further your application of XRD.

0915 **Using X-ray powder diffraction with Rietveld to determine crystalline components in respirable dust collected from renovation of brick and concrete buildings.**
Johanne Østereng Halvorsen og Torunn Kringlen Ervik - *Statens arbeidsmiljøinstitutt*

Construction workers are exposed to a complex mix of inorganic dust and other components. Exposure to respirable crystalline silica (quartz), has known adverse health effects like silicosis and increased risk of lung cancer. Workers who conduct interior demolition are particularly at risk as renovation and demolition can produce large quantities of dust in areas with low ventilation. Crystalline silica content in respirable air samples can be determined by XRD by using the NISOH 7500 method where standards of are used. The Rietveld method has been assessed as a simultaneous multicomponent analysis to determine crystalline and amorphous components in inhalable dust samples. The aim of this work is to determine the inorganic components present in the respirable dust fraction using Rietveld as a multicomponent analysis with XRD. Respirable dust from workers demolishing brick and concrete materials has been collected from different projects. Gravimetric analysis of 67 respirable dust samples gave concentrations of 1.34 mg/m3 (min 0.028 mg/m3 to max 6.0 mg/m3). The Norwegian occupational exposure limit (OEL) for respirable nuisance dust is 5.0 mg/m3. Initial determination of quartz content by XRD reviled an average respirable quartz content of 0.26 mg/m3 (n=14), with a minimum of 0.0061 mg/m3 and a maximum of 0.62 mg/m3. The respirable dust contained on average 11 wt% quartz. It shows that the respirable quartz exposure levels may be exceed OEL, 0.05 mg/m3 for respirable quartz, although the total dust exposure are below the OEL. Applying a multicomponent method such as Rietveld to respirable dust samples may be useful to better understand the complex workers' exposure.

0935 **Overcoming fluorescence and other developments in X-ray powder diffraction instrumentation.**
Markus Valkeapää – *Panalytical*

For X-ray powder diffraction (XRPD) measurements, it is beneficial to be able to choose an X-ray tube to suit the requirements of analyte material. Typical choices include Cr, Mn, Fe, Co, Cu, Mo or Ag as anode elements. The choice depends upon the requirements of the measurement. For example, spreading a diffraction pattern using low energy emission (e.g. Cr, Mn) provides improved peak separation for organic materials, whereas condensing a pattern by using a high energy emission (Mo or Ag), provides full total scattering data for Pair Distribution Function analysis and high sample penetration.In an XRD experiment, the optics and the detector are tuned to the required narrow energy window. Malvern Panalytical's 1Der strip detector can be automatically tuned to provide a narrow energy window around any chosen diffraction energy. This provides full flexibility across the whole range of Empyrean 1D applications. In a diffraction measurement, fluorescence from some elements can give rise to unwanted background intensity. A high background reduces the possibility of identifying the low intensity peaks that often provide the final clues in analysis of an XRPD-scan. This presentation shows how Malvern Panalytical's 1Der detector improves data quality in XRPD measurements. Also, an introduction to MultiCoreOptics is given. With MultiCoreOptics diffractometer's optical path can be fully reconfigured from the control software, without manual operation. Combined with a sample changer, this means that several different types of measurements can be run in a batch, XRR/GIXRD and Bragg-Brentano parafocusing geometry for example.

0955 **XRDynamic 500 from Anton Paar: A flexible powder X-ray diffractometer for manifold applications**
Timo Müller, Andrew O. F.Jones, Marius Kremer, Benedikt Schrode, Praveen Vir and Barbara Puhr - *Anton Paar GmbH*

With the automated multipurpose powder X-ray diffractometer XRDynamic 500, Anton Paar has brought new technologies and approaches to X-ray diffraction (XRD), taking materials research to the next level. For example, the TruBeam™ concept, comprising a large goniometer radius, evacuated optics units, automatic change of the beam geometry and all optics, and automated instrument and sample alignment routines, results in outstanding data quality (in terms of both resolution and signal-to-noise ratio) as well as highest flexibility and easy use. In this presentation various applications of XRDynamic 500 will be discussed. XRDynamic 500 is suitable for powder XRD, grazing incidence XRD, non-ambient XRD, PDF analysis, and SAXS measurements covering a wide range of sample types and application fields. A wide variety of sample stages and sample holders ensures that there is an optimized instrument configuration available no matter the type of sample and the high degree of automatization allows the easy change between different setups. Application examples range from pharmaceuticals and food products to minerals and metals and also include non-ambient XRD measurements during cooling or heating of the sample. Besides XRD applications, also SAXS experiments on nanomaterials will be discussed, for which XRDynamic 500 and its patented EVAC module sample stage provide the best results obtainable on a lab diffractometer. It is shown that XRDynamic 500 is a versatile instrument, giving highest data quality for many different applications.

1015 Coffee and Exhibition

Session: XRD and XRF **Chair: Hege**

1100 **Strategies for Better Matrix in XRF Elemental Analysis with Omnian General Application**
Lorentz Petter Lossius and Karin Bolstad – *Hydro Aluminium*

This is a two-part presentation. Karin Bolstad will share experiences from work to improve precision in XRF analysis for pitch, a difficult material, showing how wide an investigation can be when chasing down systematic errors – and how good the results can be, eventually.
Lorentz Petter Lossius will give two examples of characterization by XRF and XRD, with matrix modelling supported by XRD and other analysis. Omnian is the general purpose XRF elemental analysis program used. Strategies include straight scan, adding Knowns for the light elements C, N, O, F, S and selecting a component as Balance. Hydro Aluminium industrial materials are mostly of the light element matrix type high in fluorides and oxides of Na, Al, Si, Ca.

1120 **Chemical and Structural Analysis of DRI using XRD and XRF**
H. Fahlquist-Hagert, R. Yerly, D. Bonvin - *Thermo Fisher Scientific*

Direct Reduced Iron (DRI) is getting more popular to get iron from iron ores. Also called sponge iron, it is produced from the direct reduction of iron ore to iron by a reducing gas (gas-based process) or carbon from coal (coal-based process). This process uses much lower temperature compared to the traditional blast furnace; hence it is energy efficient and helps decreases the carbon footprint of iron & steel making process. In this presentation we will show how X-ray diffraction and X-ray fluorescence can help controlling the DRI process either through a benchtop diffractometer or using a unique XRF – XRD combined instrument.

1140 **The perfect X-ray couple: XRD and XRF applications at the Geological Survey of Norway**
Jasmin Schönenberger - *Geological Survey of Norway*

The Geological Survey of Norway (NGU) covers a broad spectrum in geoscience ranging from traditional mapping to the exploration of new mineral resources, from geohazards, geophysics to marine and social geology. NGU has extensive laboratory facilities and hosts parts of the MiMaC infrastructure (Norwegian Laboratory for Mineral and Materials Characterization).
Both X-ray fluorescence (XRF) and X-ray diffraction (XRD) are highly demanded analytical techniques and extensively applied for most of NGU's projects, such as:

a) The NFR-funded Bedre Helse project was in collaboration with Statens Vegvesen, Miljødirektoratet, NTNU and others. Its main goal was to study the health effect due to traffic pollution and spike tire abrasion. At NGU, commonly used road stones were characterized for their grainsize-dependent mineralogical composition. Clinical studies also revealed the health impact of differently shaped mineral grains/crystals.

b) The constantly ongoing restoration of Nidaros Cathedral requires sufficient supply of adequate building stone. Its quality demands are high as the stone should regionally be sourced, resistant to weathering and it should resemble the original material. XRD has helped to characterize the stones from different quarries.

c) NGU collaborates with Nye Veier for road and tunnel construction projects. XRD identifies sulfidic phases in country rocks that can dissolve during weathering and form acidic fluids which may have an impact on tunnel stability. Moreover, the study of clay minerals provides estimates on swelling properties.

d) The petroleum industry-funded BASE project sheds light on fracturing and weathering processes on- and offshore Norway by integrating a wide range of geoscientific disciplines. XRD plays a central role and in combination with K-Ar geochronology, time constraints for faulting and landscape evolution have been made possible.

e) The FEN complex in southern Norway has attracted interest due to its potential as a new resource for Rare Earth Elements (REE). XRF and XRD analyses along with hyperspectral imaging on drill cores can serve as a tool to prospect for the most highly mineralized zones.

These few examples show NGU's excellent expertise and its huge diversity of geoscientific projects that all serve one purpose: Geology for Society.

1230 Lunch

Session: Online-XRF **Chair: Torkild**

1330 **On-line slurry XRF - a 50-year-old tool in base metal mining**
Lars Eric Carlsson - *Boliden Mineral AB* - Invited speaker

The on-line slurry XRF-analyzer (or On-Stream Analyzer, OSA) has been an important tool in the base metal mining industry for over 50 years. In base metal mining (primarily Cu, Zn, Pb, Ni) the ore from the mine is processed in a concentrator plant before being shipped to a smelter. The ore is crushed, grinded in water and then the valuable minerals are separated. Flotation is one important process and others are gravity separation and leaching to a solution. To measure the elemental concentrations in feed, concentrate and tailings in the different stages is crucial for controlling the process.
The on-line XRF technique entered the mining industry in the late 60's and since the 90's most flotation concentrator plants have been equipped with such systems.
The first systems in the 60's were all wavelength dispersive systems, but in the 70's several systems with energy-dispersive technique were built. These type of analyzers are also installed in leaching plants and in smelter operations. In the smelters applications are the control of the electrolysis process and in water purification plant.
Boliden AB is Europe's biggest Zn-producer and produces also Cu, Pb, Ag and Au. It has five mining areas and four smelters in the Nordic countries and a Zn-Pb mine in Ireland.
Boliden developed an ED-analyzer (Boxray) in the 70's and the first prototype was installed in the Aitik Cu-mine 1976. Since then 4 generations and over 30 systems have been installed. From 2012 a separate company, Xore AB, Skellefteå, develops, builds and maintain the systems.
Some typical accuracies for different ore types, comparison of ED vs WD and also stability issues and detection limits will be discussed.

1415 **Process optimization by real time analysis of liquids' composition in Metal & Mining**
Fabio Maggiore from *Hobré Instruments BV*. Elke Thisted, Albert Mouris and Torkild Eivindson

The XRF method, with a long-proven track record, is one of the most common analytical techniques for elemental measurement in the metal & mining industry. The technology offers high safety, low OPEX, high versatility and availability, covering the whole range of concentrations, starting from ppb levels (in the range of 0,00001%) up to 100%. The technology can be applied to most of the periodic table, can be used on both solids and liquids, is non-destructive for samples, does not consume chemicals and does not require high skills. The "speed of response" gives feedback within seconds of what occurs in a hydrometallurgical process, indicating reactions occurring or not, rate increase/decrease, reagents depletion or formation of unwanted species leading to emergency situations. Process Engineers often require knowing what is going on inside a reactor at all moments, in order to intervene early. Today, this is possible thanks to a multi-element XRF analyzer. A metallic matte must be leached to dissolve some elements and a thorough control composition is key to optimize the following refining stages, since ores are always variable. Here "speed of response" and "high accuracy" of an online analysis is essential for the Metal & Mining processing industry. In this presentation, the authors introduce their own "case study", made by Glencore Nikkelverk, that has installed the "C-Quand online XRF analyzer" fabricated by Hobré Instruments, a world leading manufacturer of analyzers. The online XRF analyzer is employed for the control of the critical leaching phase of the Ni-Co-Cu

matte, substituting a much more complicated and costly analytical technique. The results, challenges and the improvements of the first two years of utilization of the online XRF analyzer will be shared, showing that Process Control cannot live without proper measurement, thus measurement on representative samples with an online XRF analyzer will truly become the heart of hydrometallurgical processes in the future.

1435 **Application of Online XRF at Glencore Nikkelverk – from a user perspective**
Elke Thisted - *Glencore Nikkelverk*

Glencore Nikkelverk purchased an Online EDXRF analyzer in the fourth quarter of 2018 for measuring elements in three different process solutions. The presentation will show the timeline of the last 4 years including:

- the installation period,
- the test period where the instrument ran parallel with the traditional analyses (photometry)
- the ups and downs, challenges, and solutions at site.
- the instrument in operation for process control.

And finally, the question will be answered “Are we there yet?”.

1455 **Online ED-XRF as a tool for the green transition in industry**
Hege Indresand - *NORCE Norwegian Research Centre As*

Online ED-XRF analysis of metals (Ni, As, V, Cd, Pb, Mn, V) in airborne dust around metal production sites in Norway have been tested the last few years to evaluate air quality. This presentation will show how such measurements of manganese around ferroalloy production plants have narrowed zero-emission strategies. Several measurement campaigns have shown that elevated Mn in hourly measurements occurs episodically related to process fluctuations and weather influences. Looking deeper into the elevated Mn episodes have the potential to unlock important sources and validate further abatement work.

1515 Coffee and Exhibition

Session: XRF **Chair: Elke**

1600 **Fusion of sufidic ores**
Rainer Schramm – *Fluxana GmbH & Co.KG*

Presentation of a concept study to show all necessary steps to get precise results.

1620 **Determination of chlorine in silica fumes - ensuring traceability by developing an independent XRF method**
Jacek Anyszkiewicz - *Łukasiewicz Research Network - Institute of Non-Ferrous Metals* - Tadeusz Gorewoda, Justyna Kostrzewa,Ewa Jamroz, Izabela Maj, Alf Guldhav

The development of new certified reference materials (CRM) in accordance with the requirements of ISO 17034 standard requires the measurement traceability of the certified values. It is not a problem for the wet methods, like ICP-OES or AAS, in which certified reference standard solutions are commonly used and easily available. The problem is when we want to use instrumental methods that require calibration with standards to characterize a parameter (in this case the concentration of an element). The laboratory performing such analyses should follow the requirements of ISO 17025 standard and the CRMs producer should have evidence to ensure the traceability of the certified value. This paper presents the development of a method for chlorine determination in a candidate for CRMs – silica fumes (microsilica). For this, X-ray fluorescence spectrometry was used. The samples were prepared as fused beads. The spectrometer was calibrated on synthetic standard samples which ensured measurement traceability. The results were compared with the other independent methods: the potentiometric argentometric titration and XRF standard addition.

1640 **Metal sulphide analysis made easier with complete methods using synthetic standards**
Armand Jonkers - *Malvern-Panalytical*

The use of fused beads has been proven to be very successful as a sample preparation method. The accuracy obtained for major elements can often be a factor 5-10 better than can be obtained on pressed powders. Meanwhile this has become common knowledge. Calibrations are usually based on certified reference materials, from the minerals or ores to be analysed. These have proven to be valuable for many years. As materials are nowadays sourced from different regions, the compositions of these ores may vary significantly. Regretfully, often standard reference materials are not available at all for all materials to be analysed, as they

cover only part of the elements to be analysed or do not cover the requested concentration ranges. Additionally, once a potential supplier has been found, these standards may have run out of stock. For this reason, alternatives to these traditional calibration standards are very welcome. Differences between types of calibrants (Often called calibration standards) that are used today will be explained, one of these sets will be used in a practical example for the analysis of CuS.
Sulphide based materials were traditionally analysed as pressed powders, as they were thought to be impossible to fuse reproducibly due to volatization of Sulphur in the form of SO2. The problems with representativeness of standards for the requested analysis task, as well as the sample preparation makes setting up a method difficult. Pressed powder analysis simply does not give the requested accuracy. For this reason, laboratories have often diverted to methods like AAS or ICP, resulting in higher throughput times as well as largely increased costs. Proof will be given that demonstrates that Sulphide based ores like Copper-Sulphide ores can be prepared and measured with high precision as well as accuracy, provided the measurement and sample preparation parameters are controlled well, combined with the use of a set of calibrants that are free of line-overlaps, set-up according to ISO 17034 by Malvern Panalytical.

1930 CONFERENCE DINNER **Chair: Lorentz Petter Lossius**

Wednesday 7 September

Session: XRF **Chair: Vijitha**

0830 **Ultrasensitive determination of Hg using benchtop EDXRF and TXRF instruments and carbon-based nanomaterials**
Marcin Musielak, Maciej Serda and Rafał Sitko - *University of Silesia*

The determination of mercury at very ultratrace levels, in the presence of complex matrices, faces numerous troubles resulting from insufficient limits of detection (LOD), especially in the case of X-ray fluorescence techniques, i.e., energy-dispersive X-ray fluorescence (EDXRF) and total-reflection X-ray fluorescence (TXRF) spectrometry. At this point, many separation and/or preconcentration methods are often proposed to solve the abovementioned issues. Considerable attention focuses on these based on the solid-phase extraction method, where carbon nanomaterials are implemented. However, carbonaceous materials often pose additional problems with a lack of selectivity toward mercury ions, low adsorption capacities, or long adsorption time. In this work, mercury ions are adsorbed on graphene oxide (GO) or carbon nanotubes (CNT) modified with thiosemicarbazide (TSC). GO-TSC and CNT-TSC allowed us to develop novel analytical procedures for ultrasensitive and selective determination of mercury ions using dispersive micro-solid phase extraction combined with EDXRF and TXRF techniques. After the adsorption process, GO-TSC or CNT-TSC nanoparticles are deposited onto a thin membrane filter and measured directly by EDXRF. In the case of TXRF, the nanomaterial with adsorbed mercury is dispersed in water and pipetted onto a quartz reflector. The developed methods were fully optimized and validated using certified reference materials (CRMs), i.e., waters, tissues, plant leaves, and food, also spiked samples of beverages. Described methods allowed lower the LODs of EDXRF down to 60 pg/mL for liquids and 73 ng/g for solid samples. LODs of TXRF were even better, i.e., 2.1 pg/mL and 1.8 ng/g for liquid and solid samples, respectively.

0850 **New fast and fantastic technique for analyzing ores minerals oxides slags etc by hot pressing with Lucite**
Sven Erik Bäckman - *Degerfors Laboratorium AB*

Analyzing Ores and other oxidic materials are usually done by pressing and making glass beads. Pressing is fast method but you can lose in precision. Making glass beads gives you a very good precision but time consuming. This presentation describes a new technique that is both fast and gives a good precision

0910 **Is there more than classical bulk analysis in XRF?**
Armand Jonkers - *Malvern-Panalytical*

XRF is known predominantly as a technique for the analysis of solid bulk samples such as ores, metals, raw materials, as well as intermediate and finished products. Also, in the analysis of alternative fuels and oils the technique is abundantly used. Though possible, only a few customers analyse gaseous samples. This lecture will deal about high precision liquid analysis, multilayer analysis and an innovative method for the analysis of double layers.
A new sample preparation and presentation technique for liquid analysis, allowing precisions far beyond what any ICP or AAS can deliver will be presented. In the example it is used for the analysis of thin coatings on

metal wire. The method eliminated homogeneity effects and (can be analysed using the Small Spot Mapping option), to give an extremely high precision for the overall coating thickness. Of course, the technique can be used to cover other samples as well. In addition, the method allows for spillage protection due to ripping foils. The possibilities and limitations of hi-tech analysis of multilayers will be discussed, based on measurements performed for the semiconductor industry, that bases its quality control to produce daily products like memory sticks, chips, LED's, hard disks etcetera on the measurements of our equipment. Here sub-atomic layer thickness precision can be achieved. But layer thicknesses of micro-meters can be measured just as well and is common practice in the steel industry (e.g., galvanised steel).
Based on the concepts of multilayer analysis, a new method (patent pending) was developed to analyse two-sided coatings on a thin substrate, an analysis which was not possible using conventional XRF analysis techniques. These coatings are common practice in the wrapping industry and protect our chips (the other type) against moisture and oxygen to keep them crispy.

0930 Coffee

Session: Sample preparation **Chair: Alf**

1000 **Best Operating Practices for XRF users including the importance of sample preparation**
Carmen Kaiser Brügmann - *Rigaku Europe SE*

Quality Control in industry is imperative, this presentation will highlight the XRF best operating practices that will ensure the daily monitoring and compliance of quality control data. The presentation will focus on how to ensure the XRF method is fit for purpose. Data will be shared based on the pressed powder method for the Primus IV. The fundamentals to stable, reproducible analytical results from your XRF spectrometer is not only a stable, working spectrometer, but also a stable, reproducible sample preparation that is appropriate for the material being measured. Choosing an appropriate sample preparation method depends both on the material itself, and on the limitations of the analytical technique with respect to the elements you wish to analyse. We will focus and present the background to sampling and sample preparation. Topics covered will be how to check that your samples have been prepared appropriately and what effects bad sample preparation can have on your measurement result. Daily check analysis is required in order to ensure that the quality from sample preparation through to the XRF instrument is monitored continuously and the data is trended. For an analytical result to be fit for its intended purpose it must be sufficiently reliable that any decision based on it can be taken with confidence. Thus, the method performance must be validated and the uncertainty on the result, at a given level of confidence, estimated. Uncertainty should be evaluated and quoted in a way that is widely recognised.

1020 **Sample preparation for X-ray analysis – new insights**
Martin Lischka - *Herzog*

This talk will focus on sample preparation for X-ray analysis and its challenges to establish a solid and repeatable method. Error sources in the preparation of pressed pellets or in borate fusion can be identified by replication experiments and backed up by key performances indicates (KPI) of the equipment used. Those data can be used not only to monitor equipment performance. Furthermore, they can provide additional information to the laboratory stuff supporting data interpretation and validation. Two case studies will be presented to show recent trends in sample preparation and method development.

1040 **Source of errors in sample preparation for XRF analysis**
Frederic Davidts – *XRF Scientific*

1100 **Real time monitoring of chemical components,**
Mikael Ramström and Krag Petterson - *Acoem AB*

Today most of the real time monitoring of particles is performed by measuring the PM mass. In many cases this does not tell us anything about the origin of the particles. The high time resolution multi-metals monitor of ambient air is a good tool to use parallel to a local PM monitor to establish very important information of the particle content.

1120 CONFERENCE CLOSING

1200 LUNCH