NORWEGIAN CHEMICAL SOCIETY
DIVISION ANALYTICAL SCIENCES

PROGRAM including abstracts
FOR
THE 20th NORWEGIAN X-RAY CONFERENCE

3 – 5 SEPTEMBER 2018
Strand Hotel Fevik, Grimstad

Monday 3 September

1100 REGISTRATION outside the conference hall “Himmel og hav” at Fevik Strand Hotel
Distribution of program

1100 Exhibition Opens

1200 LUNCH

1300 **Opening: Torkild Eivindson, Leader of the Norwegian X-ray Conference Committee**

Session: XRF **Chair: Elke Thisted**

1310 "ED-XRF IN EMISSIONS AND AIR QUALITY MONITORING - 1962 TO 2018 AND BEYOND"
Dr. John Cooper, Sunset CES Inc. (SCI) (Invited speaker)

Over the last 50+ years, great strides have been made in energy dispersive X-ray fluorescence (ED-XRF) technology as applied to air quality monitoring of ambient aerosols and stack emissions. This period covers detector sensing technologies from vacuum tubes, scintillators, and proportional counters to liquid nitrogen-cooled, lithium-drifted semi-conductor detectors to room temperature silicon drift detectors; high-volume fiber filter sampling to continuous, size-selective PTFE filter tape sampling with near real-time metals analysis; detection limits from sub-µg/m^3 to sub-pg/m^3; and evolution of spectral deconvolution to on-line data interpretation. The first part of the presentation will provide a first-hand perspective of the historical evolution of ED-XRF technology as applied to air quality management, after a brief overview of ED-XRF system components for aerosol measurements. The middle part of the presentation will focus on contemporary technology and its application to both source (stack, process, fence-line emissions and fingerprints) and ambient (urban and remote) aerosol monitoring. The last segment of this presentation will provide a view of the future from a historical perspective with a “can-do” attitude.

1355 “Ambient PM2.5 - source profiles from around the globe”
Professor Johan Boman, University of Gothenburg

For decades the first author has been doing sampling and XRF analysis of particulate matter (PM) with co-workers and students from many places around the world. The sampling conditions season locations and duration of the sampling have shifted but the elemental content of the collected PM samples have all been determined by different XRF based techniques. The elemental composition in the collected PM have been further analyzed for similarities in variations to estimate possible source profiles for the PM pollution. This poster will highlight similarities and differences from the various locations. The co-authors and many others have all contributed to the different results summarized here. Without them this work would not been possible to do. Thank you!

1415 “Using an online ED-XRF instrument in understanding industrial dust emissions”
Hege Indresand, Teknova AS

An on-line ED-XRF system (Xact 625i) was used to speciate metals in dust emissions (PM10) in a 3-month study at three manganese plants in Norway (Eramet Norway AS). Sampling locations were set up ca 100-500 m from sources outside the plants in the predominant wind direction. The XRF analysis was able to capture temporal variations in composition that provide very useful inputs for industries’ current goals in dust monitoring reporting and control. The results are focused around the instrument performance, source apportionment and correlations with meteorology.

1440 Coffee, poster session and Exhibition

1530 “Benchtop ED-XRF moving forward by a factor six or more”
Armand Jonkers, Malvern Panalytical

With the introduction of the new Epsilon4 by Malvern-PANalytical, the successor of the Epsilon3 series of ED-XRF spectrometers, yet another leap forward is made with respect to performance in ED-XRF. An increase in sensitivity that has never been shown before was obtained by implementing new detector technology, based on ultra-fast electronics, thus allowing the collection of 1.5 Mcps @ 50% dead time. Moreover, the spectrometer shows a high energy-resolution that is typically 135 eV @ Mn Kα and unlike most spectrometers, stays very good up to very high count rates. Together with the new deconvolution algorithms that even takes care of possible diffraction peaks, results in an unmatched performance. Practically the measurement times may be reduced by up to a factor ten, depending on the application. With the large detector area it is now possible to obtain relatively high sensitivity for elements starting from Na, that was previously only possible using a detector with an ultra-thin window. For elements C, N, O and F, still the SDDultra is available, which now with the faster electronics can also be driven to high count rates. Since the options of finger printing, thin layer analysis, standardless analysis and automation are available, while a sample changer and large sample analysis are already standard, the spectrometer can be put to use in nearly every laboratory.

1605 “Internal Standards In XRF Analysis – Precision With Simplicity”
Stanislav Ulitzka, Twotheta pty ltd

Internal standards have been used for many years in ICP analysis but their use in XRF fluorescence analysis has only been developed and applied over the last few years. Keith Norrish and his team at CSIRO originally pioneered the internal standard method using a flux with Terbium for the analysis of iron ores. The Tb ratio is used as matrix correction for Fe but its biggest advantage is the automatic and simple correction for errors due to bead curvature and other sample preparation defects. Since the method is capable of obtaining very accurate and precise results for iron LOI can be calculated as a difference and doesn’t have to be measured by high temperature TGA. This can significantly reduce the time and cost of analysis. Following this principle fluxes with internal standards have been developed for the analysis of a number of other elements. For example, Thulium (at only 1% concentration in the flux) has been tested and successfully implemented at various Nickel mining and processing operations in Australia and overseas. With a comparable precision fast turnaround and much lower costs the XRF analysis using a flux with Thulium is challenging established and widely used methods (for example the DMG method). Another element of considerable commercial interest (copper) can be analysed and corrected for bead curvature and other sample preparation defects using a flux containing Tantalum. The method has been implemented in a WA Copper mine and is used for the analysis of a wide range of copper products ranging from feeds and tails to concentrates. A comparison with an external reference method has shown excellent agreement and confirmed the suitability of the IS method for routine analysis The authors have demonstrated that using commercially available lithium borate fluxes with internal standards (Ta for copper Tm for nickel and Tb for Iron) can offer significant analytical and financial advantages.

1630 END

1645 Departure for social trip to the local brewery “Nøgne Ø”

2000 DINNER (Fevik Strand Hotel)

Tuesday 4 September

Session: XRD **Chair: Lorentz Petter Lossius**

0830 "In-situ/operando studies of battery materials and catalysts using home-lab and synchrotron powder X-ray diffraction techniques"
Professor Helmer Fjellvåg, University of Oslo (Invited speaker)

The talk will provide examples on how powder diffraction methods are used for investigating various types of functional materials at process/operating/non-ambient conditions. These examples include electrodes for Li-ion batteries; bimetallic nanoparticles for catalysis; microporous materials for absorption and catalysis; metallic nets for catchment of volatile components; phase transformations and oxidation of reduced oxides, decomposition of clays, etc. – with basis in data collected in our home lab, RECX at UiO, and at the European Synchrotron Radiation Facility at ESRF. Some examples will show how X-ray absorption spectroscopy (XANES) and tomography data provides additional insight to electronic changes and amorphization/recrystallization of materials.

0920 "Secondary Alumina – HAL_SecAl, a Calibration Set for XRF and a Phase Set for XRD"
Lorentz Petter Lossius and Karin Bolstad, Hydro Aluminium PMT, **David E. Simon**, DESConsulting

Hydro Aluminium and primary metal producers generally have to handle several major internal process streams of materials that are significantly changed from the original purchased raw materials. These streams include the recycled anode material; the anode cover material (ACM); spent potlining (SPL); and the largest internal material stream, the secondary alumina from the process gas cleaning. Originating as Smelter Grade Alumina (SGA) or primary alumina, SGA is termed secondary alumina after use as the process gas and particulate cleaning agent in the gas treatment plant (GTP). Good analysis of these materials is part of good operational practices and the paper describes experiences from elemental analysis and phase analysis of secondary alumina including work to develop a Certificate of Analysis HAL_SecAl-XRF. This is a 18-part set with 30 different elements based on analysis results from ICP XRF Sintalyzer and combustion elemental analysis. Combining these analyses strengthened the quality of the analysis. However, the set can only be counted as an in-house Reference Material and is far from Certified Reference Material stage. Phase analysis by XRD is described including a study of the transition alumina phases used in the modelling. The aim was to determine the trace-level adsorbed fluoride phases collected by the alumina in the process gas cleaning process.

0945 "Alternative method of quantitative phase analysis available for new Rigaku SmartLab diffractometers"
Maciej Grzywa, Rigaku Rese

Recently Rigaku Corporation introduces new SmartLab and SmartLab SE diffractometers providing expanded functionality thanks to additional hardware components and new SmartLab Studio II controlling and analyzing software. SmartLab Studio II is an integrated software platform covering the full spectrum of operations such as alignment measurement analysis and report creation. Among other features a new method for quantitative phase analysis using X-ray powder diffraction data (observed integrated intensities) and chemical compositions of individual phases is realized in this software. The method requires no additional measurement for standard samples etc. The present method also does not require crystallographic databases which store crystal structure parameters and/or reference intensity ratios.

1015 Coffee and Exhibition

1040 “Closing the gap between benchtop- and traditional XRD”
Leif Viskari, Blue Scientific Ltd / Bruker AXS GmbH

Over the recent years benchtop versions of traditionally larger analytical devices have come to receive much attention. As for many such devices benchtop XRD can be argued to have been perceived as a compromise between compactness and data quality. This presentation will show that contemporary benchtop XRDs such as the Bruker D2 PHASER indeed are closing the gap to traditional systems. The specific advances and their roles will be discussed in detail and comparisons will be shown between benchtop- and traditional XRD systems.

1100 “Quantitative Analysis of Electrode Materials by X-Ray Micro Computed Tomography(µCT)”
Stein Rørvik, SINTEF Industry

Micro X-ray Computed Tomography (CT) is an instrumental method for recording inner structure 3D images without damaging the observed volume. A methodology has been developed by SINTEF for CT of carbonaceous materials, yielding detailed structural views and quantification of the pore, void and grain distribution within volumes of 10 mm to 130 mm diameter.
Example results are shown from a study of anode isotropy. A series of cores from a prebaked industrial anode was analyzed. This particular anode was selected due to visible surface cracking. The CT data was calibrated and connected to the physical position in the anode. The data was analyzed with various methods that quantify the degree of anisotropy. The analysis show that the anodes have a 10-15 cm wide zone of high anisotropy, while the top and bottom of the anode are more isotropic. Some possible reasons for the anisotropy are discussed.

1130 “Technologies enabling current state-of-the-art in benchtop WD-XRD”
Leif Viskari, Blue Scientific Ltd / Bruker AXS GmbH

Recent developments in benchtop WD-XRF have allowed the analytical performance to be pushed towards better precision and better detection limits whilst maintaining the compact physical format essential for a table-top device. This presentation will shine light on the technical advances incorporated in the Bruker S6 JAGUAR that enable the current state-of-the-art in benchtop WD-XRF and how these advances open up new application spaces for such devices.

1150 “XRF and XRD Combined Analysis of Secondary Alumina and Recycled Carbon Anode Material after Wet Magnetic Separation”
Lorentz Petter Lossius and Karin Bolstad, Hydro Aluminium PMT

Secondary alumina is a major internal process stream at aluminium metal plants. In a preceding presentation, a Rietveld based method for modelling secondary alumina was presented. This presentation is an example of use in a wet magnetic separation study run using secondary alumina fines. The magnetic fraction from wet magnetic separation is higher in impurities, and the study was a test to see if the selected secondary alumina XRD phase set is sufficiently accurate to allow modelling and quantification of minor non-alumina phases.

1205 Lunch

Tuesday 4 September

Session: HH/ED-XRF **Chair: Hege Indresand**

1300 "PORTABLE XRF – THE SILVER BULLET OR FOOLS GOLD?"
Maggie Loubser, GeoMag GeoChem (Invited speaker)

X-ray Fluorescence spectroscopy is a mature technique with the theory well described and routinely applied in process control, exploration, mining and manufacturing apart from research and development applications. The “big shrink” as in most analytical technologies was the result of improved electronics and computer chips, and together with the advance of the SDD detector Energy Dispersive XRF was ready to enter the playground. Portable XRF moved from a metal sorting scanner to an actual quantitative analytical tool. But here lies the biggest challenge, because of reduced cost and ease of operation this tool is now more accessible to people who often do not have the insight in the limitations.
One of the major advantages of portable XRF, is precisely the fact that the instrument can be taken to the material to be analysed, eliminating sampling, sample preparation and transporting samples to a laboratory. All of which takes time, can add risk (as in art and conservation), and destroys the sample. But herein lies the greatest risk too – because users forget that the largest contribution to uncertainty in XRF analysis is representative sampling and sample preparation. If you are going to act like a cowboy, your results will be fit for the Wild West.
In this paper the differences between analysis in the field vs. the laboratory will be explained, and methods to improve accuracy or interpret the data based on the known inaccuracies will be presented. Geological studies will show how to get fit for purpose data in various scenarios. Case studies will be shown in the field of art and conservation, where the ability to bring a non-destructive technique to the actual object probably had the biggest impact, again, not without pitfalls. Lastly, the possibilities of using the technique in process control will be investigated.

1345 “Application of Portable XRF for Investigation of Archaeological and Cultural Heritage Materials – Some Case Studies”
Kidane Fanta Gebremariam, Universitetet i Stavanger (UiS)

Handheld portable X-ray Flourescence (pXRF) instruments are increasingly utilised for the investigation of archaeological and cultural heritage objects. This mainly originates from the possibility it offers to conduct mutlielemental analysis in a non-destructive and non-invasive manner as maintaining the integrity of the precious and unique cultural heritage objects is given the utmost importance. The advancements in the X-ray source optics detection system and software development have made the X-ray fluorescence instrument more powerful portable affordable and thus accessible to wider users. Archaeometry and heritage science have benefited from these developments and will do so in the future facilitating the investigations to gain better insight into the human past culture materials and technologies. It also supports the well-informed conservation of the unique objects. Here will be presented some case studies from the utlization of portable XRF for the investigation of archaeological historical artistic and cultural heritage materials of diverse characteristics. The studied materials include among others mortars plasters crucibles lithic objects gildings paintings polychrome sculptures illuminated manuscripts and slag materials. The applications range from in situ examinations for quick on-site identifications in remote sites to the use in museum and laboratory settings as part of investigations involving complementary multianalytical techniques. The complementarity of the portable XRF approach with other ED-XRF analyses X-ray diffraction microscopic and spectroscopic techniques will be covered highlighting the advanatages the challenges and how the drawbacks were addressed. The application area demands highly multidisciplinary approach from the formulation of the analytical problem to be tackled to the interpretation of the experimental results. It is like working at the interface of humanities art technology and sciences. While presenting the case studies a glimpse of working in this field that involves collaborations with archeologists conservators building experts curators historians and artists will be given.

1410 “Challenges in using portable XRF for pigment analysis on Norwegian church art”
Susanne Kaun, NIKU

One of the specializations of the conservation department at the Norwegian Institute for Cultural Heritage (NIKU) in Oslo is the examination and treatment of church art. The knowledge of the used pigments and metal leaf can give valuable information on painting techniques and original colors. The presentation addresses the benefits and challenges in using portable XRF for pigment analysis on easel paintings and painted wooden objects from Norwegian churches. The instrument used is a Niton Xl3t GOLD+. The identification of pigments is done by combining the element composition with the information on the color of the paint as well as the knowledge on historical painting materials. The major advantages of using portable XRF on valuable cultural heritage objects is that it is non-invasive which means no sample taking is needed. Pigments with characteristic elements such as cinnabar or orpiment are easy to identify even if they changed the color. Organic pigments like indigo do not contain detectable distinctive elements and can therefore not be identified by XRF. Blue or green copper pigments cannot be separated into different types. On the other hand the all-over element analysis can give useful information on fillers such as barytes and chalk. Potable XRF turned out to be especially suitable for the identification of gilding material. Beside precious materials such as gold and silver also imitation gold could be identified. The major challenge in using portable XRF on painted objects is that the material is not homogenous and it is problematic to give information on the composition of layers. The omnipresence of lead pigments in that type of objects made the interpretation of the results difficult as lead peaks are interfering with peaks from several other elements. In addition the survey of three-dimensional objects like sculptures was limited due to the geometry of the instrument. With some limits portable XRF is a very helpful and easy-to-use instrument for investigating church art. Its non-invasive character makes it a valuable instrument for conservators.

1435 Coffee and Exhibition

1530 “HandHeld versus Portable and Benchtop ED-XRF - what’s the real difference?”
Bjoern Klaue, Spectro Analytical Instruments

Over the past 15 years HandHeld XRF has become a very important tool for field analysis in many environmental geological and mining applications. Several attributes make HH-XRF a powerful tool for onsite analysis: portability battery operation robustness pre-calibrated applications and simple data handling. Some advertised HH-XRF performance levels have also generated criticism with regards to sample presentation data evaluation and traceability. This discussion will present a different understanding of appropriate results acceptable errors or even if a true quantitative analysis is possible. Accuracy and precision requirements for field analysis or screening applications are typically very different from “laboratory quality” methodologies. Deviations and errors from correct values are most prominent for difficult or highly variable matrices trace components and light elements. Aside from typical user errors and poor sample preparation this paper will discuss the true technical differences between field and laboratory devices. The actual hardware components of modern HH-XRF devices in the form of mini X-ray tubes primary filter changers and detectors are not the key limiting factors. Driven by limited computational power and data analysis time the performance differences are hampered mostly by the evaluation algorithms applied in handheld devices. The key differentiator is the use of the proper algorithms for spectra deconvolution to obtain peak analyte signal net intensities. These net intensities are then fed into more advanced calibration models for optimal quantification. We will show some prominent examples that demonstrate very clearly why the data evaluation approach currently used on-board for most conventional handheld devices is limited. Compared to portable or laboratory ED-XRF spectrometers there are of course influencing hardware factors like tube power filter regimes additional excitation options temperature stability electronic noise etc. that HH-XRF currently cannot overcome. Examples will be presented to show the differences.

1550 “Production of monitor control and calibration samples for XRF”
Rainer Schramm, FLUXANA GmbH

With the production of several thousand monitor control and calibration samples per year FLUXANA is one of the number one producers of such samples world wide. The presentation shows the variety of production possibilities to get such style of samples. Drift correction and monitor samples are used to keep the long term stability of all xrf calibration packages. Mainly they are based on silicate or borate glasses containing elements in high enough concentrations to get a high intensity. However also sinter or natural materials could be used from time to time. Certified reference materials are produced in powder form mostly combined with a proficiency test to include as much laboratories as possible. Finally a certificate in agreement with ISO 17034 is produced. Customized calibration samples could be produced as fused bead or as a glass powder containing flux and sample at a time. Both together will speed up the calibration time a lot and helps to save development costs and time. Finally a new procedure to produce control samples is presented. Nowadays the production of a sample with the help of a 3D printer could be performed. The fact that a 3D printer builds up an object layer by layer could be also used to design samples with known element content if different materials are used for printing.

1615 “Can Energy Dispersive XRF match the analytical performance of Wavelength Dispersive XRF? Case Studies”
Raphael Yerli, ThermoFisher

In recent years energy-dispersive X-ray spectrometers (EDXRF) has displayed considerable performance improvement due to technological advances in detector technology and electronics. Count rate capabilities have increased to several hundred thousands counts per second and in some cases attain one million counts per second. This begs the question whether EDXRF approaches or matches the performance of wavelength-dispersive systems (WDXRF). We compare both techniques for two applications; analysis of glass and airborne particulate matter collected on filters. While the outcome would have been clear a decade ago we show it’s less straightforward today. At similar power levels glass analysis by EDXRF has come a long way but spectral interferences as well as light element sensitivity remain an issue. Analysis of filter media is a typical EDXRF application but the replacement of the Si(Li) detector by the silicon drift detector (SDD) affected the sensitivity of heavy element detection using K lines. Meanwhile there’s an interest to perform this application with WDXRF as well. We’ll show what’s possible with WDXRF at moderate power and compare this with results obtained with an SDD based EDXRF system.

1640 END

1930 CONFERENCE DINNER Chair: Lorentz Petter Lossius

Wednesday 5 September

Session: XRF **Chair: Alf Yngve Guldhav**

0900 "Sample Preparation for XRF analysis"
Mark Tutty, SPEX Europe

Looking at the importance of sample preparation in getting high quality analytical results by XRF. Will consider the practical and economic advantages / disadvantages of preparing samples as pressed pellets or as fused beads.

0925 "Analysis of surface coating on anodes in a Ni-electrowinning plant by HH-XRF"
Torkild Eivindson, Torjus Åkre, Glencore Nikkelverk

A new type of box anodes is replacing old anode technology in the Ni-electrowinning industry. This new box anodes are made of a Ti-grid, covered with a layer of precious metals. The precious metals make the box anodes more energy effective compared with the older anode technology. It is of great economical interest to run this anode as long as possible. HH-XRF is used to measure the layer thickness, and monitor the lifetime of the anodes. This presentation presents the user made XRF-calibration, and some challenge with this measurement.

0950 Coffee, checkout of hotel

1030 "Small spot XRF analysis becoming a routine tool in the laboratory"
Armand Jonker, Malvern Panalytical

Small-spot analysis by XRF has become increasingly important due to increased quality demands of many products. This includes the analysis of inclusions (e.g. in glass, steel) and homogeneity (e.g. cement mortar, ceramics, CRM material), but also the analysis of small samples (e.g. chips of precious material, toys, art, ROHS regulation for in Electronics and Forensics). Malvern-PANalytical shows a range of solutions that offer answers to questions where non-conventional samples need to be analyzed, including the on-line analysis of liquids using the X-Flow and Epsilon1-for flow analysis. Another solution offered involves the Epsilon1 for small spot analysis. The Zetium that was introduced before with small spot analysis, now available with Hiper optics, shows boosted performance with a sensitivity improvement of a factor 10, thus reducing the measurement significantly and making the analysis available for routine investigations, even at low concentration levels. The lecture will show a number of analytical examples for above mentioned solutions.

1055 “Latest results using small spot and mapping by WDXRF”
Raphael Yerli, ThermoFisher

The latest types of high-power wavelength dispersive X-ray fluorescence instruments (1500W to 4200W) benefit from enhanced performance thanks to new X-ray tubes using thinner beryllium (Be) window. Furthermore, with close coupling between the X-ray tube and the sample the performance of WDXRF instruments has improved across the periodic table and at all power levels. Obviously, this enhanced performance is beneficial when analyzing small samples or small spots on the surface of the sample. Cartography of a sample surface is now also possible, and it opens up the WDXRF technique to analysis of non- homogeneous sample. A whole new world of applications is now possible despite an intensity decrease of factor 10 thousands when measuring a 0.5 mm spot compared to the usual 29 mm diameter of analysis. In this presentation several applications will be presented for which these new capabilities of WDXRF instruments and the use of standard-less analysis packages such as UniQuant are very beneficial.

1115 “Mapping of element distribution in process industry”
Elke Thisted, Glencore Nikkelverk

The presentation will show the planning analysis and results of mapping a filter press at Nikkelverk with a handheld XRF. The results of this study will be used to improve sampling procedures for more representative sampling of the filter sludge/residue.

1135 CONFERENCE CLOSING

1200 LUNCH