Technical Papers –Monday 13-September-2010 (Afternoon)

1:30 pm Plenary Lecture

A Comparison of Commercially Available Methods for Analyzing Rock Samples for Rare Earth Elements

R.E. Lett and K. Paterson, BC Ministry of Mines and Petroleum Resources

The recent rapid increasing of using rare earth elements in the manufacture of superconductors and hybrid vehicles combined with depletion of existing global supplies has stimulated exploration for new geological rare earth element resources. Clearly, confidence in a precise estimate of a potential rare earth element deposit size will depend on several factors including reliability of rock sample assays. Rare earth element (La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu) geochemistry is also a valuable tool for discriminate between volcanic rock types and for classifying different geotectonic terrains. Again, confidence in reliable geochemical discrimination will depend on the accuracy of the method used to analyse rock samples. X-ray fluoresce (XRF), lithium metaborate fusion or sodium peroxide sinter followed by inductively coupled plasma mass spectrometry (ICPMS), instrumental neutron activation (INAA) and acid digestion-inductively coupled plasma mass spectrometry are among geochemical methods commonly used to determine rare earth elements. Each method has advantages and limitations.

2:00 pm

Mineral Liberation Analysis – Methodology and Industry Applications

Nicola McKay and Randy Blaskovich Teck Applied Research and Technology

The Mineral Liberation Analyzer (MLA) was developed in Australia in the 1990s to provide automated mineralogical data in support for the mining industry. The system consists of a tungsten filament FEI Quanta 600 Scanning Electron Microscope (SEM) fitted with an automated stage, dual Bruker XFlash detectors, Esprit Spectrum software and proprietary JKTech MLA software. MLA test samples are prepared as polished epoxy-resin grain mounts. MLA-based mineral identifications are based on backscatter electron segmentation of mean atomic weight, X-ray analysis of each segmented grey level and matching of the data against a user defined library of mineral compositions. The measured data can be extracted in a variety of ways to obtain metallurgically significant information such as the percent distribution of minerals, mineral grain size distribution and mineral liberation. The 2 MLAs at Teck's ART laboratory in Trail, BC, are used to support our exploration programs, mine start-ups & expansions, environmental planning and to diagnose metallurgical circuits.

2:30 pm

Recent Developments in Automating Sample Preparation

Ian Devereux and Brad Hunting, Rocklabs Ltd.

With all laboratory automation projects, the biggest challenge is to select the most appropriate technology. There is never just "one size fits all".
Factors to be considered include the number and weight of the samples being processed per day, the methods of analysis to be used, the availability of technical staff on site, the cost of laboratory personnel, country of location, ease of access for back-up technicians and the budget available.
Rocklabs latest project was for a commercial laboratory in New Caledonia which is close to New Zealand. The selection of technology included an industrial robot and a new automated batch mill to provide a finely pulverized sample for XRF analysis. The samples are nickel laterites and saprolites from exploration and development projects. These are some of the most difficult samples to process of any in the mining industry.

3:00 pm

ARL Fire Assay Analysis

Eric Tusseau, Thermo Fisher Scientific

Analysis of precious metals traces in fire assay lead buttons, with optical emission spectrometer. Cupellation stage suppression and automation of lead button making. Benefits of the method, results, analytical performance: accuracy, precision and stability. Examples and layouts.

4:00 pm

Urban Ore: The Next Frontier in Resource Recovery

Christa Ford, Teck Metals Ltd.

We will outline how products from Trail Operations are a part of products we use in everyday life, what processes are being developed to recover metals from consumer products and the assaying challenges associated with this type of recycling initiatives.

4:30 pm

Laser Induced Breakdown Spectroscopy: A Unique and Complementary Elemental Analysis Tool for a Wide Range of Industrial and Research Applications

Scott W. McGeorge, Transition Technologies, Inc.

Laser Induced Breakdown Spectroscopy (LIBS) is an ultraviolet or infrared laser-based technique for elemental analysis. The laser serves as both the sampling/ablation source and the excitation source. Therefore the capital cost is much less than laser ablation ICP ES or MS systems.
LIBS provides the advantage of almost full-periodic table application with minimal sample preparation. Ablation spot sizes can be as small as 5 micrometers or as large as 2 mm; bulk sampling or mapping of a larger surface area can be achieved.
Elemental analysis of low atomic weight elements can be very difficult or impossible with X-Ray Fluorescence systems but LIBS can measure these light elements, including Be, B, Li, H, and C, and provide good quantification. This presentation will cover the basic theory and operation of LIBS with a focus on geological matrices. Other applications of interest to large industrial operations, i.e. discrimination of fuel mixtures for homogeneous charge compression-ignition engines, will be referenced.

5:00 pm

Advances in the Preparation of Certified Reference Materials and Proficiency Testing Program – Mineral Analysis Laboratories at CCRMP

Maureen E. Leaver, Diane Desroches and Joseph Salley, Canadian Certified Reference Materials Project, CANMET – Mining and Mineral Sciences Laboratories

The discussion will include recent improvements in the preparation of CCRMP certified reference materials, a comparison of various statistical parameters observed in successive generations of materials, use of ISO Guide 33 for the assessment of laboratory data obtained with reference materials and some observations on recent data from Proficiency Testing Program – Mineral Analysis Laboratories.

Technical Papers –Tuesday 14-September-2010 (Morning)

8:00 am

Eliminating the Matrix Barrier in ICP-MS Analysis.

Jeffery Bown, Thermo Fisher Scientific

The major practical limitations in ICP-MS analyses, namely matrix tolerance, interference removal, detection limits and sample throughput, are all linked to the nature of the sample itself.

Major technological advances in ICP-MS technology in recent years have produced instruments with vastly improved sensitivity and increased linear range. Now the focus has turned to addressing issues arising from the need high-throughput analyses of heavy-matrix samples like brines, sediment and geochemical digests.

The development of a unique matrix tolerant interface in ICP-MS has removed a major barrier to high throughput analysis of heavy matrices including industrial chemicals, tribology samples, soil, sediment and geochemical materials. In addition, 3rd generation Collision Reaction Cell technologies allow for cost effective interference free analysis for the first time, even in the most challenging of matrices.

Concurrent development of new high throughput sample introduction accessories has effectively doubled sample throughput without compromising instrument performance. The latest breakthroughs in sample introduction technology allow the analysts to run samples with dissolved solid levels ten times higher than was previously possible in ICP-MS while also enabling automated on-line dilution where necessary while simultaneously increasing plasma robustness and reducing potential interfering species even further.

The technology behind enhancements to improve sample throughput of complex matrices shall be discussed in detail and illustrated with real world data from current state of the art ICP-MS instrumentation.

8:30 am

ASXPRESS™

Michael Sgroi, Cetac

The CETAC *ASXPRESS*™ Rapid Sample Introduction System, when coupled to a CETAC autosampler, optimizes sample introduction by significantly increasing sample throughput and reducing costs of materials, power, maintenance and labor for ICP-AES and ICP-MS analyses. The system is designed to allow multiple functions to occur simultaneously which would otherwise take place separately.

A standard analysis system relies upon a single peristaltic pump to both deliver samples to the nebulizer and rinse the sample flow path between sample deliveries. The *ASXPRESS*™ system utilizes a high speed vacuum pump in addition to the ICP peristaltic pump. The 6-port valve allows the use of both pumps simultaneously, reducing total sample analysis time significantly.

The same data quality can be achieved as with the traditional analysis setup, only this data is acquired in a fraction of the time. The overall stability is improved and demonstrated by QC samples that pass over a greater period of time and throughout a higher total sample volume.

9:00 am

The Challenge of Mining Analyses: From Rocks to High-Purity Metals with the NexION 300Q

Aaron Hineman PerkinElmer Inc.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is widely used in geochemical and metallurgical applications for elemental analysis. This presentation describes the analysis of ore samples and their final high-purity products using the recently introduced NexION 300 ICP-MS by PerkinElmer. Similar to its predecessor, the ELAN, NexION 300 uses a 40 MHz free-running generator which is particularly suited for demanding applications. In addition, NexION 300 is equipped with a triple-cone interface and an orthogonal ion path which provides exceptional stability while dramatically reducing the maintenance frequency.

10:30 am

Performance Features of the Agilent 7700x for Geochem Analysis

Emmett Soffey, Agilent

Some of the most important features to consider in ICP-MS for geochemical applications are sensitivity, matrix tolerance, linear dynamic range, interference removal and ease of use. The Agilent 7700x, with its 3rd generation cell technology and HMI (high matrix introduction) feature combines all of these qualities into a single compact and powerful instrument. An overview of this technology will be provided.

11:00 am

REE and Trace Element Analysis of Rocks and Minerals with the Agilent 7700x ICp-MS Using a Combined Fusion-Dissolution

Charles Knaack, Washington State University

We have developed a combination fusion-dissolution method that effectively decomposes refractory mineral phases and subsequently removes the bulk of unwanted matrix elements. After digestion samples are analyzed on an Agilent model 7700x ICP-MS. Sample throughput is increased and instrumental exposure to sample matrix is minimized through the use of ISIS-DS discrete sampling protocol and HMI matrix dilution. Long-term precision for the method is typically better than 2% (RSD) for the REEs and 5% for the remaining trace elements. Analyses of USGS and international rock standards show good agreement with consensus values.

11:30 am

Laser Ablation

Matt Nigro, Cetac

The elemental and isotopic composition of minerals can provide valuable information about the processes which occur during the formation of rocks and ore deposits. Laser ablation coupled with an ICP or an ICP-MS can provide for direct analysis of the elemental composition of a rock by discretely sampling selected areas with the produced aerosol being subsequently passed into the ICP or ICP-MS for analysis.
Laser ablation sampling provides advantages over other techniques in that sample preparation is minimized, analysis is fast (the whole periodic table in a matter of minutes), and small areas down to 3 microns or less may be individually targeted. Detection limits down to single parts per billion in the solid are possible for some elements.
CETAC and Photon Machines recently announced their decision to collaborate on the development of laser ablation technology. This collaboration provides the community with a complete range of laser ablation systems; a tool for every job! This paper will discuss the different options which now exist for laser ablation sampling; from solid state lasers to excimers, and from 266nm to 193nm. The presentation will highlight the various figures of merit for the technique and discuss which laser is right for which application.

12:00

NEWS in ELEMENTAL ANALYSIS H to U from % to ppt BRUKER's Elemental Analysis Range

Andrew Toms, Bruker, and Alexander Seyfarth, Bruker AXS Inc.

The 2 part talk will illustrate the new BRUKER ICP-MS product offering by illustrating new application concepts enabled by the high end ICP-MS system enabling the linear analysis trough 9 orders of magnitude. The unique ion lens combined with the rugged MS engine enables a PROCESS GRADE reliability with R&D performance! For solid samples the ICP-MS can be combined with a LIBS system enabling the measurement of small solid samples directly.
In Part 2 we will show other applications that require less sample throughput and no digestions at all but sill are in the low ppb range: the TXRF unit S2PICOFOX is capable of ppb level determination in filters, sewage as well as human blood. For % to ppm level analysis of mining samples and minerals XRF can be used, either as HH, benchtop or floor standing high performance WD XRF. XRF is especially useful for fast unattended operation in remote sites where cost of analysis and reliability are of utmost concern.
Combustion (Gas) analysis for C and S as well as O,N, H closes the gap to enable the analysts cover the periodic table with BRUKER's elemental analysis techniques. We will use dedicated applications ranging from Sewage, mining to blood and metals analysis to illustrate the performance and capabilities of the instrumentation.

Technical Papers –Wednesday 15-September-2010 (Morning)

8:00 am

Fingerprint and Standardless Analysis with benchtop XRF

Allan Ball, Panalytical

Recent developments in XRF software allows for quick sorting of material based on fingerprint techniques and standardless analysis.

8:30 am

An Easier and Faster Way to Perform Sodium Peroxide Fusion

Jean-Ludovic Martel, Xstrata Nickel Raglan Mine

This presentation is an overview of the sodium peroxide fusion method used at Xstrata Nickel Raglan Mine for Exploration, production and mill samples. This presentation may provide good advice for those who performed sodium peroxide fusion the traditional way.

9:00 am

Determination of Antimony and its Species in OECD 203 Matrix

Barry Joyce, Ruiping Wang, Marcin Pawlak Mining and Mineral Sciences Laboratories, Natural Resources Canada

The Mining and Mineral Sciences Laboratories (MMSL) of Natural Resources Canada (NRCan) have recently had increasing numbers of requests for the analysis of different species of metals such as antimony. This is due primarily to that fact that the toxicity and biological behavior of antimony depends on its chemical form and oxidation state.

Following a client request, the analytical service group (ASG) of MMSL developed a method to measure the concentration of antimony species, Sb(III) and Sb(V). This was in support of a study on the rate and extent of the release of inorganic antimony and its species at the ppb concentration level from antimony bearing substances into a testing medium OECD 203. OECD 203 is an aqueous ecotoxicity medium containing $CaCl_2$, $MgSO_4$, $NaHCO_3$ and KCl.

To achieve separation of Sb(III) and Sb(V) at ppb concentrations, an HPLC was directly coupled to an ICP-MS. Good baseline separation as well as stable and well defined Sb(III) and Sb(V) peaks were attained using an AS14/AG14 ion exchange column and 2mM concentration of EDTA as the mobile phase. With a 25µl injection volume, the limits of detection of 0.2µg/L for both Sb(III) and Sb(V) were obtained. Under these optimized conditions, analysis times of less than four minutes were achieved.

The analysis of species was performed on two separate LC-ICP-MS systems under different operating conditions. The two LC-ICP-MS systems consisted of a HPLC (Dionex) coupled with a Perkin Elmer 6100DRC ICP-MS and a Thermo Fisher X-Series II LC-ICP-MS. The speciation results from both systems were in very good agreement.

Both ICP-MS units were used to determine total elemental antimony and although the calibration standards were from a different source, the results generated were comparable to those from the speciation analysis using the LC-ICP-MS technique.

In addition, the method was validated by comparing total elemental antimony against the sum of Sb(III) and Sb(V) and verified against total antimony in a certified reference material. This simple method was robust and produced timely results for thousands of samples.

10:30 am

X-ray diffraction in iron and steel industries – grade control, process optimization and minimizing energy consumption and CO_2 emissions

Uwe König, PANalytical B.V., Almelo, Netherlands, Jennifer Anderson, PANalytical, Luciano Gobbo, PANalytical, and Katherine Macchiarola, PANalytical, Inc.

X-ray diffraction (XRD) has evolved from a research tool capable of providing useful information in terms of quantification of the crystalline phases and the amorphous content to a fully mature technique for quality and process control in mining, steel, cement and aluminum industries. Knowledge of the mineralogical phases present in an ore body allows operators to optimize mining and process operations (flotation, separation, etc.). Furthermore, XRD can be used as an efficient tool for identifying the means to reduce CO2 emissions through knowledge of phase composition of starter materials and the subsequent selection of those materials that process more efficiently into finished products.

Cluster analysis of XRD data facilitates multi-dimensional compositional mapping of ore deposits and drill cores, identifying regions of favorable mineral compositions. Automatically sorting closely related diffraction data into separate clusters and identifying the most representative scan of each cluster as well as outlying patterns. The paper describes several case studies where XRD and data clustering are used for grade control of iron ores, analysis of the iron ore sinters, determination of process relevant variables like the metallization in Direct Reduced Iron (DRI) and the phase analysis of ferrochrome slags. Details of the techniques used, sample optimization methodologies, results, data precision and limitations will be presented and discussed. The analytical approach presented here has enormous potential as an inexpensive, reliable tool, useful in the characterization of materials and the minimization of CO_2 emissions and energy consumption.

11:00 am

Advances in XRD and XRF Spectroscopy! Combining XRD and XRF in ONE instrument or by intelligent data integration!

Alexander Seyfarth, Bruker AXS Inc., Heiko Ress, Bruker AXS Inc., Karsten Knorr, Bruker AXS

During the oral presentation we will show the newest addition to the XRD product line: using the XFLASH™ 4th generation Silicon Drift Detector in the benchtop XRD unit D2 PHASER.
This is the first time that a SDD detector is used in an XRD system and it offers new capabilities:

- Efficient suppression of "undesired" fluorescence" radiation
- Selection of monochromatic excitation for improved pattern resolution
- EDX XRF based elemental determination of Ca in Aluminum bath in conjunction with XRD data
- EDX XRF based qualitative analysis and combined phase ID

The obtained data then present s itself to be automatically quantified by BRUKER's unique TOPAS standardless XRD program, yielding the ability to deliver XRD and XRF process data from a SMALL BENCHTOP unit. We will be showing Application studies ranging from the Aluminum Refining Industry to Minerals and Mining projects as well as applications from Exploration. The talk will conclude with an outlook on new methodology which allows the chemo metric treatment of classic XRF and XRD data.

11:30 am

Assayer Certification Program (ACP) British Columbia

Manzur (Mac) Chaudhry, Chair, Board of Examiners for Government of BC Assayer Certification Program

This presentation will provide a history of the Government of BC Assayer Certification program and it will give an update on activities of the Foundation and Advisory Council partnership that supports this program.

12:00 noon

Quality Assurance/Quality Control (QA/QC) Requirements in a Modern Chemistry Laboratory

Joseph A. Mastroianni, Scientech

This presentation covers the Laboratory's requirement to develop a Quality Management System (QMS) that meets their customer's needs.

Laboratories are required to demonstrate the accuracy and precision of their test methods in order to meet customer, regulatory and process requirements.

The QMS is used to define the overall requirements for demonstrating accuracy and precision including; instrument quality checks, intra and inter-laboratory check programs, methods for determining analytical method accuracy and precision requirements (also known as the Valid Requirements), analytical method selection and verification (including LOD, ULQ determinations), analytical method interferences, development and use of Control Charts, determination of uncertainties (analytical and total) and the review and reporting of final analytical method results.

Scientech, a Curtiss-Wright Flow Control company, has expert knowledge in developing and implementing laboratory QMS, providing relevant training and providing appropriate software solutions for laboratory control charting (Lab Stats Pack™).