Analystes des Minéraux Canadiens

Canadian Mineral Analysts

39th Annual Conference

Ottawa, Ontario

September 16 – 20, 2007

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CMA 2007
39th Annual Conference and Exhibit
Ottawa

Conference and Social Program

Sunday, September 16th
12:00 pm – 6:00 pm Conference Registration

Monday, September 17th

8:00-12:00	Conference Registration
8:00-8:30	Coffee Break
8:30-3:30	Fire Assaying Workshop
	Presented by: Quality Analysis Consultants-Dr. Wes Johnson
10:00-10:30	Coffee Break
12:00-1:00	Lunch
2:00-2:30	Coffee Break
5:30-7:30	Exhibitors Wine and Cheese Official Unveiling
	Guest Speaker: Denis Lagace. Director General, Policy and Planning Branch and Explosives Safety and Security Branch, Minerals and Metals Sector, Natural Resources Canada

Tuesday, September 18th

8:00-5:00	Exhibits Reconvene
8:00-8:30	Coffee Break
8:30-12:00	Session 1: Technical Presentations
10:00-10:30	Coffee Break
12:00-1:00	Lunch
1:00 4:00	Session 2: Technical Presentations
2:00-2:30	Coffee Break
5:00 10:00	Chelsea Dinner Train Event
	(Bus departs from Lord Elgin Hotel lobby)

Wednesday, September 19th

8:00-12:00	Exhibits Reconvene
8:00-8:30	Coffee Break
8:30-12:30	Session 3: Technical Presentations
10:00-10:30	Coffee Break
1:00-2:00	CMA Business Lunch
2:00-3:00	CMA Business Meeting
5:30-9:30	Annual Banquet
	Guest Speaker - Hon. Mrs. Beverley Lepine, CEO Royal Canadian Mint

Thursday, September 20th
9:30-12:00 Tours to NRC and RCM (departure from Lord Elgin Lobby)

Workshop Presented by:
Quality Analysis Consultants - Dr. Wes Johnson
(Lady Elgin Room)

Monday September 17th, 8:30 - 3:30

Fire Assaying: Basic Principles of Fire Assay Analysis and Related Trouble shooting Techniques

Workshop Facilitator: Dr. Wes Johnson of Quality Analysis Consultants.

The workshop is a full day presentation and discussion on Fire Assaying. The morning session topics will cover a technical review of Fire Assay Techniques with discussion centered on general principles .

The afternoon session topics will cover trouble shooting in fire assay processes, and instrument techniques for samples derived from the fire assaying processes such as FAS and ICP.

CMA 2007 Scientific Program

Tuesday, September 18, 2007

Session 1
Chair: Jon Forrest – Royal Canadian Mint

8:30 Analysis Of Limestone Using a Low Powered WDXRF System
Al Martin

9:00 Determination Of Silver 7 Gold In Silver Bullion By X-Ray Fluorescence Spectrophotometry
Mike Hinds

9:30 Quantitative Determination Of Calcite And Dolomite in Carbonated Soils and Sediments Using a Coulometric Titrator
Isabelle Girard, Raymond Boisvert, Alain Grenier, Claudia Moore

10:30 Universal Sample Preparation Methods Revisited
John Anzelmo, Alexander Seyfarth

11:00 Plasma Spectroscopy Interference Reduction Strategies
Ron Cardinall

11:30 Application of Advanced X-Ray Diffraction Analyses to Solving Issues Related to Ore and Feed Quality: A Case Study Involving a Magmatic Fe-Ni Cu-S Mine, Sudbury, Ontario, Canada
Ian Campbell, Katherine Macchiarole, Jennifer Anderson, Andrew McDonald

Tuesday September 18, 2007

Session 2
Chair: Christine Scriver – National Research Council Canada

1:00 "Method Development in a Global Environment"
Kevin Bunten

1:30 Current State of Proficiency Test and Reference Materials for Gold Bullion
Mike Hinds

2:30 A Benchtop Total Reflection X-Ray Fluorescence Spectrometer for Depth Profiling and PPB Levels of Detection
Al Martin

3:00 Sample Preparation for Trace Element Analysis in Precious Metal Alloys Using the Spark Ablation Technique
Jon Sewell

3:30 Semi-Automated Sample Preparation System
Kurt L. Headrick, Brock McNichols

Wednesday September 19, 2007

Session 3
Chair: Dr. Wes Johnson-Quality Analysis Consultants

8:30 Wide Range of Analysis of Oxides in Geological Material Using X-Ray Fluorescence Spectrometry
M. S. Olde Weghuis, B.A.R. Vrebos, S. Kuiper, M.N. Ingham

9:00 CRI (Collision Research Interface) – A Simple Approach to Interference Management ICP-MS
Doug Shrader, Shane Elliot, Xue Dong Wang, and Iouri Kalinitchenko

9:30 Lab QC Versus Mine QC. Why They Are So Often In Conflict And Suggestions To Improve Both?
John Winterbottom

10:30 The Presentation Addresses the Mandate and Structure of the SCC, It's Regional and International Links, Accreditation Programs and Program Specialty Areas, with a Particular Emphasis on Laboratory Accreditation in the Field of Mineral Analysis Testing
Rassoulou Diallo

11:00 Automatic Rietveld Based Quantitative Phase Analysis of Minerals Using Topas in Combination With same Sample WD-XRF
A. Seyfarth, Rainer Schmidt

11:30 "Using Geometallurgy To Reduce Risk"
Richard Wagner

12:00 Automated Flux Dispenser
Bill Clifford

12:20 Preparation of Inorganic Certified Reference Materials at the National Research Council Canada
Christine Scriver, Scott Willie, Ralph Sturgeon

Abstracts

Analysis of Limestone using a Low Powered WDXRF System

A. Martin, Rigaku Americas Corporation

The approximately 970+ power plants that burn oil and coal to produce electricity in America have been sited as a major source of Sulfur Dioxide (S02) emissions. This pollutant can irritate the upper respiratory tract and damage lung tissue, as well as yellowing plant leaves and eating away iron, steel, marble and other solid materials. Through this, the demand for limestone is increasing due to a legislated requirement for S02 flue gas desulphurization systems (scrubbers) to be installed in these facilities. Because of this ever increasing demand, limestone quarry lab technicians are required to take samplings frequently and respond quickly to certify product ready to release by truck or train load.

This discussion demonstrates an efficient method for limestone analysis using a pressed pellet technique combined with a low powered WDXRF system. The analysis of light elements, such as sodium and magnesium, can be performed with better sensitivity and overall improved element resolution compared to similar EDXRF units and without the need of higher powered WDXRF systems. The target area for this procedure are the limestone quarries but it can be seen that down-line companies can also benefit from this routine as the method satisfies the ASTM repeatability requirements for C1271 (Standard Test Method for X-ray Spectrometric Analysis of Lime and Limestone). Quarries obtain the added bonus of also being able to analyze typical geo-exploration samples for pit development/expansion.

Determination of Silver and Gold in Silver Bullion by X-Ray Fluorescence Spectrometry

Michael W. Hinds, Royal Canadian Mint

This paper will present the current state of the X-ray Fluorescence (XRF) Spectrometric method designed to determine gold and silver in silver dore bars. Samples with a minimum diameter of 29 mm are collected from the molten silver material. The samples are pressed and polished prior to analysis on the XRF using 27 mm aperture sample cups. Other elements such as copper, bismuth, lead, and tellurium are also determined to ensure maximum impurity levels are not exceeded for the refinery operations and to ensure accurate XRF determinations for all elements of interest due to inter element effects. The present XRF method with a 12 mm aperture determines Ag at 905 fineness with standard deviation of ± 0.32 fineness. Using a 27 mm aperture will give a lower standard deviation for the same counting time and will provide greater counts for trace elements in the sample. Better accuracy is also expected by using calibration standards cast from typical silver dore material with some extra elements added in the casting stage.

Quantitative Determination of Calcite and Dolomite in Carbonated Soils and Sediments Using a Coulometric Titrator

Isabelle Girard, Raymond Boisvert, Alain Grenier, Claudia Moore, Natural Resources Canada

A new procedure for the quantitative analysis of calcite and dolomite in carbonated soils and sediments has been developed at the Sedimentology Laboratory of the Geological Survey of Canada. The quantification is performed by selectively decomposing calcite using a citric acid solution generating gaseous carbon dioxide. The evolved carbon dioxide is then quantified using a coulometric titration instrument. Dolomite can be determined by calculating the difference between total carbonate content and calcite content. The detection limit, quantification uncertainty of the measurements and ruggedness data are presented along with plans for future developments .

Universal Sample Preparation Methods Revisited

John Anzelmo, Alexander Seyfarth, Claisse USA Bruker AXS, Inc.

It is well known that the preparation of metals and liquids for XRF analysis is fast, and easy, and usually requires little strategy. The preparation of powder samples such as silicates, carbonates, slags, cements, ferro-alloys and other powdered materials however requires careful planning in the way of additives, lubricants, binders, backing and holders. The ratio of binder to sample must include forethought regarding the homogeneity of the briquette, the stability of the briquette, the cleaning and cleanliness of the grinding vessel, and the performance requirements of the analysis.

Various strategies will be revisited. Due to the well known "mineralogical effect", it may be necessary to use the fusion method of preparing homogenous glass disks with a common amorphous structure, to insure accuracy in the XRF determination. An overview of the fusion process, automation strategies, and XRF calibration will be discussed. Case studies will be shown ranging from mining to process control.

Plasma Spectroscopy Interference Reduction Strategies

Ron Cardinali, Product Specialist, PerkinElmer LAS Canada Ltd.

Assayers and mineral analysts are faced with some of the most unique challenges that are found in spectroscopic techniques. Fortunately there are many varieties of easy to use techniques available to ensure accurate analysiS. Numerous strategies in successfully reducing interferences in ICP-OES and ICP-MS techniques will be discussed, including reaction gas techniques, using examples found in the geochemical and metal analysis field.

Application of advanced X~ray diffraction analyses to solving issues related to ore and feed quality: A case study involving a magmatic Fe~Ni~Cu~ mine, Sudbury, Ontario, Canada
Campbell, Ian; Macchiarola, Katherine; Anderson, Jennifer, PANalytical Inc., McDonald, Andrew M Department of Earth Sciences, Laurentian University

X-ray diffraction (XRD) can provide useful information about the composition of ore and feed samples in terms of the identification and quantification of crystalline phases. Knowledge of the phases present can be useful in developing effective processing strategies for the beneficiation (flotation. magnetic separation etc.) of either blended ores or chemically distinct ore bodies within operating mines. The processing required to beneficiate the ore may also dictate the best ore bodies to mine. Quantitative analysiS of X-ray diffraction data is possible by various classical methods such as straight line or polynomial calibration with standards, but modern quantification analysis techniques such as Rietveld analysis or full pattern autoscale analysis are attractive alternatives. The Rietveld method offers impressive accuracy and speed of analysis and does not require the use of standards. A Rietveld refinement compares calculated *vs.* experimentally derived X-ray powder diffraction patterns for a phase, adjusting a wide-variety of crystallographic-, chemical-and modal-abundance parameters until the two are in agreement.

A case study of the XRD analysis of hexagonal and monoclinic pyrrhotite phases, which are often associated with base and precious metal ore bodies, will be presented, with comparisons to other analytical techniques. Understanding the ratios of these phases is an important component of any study relating the genesis, mineral distribution and subsequent beneficiation of magmatic ores. Details of the techniques used, sample optimization methodologies. results, data preCision and limitations will be discussed. The approaches have enormous potential as an inexpensive, reliable and rapid tool, useful in the characterization of ore materials from any geological environment.

Method Development in a Global Environment
Kevin Bunten, ALS Chemex

The development of streamlined methods in a global organization presents many interesting challenges. Employing global strategies for the analysis of mineralogical samples is important for ensuring uniform quality control and high sample throughput. We have developed a modified method for the aqua-regia digestion and analysis of ore-grade base metal materials. This method overcomes a precipitation issue encountered with samples containing high concentrations of Pb and Ag, in high sulphide samples with out the use of extra complexing agents such as ammonium acetate and sodium thio-sulphate. The challenges of method validation and reliability when dealing with materials from around the world will be discussed.
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Current State of Proficiency Tests and Reference Materials for Gold Bullion
Michael W. Hinds, Royal Canadian Mint

Over the past 15 years, there has been a steady demand for proficiency testing programs and reference materials for all forms of analytical work. The precious metal industry is no different. For producers of gold dore, the American Society for Testing Materials provides a proficiency testing program. Two sets of samples are sent out at 6 month intervals each year and cover a range of compositions. A summary of results will be presented as well as examples of the information participants receive. Alternatively, the Royal Canadian Mint has produced a set of Ag-Au-Cu alloy Reference Material (BRM series), which can be used by assayers to verify their fire assay procedures. Other reference materials have been prepared on a custom basis by the RAND Refinery. This year a group of fine gold and silver assayers have united under the auspices of the London Bullion Marketing Association (LBMA) to produce and certify the first true set of silver and gold reference materials with 20 -25 trace elements in each matrix. The current state of this endeavour will be discussed.

A Benchtop Total Reflection X-ray Fluorescence Spectrometer for Depth Profiling and PPB Levels of Detection
A. Martin, Rigaku Americas Corporation

NanoHunter is the world's first benchtop Total Reflection X-ray fluorescence (TXRF) spectrometer that provides ultra trace level elemental analysis and evaluation of the physical nature of the sample. Using patented wavelength selection and an automated variable X-ray incidence angle excitation, this instrument analyzes the full range of elements -from Al to U -in solids, liquids, and powders and can achieve lower limits of detection rivaling or surpassing ICP. The NanoHunter is not limited to extraordinary sensitivity for elemental analysis; the ability to measure samples with an excitation beam that has a variable grazing incidence angle allows the determination of additional physical and chemical information on a sample. Specifically, the instrument is capable of differentiating the nature of a deposition or coating.

Sample Preparation for Trace Element Analysis in Precious Metal Alloys Using the Spark Ablation Technique
J. Sewell, Royal Canadian Mint

Improvements in ICP-AES instrumentation and Spark Ablation techniques have led to improved detection limits and accuracy for faster and more cost effective analysis of trace level impurities in precious metals. At the trace level the sample preparation becomes increaSingly important as well as having reliable and homogeneous standards. Adjustments and modifications to the instrument are required, as well as trial and error using different applied techniques. Samples are treated and polished in an effort to reduce spiking or salting of the analyte of interest. With proper handling, accurate analysis of trace level elements are achievable at 1 ppm or lower. Having detection limits in this range, fast and accurate analysis of 99.99% purity can be obtained easily, with the potential for 99.999% purity within reach.

Performance of a Semi-Automated Sample Preparation System
Kurt L. Headrick, Voisey's Bay Nickel Co. Ltd., Voisey's Bay Mine Site
Brock McNichols, University of Waterloo, Chemistry Department

The semi-automated sample preparation system used in the Assay Labs at Voisey's Bay is described. Robust pressed-pellets for XRF analysis are obtained for both sulphide ore and clean rock samples, using one set of experimental conditions without binder. A cross-contamination study found that the effectiveness of the automated cleaning systems are petrology and element dependent, and proportional to mineral densities.

Wide Range Analysis Of Oxides In Geological Material Using X-Ray Fluorescence Spectrometry
M.S. Olde Weghuis, BAR. Vrebos, D. Kuiper, PANalytical, M.N. Ingham British Geological Survey.

Accurate and precise analysis by means of X-ray fluorescence (XRF) spectrometry is still a comparative method of analysis. Hence, reference materials are required to calibrate the analytical system. In cases involving several analytes a fairly large number of these reference materials is required. For 'easy' applications, such as the analysis of certain grades of stainless steel, the procurement of reference materials is no problem (other than the cost involved). These reference materials can then be used for extended periods of time because the specimen preparation for alloys is limited to surface lapping. For materials of a specific geological origin, however, reference materials are hard to find. One has then to work with materials from different origin, some of which contain elements of no interest for the particular analysis on hand at significant concentration levels. These 'unwanted' elements often require additional effort in the determination of net intensities and/or matrix correction.

An effort to alleviate the search for reference materials by using a set of synthetic oxide standards will be discussed. These reference materials are made from high purity compounds and can be used to calibrate an Xray spectrometer for 21 major and minor oxides. The spectrometer software uses reliable methods of background correction and a fundamental parameter method for matrix correction.

The proposed sample preparation is by fusion in a mixture of 66% lithium tetraborate and 34% lithium metaborate. This method is applicable to a wide variety of materials of geological origin. The use of synthetic standards made from pure traceable compounds brings XRF closer to being a primary rather than a comparative method. Reference materials were used to validate the method described and examples of accuracy for major constituents are given using ores and other minerals. This method has successfully been used to analyze a wide variety of minerals and ores, as validation data shows .

CRI -A Simple Approach to Interference Management in ICP-MS
Doug Shrader, Shane Elliott, Xue Dong Wang and Louri Kalinitchenko Varian, Inc.,

Management of polyatomic and mass interferences in ICP-MS has been a popular area of research and topic of discussion in recent years. A novel interference management system for ICP-MS, the Collision Reaction Interface (CRI), will be discussed during this presentation. This unique technology employs simple collision and reaction gases injected directly into the plasma through the tips of the ICP-MS interface cones. This approach quickly and effectively reduces or eliminates common interferences on elements such as As, Se, Cr, V and Fe, achieving lower detection limits in hot plasma, even for samples with complex matrices. Coupled with revolutionary gO-degree reflecting ion optics and a low noise, double off axis quadrupole mass analyzer, the CRI-ICP-MS offers an efficient yet simple solution to interference management in ICP-MS.

The de sign o f t his uni que C RI-ICP-MS will be di scussed, al ong w ith pr inciples of oper ation, performance attributes and examples of its application to environmental, biological and speciation samples.

Lab QC Versus Mine QC. Why They Are So Often iIn Conflict And Suggestions To Improve Both.
John Winterbottom, AssayNet Canada Inc.

Analytical chemist in a mine site lab should not only monitor their own quality, they should expect their customers to do the same. In a perfect world these internal and external systems would complement each other. In practice however, they seldom do. Disputes and conflicts often arise and instead of illuminating, the different systems produce more heat than light.

To define just what we mean by quality control because, in practice, it often means different things to different people. We will examine the rationale behind a quality

Accreditation in Canada
Rassoulou Diallo, Standards Council of Canada

Regulators and users of products and services are plaCing increased reliance on testing by accredited laboratories as an assurance of conformity to specified requirements.
The Standards Council of Canada (SCC) is a federal Crown Corporation, with the mandate to foster and promote efficient and effective standardization in Canada.
The S CC se rves as the G overnment's focal point for v oluntary st andardization and r epresents Canada i n i nternational st andardization act ivities. It se ts out pol icies and pr ocedures f or t he development of National Standards of Canada, and for the accreditation of standards development organizations, of certification bodies, of testing and calibration laboratories, of quality and environmental m anagement sy stems registration bodi es and of quality m anagement sy stems and environmental auditor certifiers. The SCC also promotes and supports the principle of recognition of accreditation or equivalent systems as a means of decreasing the number of multiple assessments and audits both in Canada and with Canada's trading partners.
This presentation addresses the mandate and structure of the SCC, its regional and international links, accreditation programs and program specialty areas, with a particular emphasis on laboratory accreditation in the field of mineral analysis testing

Automatic Rietveld Based Quantitative Phase Analysis Of Minerals Using Topas In Combination With Same Sample WO-XRF.

A. Seyfarth, Rainer Schmidt, Bruker AXS Inc,
The paper will give an introduction to quantitative phase analysis using the TOPAS software based on the Rietveld method. Case studies using examples from the mining industry and refractory industry are shown where the method is used in a QC environment in conjunction with WD XRF. The fundamental parameter approach of the TOPAS software allows the creation of a sturdy "recipe" for the refinement which can be used to automate the analysis and also enables the unattended operation.

Geometallurgy
Richard Wagner, SGS Canada

What is geometallurgy? Geometallurgy is the informed selection of a number of test samples that are used to determine and to distribute specific metallurgical parameters throughout the ore blocks. The metallurgical parameters are distributed onto the ore blocks using accepted geostatisical techniques. The metallurgical parameters assigned to the ore blocks which supports metallurgical modeling and allows virtual mining and processing of each ore block, on a block-byblock basis to provide ore throughput and concentrate production forecasting, thereby maximizing the deposit cash flow and optimizing the mine planning. The distribution of the metallurgical parameters on the block models are influenced by the geology, structure and lithology. Geometallurgy complements, but does not replace the traditional metallurgical approach during the development and operation of a mine ore body.
The benefits of geometallurgy are to significantly reduce the impact of spatial uncertainty in mine planning because it documents the variability in a deposit. Reducing the spatial uncertainty lowers technical and financial risks of the orebody development. Geometallury is a costeffective technique used to characterize ore variability and its effect on metallurgy and is well suited to be applied during exploration, development or operational stages of the mine development. The key deliverables are that geometallurgy develops the understanding and links between mine geology, mineralogy, ore grades, ore grindability, tonnage throughput and metallurgical performance, which leads to the forecast of expected mine revenue, profitability and performance block-by-block, year-by -year basis.

Automated Flux Dispenser
Bill Clifford, Anachemia Science

This presentation will detail the operations of the newly developed Flux Dispenser. The device simultaneously delivers up to 140 gr (+/-) 3 % to as many as seven crucibles and limits exposure to operators and technicians to lead dust.

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Preparation of NRCC Environmental CRMs

Christine Scriver, Scott Willie and Ralph Sturgeon National Research Council Canada -INMS Chemical Metrology

The production of Certified Reference Materials (CRM) has been undertaken for some 30 years by the Chemical Metrology group at the Institute for National Measurement Standards in Ottawa. This presentation will describe the role NRC performs as a center for dissemination of valid chemical measurements and their traceability in Canada as well as research conducted for environmental chemical analysis.
CRMs undergo rigorous assessment at all stages of production, beginning with a critical view of their analytical and economic demand. Once identified, the candidate material is sourced or collected and subjected to an initial homogeneity and stability assessment for the measurands of interest. If determined to be fit for purpose, the material is then suitably bottled, packaged and sterilized. The process of analytical characterization then begins, following an established NRCCINMS Chemical Metrology value assignment and uncertainty estimate philosophy which ensures linkages with other international organizations serving to ensure traceability to the SI. Examples of this process will be presented.

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