Focus ON

Geochemical Laboratory Updates

Special thanks to Chris Benn for assembling focus topics for this issue, Geochemical Laboratory Update, and Geochemical Standards Discussion

Controlling the Quality of Kimberlite Indicator Mineral Processing Using Indicator Mineral Spikes

Introduction

Overburden Drilling Management Limited (ODM) strives in its Ottawa, Canada, heavy mineral laboratory to achieve an 80-90 percent recovery rate when processing samples for kimberlite indicator minerals (KIMs) and other coarse-grained indicators. It is important for both ODM and the company's clients to know how achievable this objective is and to what degree it is being achieved. Therefore, on kimberlite exploration projects, ODM regularly conducts internal tests to measure the rate of KIM recovery. Control samples spiked with known numbers of KIM grains are employed in these tests.
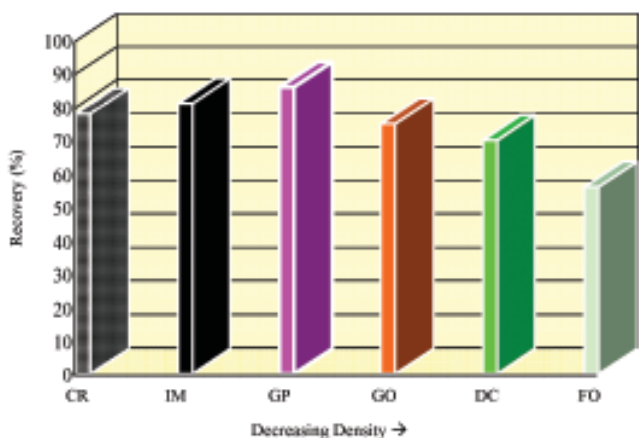

Figure 1: Relationship between KIM density and KIM recovery for 0.25-0.5 mm grains.

ODM recovers KIMs by gravity means and then sieves and magnetically and electromagnetically separates the gravity concentrates to ease KIM identification. The gravity processing involves primary shaking table preconcentration followed by a secondary sink-float separation in the heavy liquid methylene iodide (S.G. 3.32) which is diluted with acetone to S.G. 3.20 because the density of the KIMs ranges from 3.25 to 5.1 (Table 1). Both the methylene iodide and acetone are reclaimed by distillation. The sieve products employed are 0.25-0.5, 0.5-1.0 and 1.0-2.0 mm, equivalent to medium, coarse and very coarse sand, respectively.

The spike tests are designed only to measure the rate of KIM recovery during gravity concentration, not the ability of ODM's mineralogists to observe the KIMs in the final

Exploration Technology Workshop: *Discovery* Thru *Innovation*

Summary of a workshop held April 12–13, 2002 in Denver, Colorado

This two-day workshop was held in conjunction with the Society of Economic Geologists (SEG) meeting in Denver in April, 2002. It was intended to evaluate current exploration technologies and their role in integrated exploration programs, with perspectives from the large, small and junior companies. The workshop was co-sponsored by the Association of Exploration Geochemists; AEG scholarships were provided to two student attendees. State-of-the-art reviews of exploration geophysics, geochemistry, remote sensing, GIS and 3-D technology, as well as successful discoveries based upon integrated exploration, were presented, and a panel discussion investigated strategies for effective implementation and integration of these technologies into the exploration environment. The workshop was organized by Graham Closs, Mary Doherty, and Ken Witherly.

Geochemical exploration methods, from conventional to leading edge, were summarized in one afternoon by geochemists active in the mineral exploration industry. This article provides a brief summary of each of the presentation but cannot adequately convey the full message of each contributor. Individual authors may be contacted for further information.
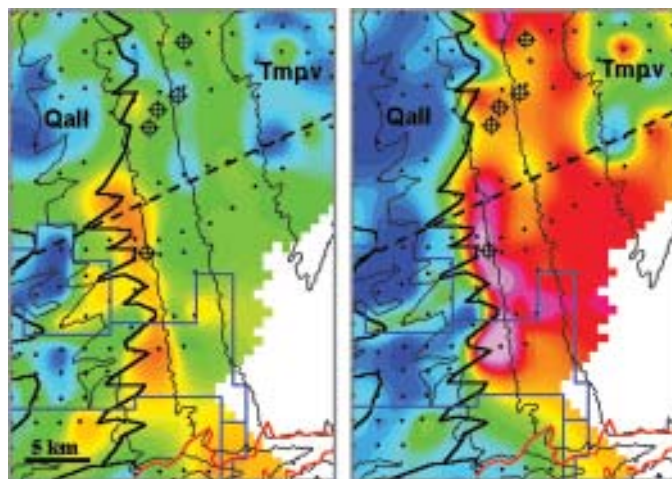

Figure 2: Comparison of geochemical images from Cu data before (left) and after (right) adjusting for landscape variation.

Graham Closs started the geochemistry session with an introduction to exploration geochemistry. Almost by definition, exploration geochemistry itself is an integrative exercise. Programs can be considered in terms of five components: (1) design and planning, (2) field sampling,

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refined products. In fact, each concentrate is re-examined by a second mineralogist to ensure that no recovered grains are overlooked. As well, only the 0.25-0.5 and 0.5-1.0 mm (medium and coarse sand) fractions of the sample are spiked because natural KIMs of 1.0-2.0 mm size are rare, generally constituting <1 percent of the total KIM population in anomalous sediments.

The most comprehensive spike test involves 10 samples and is conducted annually. This article summarizes the results obtained from the 2001 spike test. Six of the seven best-known KIM species were used: 1) chromite (CR); 2) Mg-ilmenite (IM); 3) purple to red Cr-pyrope garnet (GP); 4) orange Cr-poor pyrope garnet (GO); 5) Cr-diopside (DC), and 6) forsterite (FO). The seventh mineral, orange eclogitic pyrope-almandine garnet, was not used because it is too similar visually to Cr-poor pyrope and can be reliably distinguished only by electron microprobe analysis. It is slightly heavier than Cr-poor pyrope (S.G. 3.9 versus 3.7; Table 1) and is assumed to be at least as recoverable.

Mineral	Symbol	Specific Gravity
Forsterite	FO	3.25
Cr-diopside	DC	3.3
Cr-poor pyrope	GO	3.7
Cr-pyrope	GP	3.8
Pyrope-almandine	GO	3.9
Mg-ilmenite	IM	4.7
Chromite	CR	5.1

Table 1: Relative densities of kimberlite indicator minerals.

Advantages of Using Internal Spike Tests

ODM's clients sometimes spike their own samples but this type of spiking is problem prone. One major problem is that ODM archives a 500 g character sample from every 10-20 kg field sample and this unprocessed subsample may contain some of the spiked KIM grains. Some clients exacerbate this problem by bagging the sample and then adding KIMs to the top rather than blending them into the sample before bagging it. Then it is impossible for ODM to extract an unbiased character sample from the bag; usually the character sample will be overly KIM rich and recovery rates determined from the remaining KIM-depleted material in the bag will be misleadingly low. As well, if any spillage occurs during shipping or when the bag is opened in the lab, the spillage may be biased to the top of the bag and therefore to the KIMs.

Another common problem with client spikes is that the samples may contain chromite, forsteritic olivine, Cr-diopside or pyrope-almandine garnet from non-kimberlitic mafic, ultramafic or metamorphic rocks. These "pseudoKIMs" are difficult to distinguish visually or chemically from their kimberlitic counterparts, especially from well-traveled KIMs that have completely lost their distinctive alteration mantles and resorption textures. In other cases, the client may: a) forget to add the intended KIMs to the sample or add them to a different sample (it

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—————————————— *continued from Page 2*

happens!); b) not fully verify the KIMs before adding them to the sample; c) use KIM grains freshly milled from kimberlite rather than natural grains from anomalous sediments; d) add an unnatural number or variety of KIM grains; or e) not ensure that the grain size of the KIMs is the same as that of the sample material and therefore appropriate for the equipment chosen to do the processing. For example, the client may spike till samples only with KIM grains of coarse sand size (0.5-1.0 mm) because coarse grains are easy to identify and handle, but in natural till samples KIMs of medium sand size (0.25-0.5 mm) tend to be five to ten times more abundant (with the exception of IM which occurs subequally in both size fractions) and ODM's tabling procedure is purposely tuned when processing till to maximize the recovery of the more plentiful, medium sand sized KIMs.

A well-designed internal laboratory spiking program can eliminate all of the above problems while remaining as objective and impartial as a client spiking program. ODM has adopted strict procedures to ensure that its annual spike tests are as natural, representative and practical as possible. The ten spiked samples are distributed among at least 1000 natural project samples over a period of four to six months, thereby randomly utilizing most of the laboratory equipment and personnel and minimizing the potential for spike recognition during processing. The base samples used in the tests are actual project samples which prove to be barren of both KIMs and pseudoKIMs when first processed and are then reassembled, spiked and reprocessed. The KIMs chosen for spiking are natural, transported grains extracted from anomalous sediments. Before use, all grains of the visually least distinct (relative to ordinary heavy minerals) KIM species, GO, CR and FO, are verified by energy-dispersive

x-ray spectrometry (EDS) analysis to ensure that they are genuine KIMs Within the 0.25-0.5 and 0.5-1.0 mm particle size groups, mid-sized grains are selected to ensure that all grains report to the correct size fraction during processing. Grains close to the 0.25, 0.5 or 1.0 mm sieve sizes are expressly avoided. The grains include both typical and atypical KIM specimens; however, structurally weak grains that could break during processing are avoided. The only unnatural feature of the tests is that the overall ratio of fine (0.25-0.5 mm) to coarse (0.5-1.0 mm) grains is maintained near 1:1 rather than the natural 5:1 to 10:1 in order to equitably compare recoveries in the two size fractions.

Specifications for the 2001 SpikeTest

The 2001 spike test involved a single project — Operation Treasure Hunt of the Ontario Geological Survey (OGS, 2001) — and was designed as a quality control for this project. During Operation Treasure Hunt, 1912 samples of alluvial sediments, principally sand and gravel, were collected from a 600 km long structural corridor stretching from Lake Huron to Georgian Bay. ODM processed these samples between June 22, 2000 and March 07, 2001. The ten spiked samples were inserted between September 19, 2000 and February 26, 2001.

The samples were variably spiked with CR, IM, GP, GO, DC and FO grains. Since CR and IM are visually similar, grains of different sizes were employed whenever both minerals were added to the same sample. Altogether, from 22 to 40 grains of each size of each of the six KIMs were employed for a total of 191 grains in the 0.25-0.5 mm fraction and 155 grains in the 0.5-1.0 mm fraction.

Identifying and where possible rectifying avenues of KIM loss during processing is as important as establishing KIM recovery rates. Therefore the two main rejects generated during heavy mineral processing — table tails and heavy liquid floats or "lights" — were reprocessed once and checked for KIMs.

Test Results
0.25-0.5 mm KIM Recovery Rates

Overall 0.25-0.5 mm KIM recovery rates for the ten individual samples range from 43 to 100 percent (Table 2). Recoveries of the six individual minerals are, as expected, strongly density-dependent (Figure 1 - see page 1) diminishing steadily from 81-85 percent for IM (S.G. 4.7; Table 1) and GP (S.G. 3.8) to 56 percent for FO (S.G. 3.25). The recovery rate for the heaviest KIM, CR (S.G. 5.1) is below that for IM and GP but this is due solely to the accidental loss of four of seven spiked CR grains from Sample 10 when the heaviest part of the table concentrate was temporarily removed and micropanned for gold grains (Operation Treasure Hunt was targeted on gold grains and base metal and carbonatite indicators in addition to KIMs). No other 0.25-0.5 mm KIMs were added to Sample 10; consequently the extraneous CR loss lowered the 0.25-0.5 mm KIM recovery for Sample 10 to 43 percent (Table 2), well below the 61 to 100 percent level achieved for the other nine samples.

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Sample No	CR Spiked	CR Recovered	IM Spiked	IM Recovered	GP Spiked	GP Recovered	GO Spiked	GO Recovered	DC Spiked	DC Recovered	FO Spiked	FO Recovered	Total Grains Spiked	Total Grains Recovered	Total % Recovered
1	10	9	0	0	5	4	0	0	0	0	0	0	15	13	87
2	0	0	0	0	4	3	10	7	4	1	0	0	18	11	61
3	15	11	0	0	9	9	0	0	6	3	0	0	30	23	77
4	0	0	16	13	5	4	8	5	0	0	12	6	41	28	68
5	8	8	0	0	8	7	0	0	0	0	0	0	16	15	94
6	0	0	0	0	0	0	0	0	4	4	0	0	4	4	100
7	0	0	0	0	0	0	0	0	8	7	10	4	18	11	61
8	0	0	0	0	0	0	0	0	8	6	10	8	18	14	78
9	0	0	10	8	8	6	6	6	0	0	0	0	24	20	83
10	7	3	0	0	0	0	0	0	0	0	0	0	7	3	43
Total Grains	40	31	26	21	39	33	24	18	30	21	32	18	191	142	
Total % Recovered	78		81		85		75		70		56		74		

Table 2: Summary of KIM recovery rates for the 0.25-0.5 mm fraction.

	Lost							Found in Table							TailsFound in Heavy Liquid Lights							Unaccounted						
Spike No.	CR	IM	GP	GO	DC	FO	Total	CR	IM	GP	GO	DC	FO	Total	CR	IM	GP	GO	DC	FO	Total	CR	IM	GP	GO	DC	FO	Total
1	1	0	1	0	0	0	2	0	0	1	0	0	0	1	0	0	0	0	0	0	0	1	0	0	0	0	0	1
2	0	0	1	3	3	0	7	0	0	0	0	0	0	0	0	0	1	2	3	0	6	0	0	0	1	0	0	1
3	4	0	0	0	3	0	7	0	0	0	0	1	0	1	0	0	0	0	2	0	2	4	0	0	0	0	0	4
4	0	3	1	3	0	6	13	0	0	0	0	0	0	0	0	0	0	1	0	1	2	0	3	1	2	0	5	11
5	0	0	1	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	1
6	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
7	0	0	0	0	1	6	7	0	0	0	0	0	1	1	0	0	0	0	1	0	1	0	0	0	0	0	5	5
8	0	0	0	0	2	2	4	0	0	0	0	0	0	0	0	0	0	0	1	1	2	0	0	0	0	1	1	2
9	0	2	2	0	0	0	4	0	1	0	0	0	0	1	0	0	0	0	0	0	0	0	1	2	0	0	0	3
10	4	0	0	0	0	0	4	0	0	0	0	0	0	0	0	0	0	0	0	0	0	4	0	0	0	0	0	4
Totals	9	5	6	6	9	14	49	0	1	1	0	1	1	4	0	0	1	3	7	2	13	9	4	4	3	1	11	32

Table 3: Distribution of lost 0.25-0.5 mm KIM grains. The table tails and heavy liquid lights were each reprocessed once in seach of the missing grains.

Technical Meeting Announcement

"Radioelement Mapping and Status of the Global Radioelement Baseline and Maps." Sponsored by IAEA, in cooperation with UNESCO, the International Union of Geological Sciences, and the Department of Geology and Geological Engineering, Colorado School of Mines.

The Meeting is scheduled for *June 23 - 26, 2003* and will be held at the Colorado School of Mines, Golden, Colorado, USA.

Contact Dr. Karen Wenrich, IAEA, at <K.J.Wenrich@iaea.org> for additional information.

Of the 191 KIMs of 0.25-0.5 mm size that were added to the samples, 142 were recovered and 49 were lost including the 4 extraneous CR grains lost from Sample 10. Reprocessing the table tails produced 4 of the missing KIMs (Table 3, Fig. 2) and reprocessing the heavy liquid lights produced 13 grains including 9 low-density DC and FO grains (Table 3, Fig. 2).

0.5-1.0 mm KIM Recovery Rates

Overall 0.5-1.0 mm KIM recovery rates for the ten individual samples range from 48 to 100 percent (Table 4). Recoveries of the six individual minerals are as density-dependent as in 0.25-0.5 mm fraction, diminishing steadily from 93 percent for CR to 50 percent for FO (Fig. 3). The only exception is that 0.5-1.0 mm IM recovery, like 0.25-0.5 mm CR recovery, suffered due to the accidental loss of a significant number of grains (4 of 7) during micropanning of the heaviest part of the table concentrate from Sample

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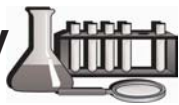
SGS MINERALS SERVICES

2002 saw SGS Minerals Services consolidate its position as a global leader in geochemical analytical services with the acquisition of Analabs Pty Ltd. and Lakefield Research Limited. SGS Minerals Services' global network provides clients with comprehensive exploration services for diamonds, base metals, gold, PGM's and industrial minerals — worldwide.

In Africa SGS has committed significant resources to upgrading its Johannesburg laboratory. By the end of the first half of 2003 the Johannesburg laboratory will have added new ICP-OES, ICP-MS and XRF instrumentation. New Fire Assay and Sample Preparation facilities using the latest in analytical technology including Nugget Crushers, LM 2 and 5 pulverizers, 50 pot furnaces and multiload and multipour technologies are under construction. The African laboratory group continued its leadership in the provision of on-site, purpose built laboratories for mines with the addition of new mine laboratories in the Democratic Republic of Congo and Tanzania and a robotic laboratory at the Scorpion Zinc mine in Namibia. SGS Minerals Services worked with IMP to install and operate the Scorpion robotic on-site laboratory and look forward to installing and operating more robotic laboratories around the world.

In Australia the SGS network of laboratories provides high quality, responsive analytical services to the Australasian exploration market. SGS Minerals Services continues to invest in the network with the recent addition of new XRF capability in Perth that will allow the laboratory to better service the Iron Ore exploration industry in Australia. In addition, the Australian Group continues to lead its competitors with an unrivaled network of on-site purpose-built mine laboratories servicing gold and base metal mines in the region.

In Mongolia SGS Minerals Services has tripled the capacity of its laboratory in Ulaanbaatar in order to better service the rapidly growing copper and gold exploration programs in the country. Exploration in Mongolia continues to grow and SGS Minerals Services intends to grow with the industry.

In South America the Group's joint-venture laboratory business in Brazil continues to be a leader in the provision of high quality, rapid exploration analytical services with significant investments in new analytical equipment in the Belo Horizonte facility in 2002. The Paraupebas laboratory in the Carajas was expanded to include Fire Assay and base metal analytical capability. Our laboratory in Lima, Peru expanded its services to include the provision of on-site, mine site laboratory operation. The Lima Group took over the operation of two mine-site laboratories in 2002 and has already significantly improved the analytical data quality and service to those mines.

In Canada the Group recently opened a sample preparation facility in Sudbury to service the local mining community, including those companies exploring for PGM's north of the Sudbury mining camp. The Fire Assay facilities in our Red Lake and Rouyn laboratories have been modernized with the commissioning of 50 pot gas-fired Fire Assay systems including multiload and multipour technology. In addition, the analytical measurement capacity of our Rouyn laboratory has been improved with the addition of ICP-OES. Our Don Mills ultratrace geochemical laboratory has developed a new method using a sodium peroxide sinter with ICP-OES and ICP-MS finishes. The ICMS90 method allows the determination of 54 elements at trace to ultra-trace levels. Use of the sinter reduces the loss of volatile elements from As to Zn compared to the more traditional lithium metaborate fusion, while a novel fusion vessel eliminates contamination. The Lakefield Mineralogy group has added new exploration services including QEM Scan™ and Image Analysis diagnostic mineralogical services, to boost their already extensive petrography and petrology services. In 2002 the SGS Minerals Services Diamond Exploration Group, headquartered in Lakefield, Ontario commissioned two 1 tonne per hour DMS diamond recovery plants with full X-ray sorting capability and processed over 1,000 tonnes of diamond bearing kimberlite. In 2003 the Diamond Group will be running a large 25,000 tonne bulk sample in Northern Saskatchewan featuring a new 10 tonne per hour DMS diamond recovery plant. The Diamond Group continues to innovate and has developed new diamond recovery flowsheets for diamond deposits in the Wawa area that do not respond well to conventional DMS technology.

SGS Minerals Services has participated in the CAMIRO Deep Penetrating Geochemistry research program since its inception. The results from the program in the Abitibi, some of which are just being released from confidentiality, demonstrate the clarity that the use of the MMI™ geochemical analytical technique bring in deep overburden areas. The CAMIRO program has clearly shown that the MMI™ technique can be used to detect low-level geochemical anomalies over mineralization in areas with thick overburden. Sampling the A soil horizon as opposed to the more traditional B horizon has proven to be a key component to the success of the program. Further evidence of the effectiveness of the MMI™ technique was the commercial success in 2002 when gold mineralization was found in the Assean Lake area, Manitoba. Contact us for more information about the MMI™ technique and relevant case studies.

Data handling systems are integral to our business. Our relationship with CCLAS™ has seen the development and commissioning of the CCLAS EL™ LIMS in our Peruvian laboratory. The CCLAS EL™ LIMS will be rolled out across our laboratory network in 2003 and 2004. The CCLAS EL™ LIMS is allowing the Group to develop web based reporting and data manipulation systems that should be available to clients in late 2003.

The Group has four laboratories around the world that have achieved ISO/IEC 17025 accreditation. Our laboratories in Don Mills and Lakefield in Canada, Johannesburg in South Africa and Perth in Australia have accredited scopes that cover the complete range of testing services required by exploration geologists. One of the key requirements of ISO/IEC 17025 accreditation is that

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measurement uncertainty must be determined for each method and sample matrix and the laboratory must demonstrate a rigorous quality management system, coupled with demonstrated competency with the analytes and samples usually processed. SGS Minerals Services intends to continue its leadership in the provision of high quality, bankable exploration data, by accrediting more of its laboratory network to the ISO/IEC 17025 standard over the next 18 months. The Group operates an Internal Round Robin (IRR) program that has a scope, frequency and reporting excellence not found anywhere else in the world. The IRR program allows client laboratories to participate and in selective situations makes the results of the IRR available to clients. The SGS Minerals Services IRR program truly challenges our laboratory network and makes them better!

SGS Minerals Services has positioned itself to be able to provide geologists with a complete exploration service. From ultratrace analytical and mineralogical services to the largest network of on-site purpose built analytical laboratories, SGS Minerals Services can help you with your total requirement. Please don't hesitate to contact us to explore how SGS Minerals Services can become your global exploration partner.

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AEG Presidential Address

Steve Amor

I would like to thank Chris Benn, in particular, for his efforts in assembling in this issue a provocative (and I mean that as a compliment!) and interesting array of articles on the analytical aspects of our profession. The deliberations of the Exploration Technology Workshop in Denver last year, in particular the comments of Bill Coker, indicate that the task remains to pass on this information to our co-explorationists, some of whom lack knowledge of current geochemical practice. By virtue of its reader-friendly format, **EXPLORE** can play a significant role in the accomplishment of this task.

A large, vigorous and vocal membership in our association can also promote the intelligent application of geochemistry and its integration into effective exploration programs. I would like to draw your attention to, and commend, the efforts of Robert Jackson and his New Membership Committee to reverse the decline in membership numbers over the last couple of years. I am sure they would welcome members' input into this subject; and since **EXPLORE**'s readership extends far beyond the few hundred members of AEG, those of nonmembers too as to what it would take for them to join or rejoin us.

The time lag between the deadline for submission of this piece (about one hour away, as I compose these lines!) and the appearance of the **EXPLORE** in which it appears, means that I can anticipate a number of impending decisions and announcements without being able to say exactly what they will be. On this occasion, they will include a new Web page for the Dublin Symposium, including facilities for online registration. Early registration will provide you with financial advantage, as well as facilitating the LOC in their planning activities.

Two other features that I anticipate are web pages concerning the Canadian Council of Professional Geologists, and how our association can best promote the interests of its members and their discipline, along with a new draft Code of Practice for the holding of Symposia that we sponsor (and an invitation for comment). There will also have been a Council meeting, the first such under my Presidency, on March 26th.

It's only fair that I report on the outcome of a previously-unresolved issue from my piece in the last **EXPLORE**: the appointment of a new Webmaster, Rodrigo Vázquez. The tighter appearance of both the homepage and the members' gateway are thanks to his web design skills.

The debate on our association's name is still very much open, possibly contrary to the impression gained by some members. Up until now, we have agreed in principle that a name change is justified, and opinions as to what the new name should be have been expressed, without a sufficient majority for us to go ahead with the change. It is my hope and intention that a formal motion in favor of one name of the other be proposed, at least, before my mandate as President is more than half a year old. We have extended the deadline for the closing of the comments page and I invite anyone who has an opinion of this subject (including the title of the biennial Symposia) to post them on the website, or communicate them to the individual Council members or the Editors of **EXPLORE.**

Steve Amor
President, Association of Exploration Geochemists, 2003

Geochemical Laboratory Updates...

AMDEL

Amdel is a leader in the provision of quality and innovative laboratory and technical services to a broad range of industry sectors including minerals, petroleum, environmental, food, beverage, pharmaceutical and agriculture. With over 40 years of history Amdel is the leading name in its business field and recognised for quality and innovation. Today Amdel employs more than 500 people and turns over in excess of $40 million annually. Our aim is to be

- the customer's first choice
- an innovative organization that exceeds customer expectations
- a highly successful global laboratory and technical services company.

In March of 2002 Amdel became a wholly owned subsidiary of The Gribbles Group. The group is committed to maintaining a strong focus on the traditional resources-based markets, including the extractive industries such as gold, base metals, iron ore, mineral sands, industrial minerals, and oil and gas, while our penetration into other markets, such as food, agriculture, environmental, pharmaceuticals, general manufacturing and the steel industry, will be further developed and expanded.

Amdel offers a wide choice of services to the mineral industry based on the technical excellence of its staff and modern laboratory equipment and procedures adapted to our business. Some of the services Amdel offers include atomic absorption spectrometry (AAS), inductively coupled plasma optical emission spectrometry (ICP OES) and mass spectrometry (ICP MS), x-ray fluorescence (XRF), x-ray diffraction (XRD), fire assay, sulphur by high frequency induction furnace and colorimetric, selective ion electrode, gravimetric, volumetric analysis, petrography and mineragraphy preparation and identification and metallurgical test work designed to develop new processing routes or optimize existing routes. These analytical tools and procedures are developed and optimised by Amdel's dedicated chemists, mineralogists and metallurgists.

But it is not enough simply to be the best technically Amdel recoginises that information must flow seamlessly back to the client in a timely fashion. Amdel has committed to the next generation of laboratory information management system by Comlabs, e-CCLAS, with all laboratories to be connected by the end of this year.

Amdel's head office is in Melbourne, Victoria with laboratories in Adelaide and Whyalla in South Australia, Perth and Kalgoorlie in Western Australia, Sydney, Broken Hill, Newcastle and Orange in New South Wales, Melbourne in Victoria, Mount Isa in Queensland and Auckland and Macraes in New Zealand.

Anthony Bertridge
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ATOKA GEOCHEMICAL SERVICES CORP.

2002 saw Atoka Geochemical Services Corp. and Atoka Coal Labs continue to expand their services as a global leader in petroleum surface geochemistry methods and coal bed methane. Atoka, in its association with TIPM of Calgary, Alberta, provides clients with comprehensive exploration and development services for petroleum and coal bed methane — worldwide.

Despite the downturn in worldwide exploration for petroleum in the face of increasing product prices, Atoka Geochemical Services Corp. has been involved in a significant conventional gas discovery in the San Joaquin Basin by Tri-Valley Corporation. Atoka continues to analyze samples from several basins in Canada and the United States utilizing its proprietary iodine method. This technology detects the presence of microseepage in the soil caused by petroleum migrating upward from depth. Atoka has branched out and added other secondary methods such as measurements of soil gas, partial leach extraction, enzyme leach, pH, Eh and conductivity at our clients request. Atoka advocates an integrated approach to exploration for finding new petroleum reserves by combining surface geochemistry, geology, geophysics and engineering parameters.

Atoka Coal Labs continues to expand in to new areas where it is doing coal desorption\adsorption work. Atoka is the major laboratory involved in coal desorption\adsorption work in Eastern Kansas and Missouri and in the Illinois Basin. Atoka is involved in several publications that are in press or that are being reviewed on coal bed methane gas resource in the Western Interior and Illinois basins. Atoka is also the lead lab analyzing the coals for gas in the Onakawana Basin in Ontario for Admiral Bay Company of Vancouver and James Bay Company of Toronto. Atoka continues to help its clients evaluate the gas potential of several large acreage blocks of coals in Wyoming, Kansas, Missouri, Illinois, Alberta, Utah and Ontario. Atoka has also recently patented, with Bowler Petrophysics of Denver a proprietary method of calculating gas reserves in coal from mechanical logs. In addition, Atoka has been able to develop methods to correlate maceral composition to permeability data and gas resource potential utilizing its large database.

Atoka Geochemical Services and Atoka Coal Labs has positioned itself to be able to provide geologists, geophysicists and engineers with a complete exploration service for petroleum and coal-bed methane. Please do not hesitate to contact us or visit our websites at www.atoka.com and www.atokacoallabs.com to explore how Atoka Geochemical Services Corp and Atoka Coal Labs, Corp. can become your global exploration partner.

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FOCUS: Certified Reference Material Discussion
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Reference Materials

Reference materials (RMs) are the backbone of every laboratory's efforts at quality control and quality assurance. Ideally, all of the RMs used by the laboratory will be certified materials (CRMs) whose reference value uncertainties will be small in comparison to the accuracy and precision requirements for end use of the laboratory's data. In reality, this has rarely been the case for geoanalysis.

Assayed ores and concentrates have been available as RMs from the very beginning; the National Bureau of Standards issued an argillaceous limestone, NBS-1, in 1906, and a zinc ore, NBS-2, shortly thereafter. But it was not until 1951 that G-1 and W-1 became available to support geoanalysis generally. The reference values were developed by classical methods for the major oxides only. Once Dc-Arc AES became prevalent in exploration and other geochemical laboratories, some trace element data were added, but the reference value uncertainties for these trace elements were very large.

Subsequently USGS developed the GXR series of RMs to support exploration geochemistry. These materials were first issued in 1975, and continue in use even today. Thirty elements were determined by six-step De-Arc- AES, while the more important elements for exploration purposes (Ag, As, Au, Ba, Be, Bi, Ca, Cd, Co, Cu, Mo, Ni, Pb, S, Zn) were also determined by a number of other methods. In particular, the AAS data were obtained after selective leaching of the samples, using common exploration techniques that are specific to mineral speciation. The critical elements in the GXR samples occur at concentrations greatly enriched over the concentrations typical of most silicate RMs.

Analytical methods have changed greatly since the GXR materials were issued. New instrumental methods have been developed that allow far more simultaneous multielement analysis than was possible then. The detection limits for many elements have been lowered, and whole new suites of elements of interest to geochemists are now being measured routinely. Additionally, the precision of measurements has greatly improved. However, better precision has not been accompanied by higher degrees of interlaboratory agreement. Thus, the reference values initially established for materials like the GXR series through round-robin certifications have not kept pace with changing measurement capabilities.

The problem is a circular one, since each advance in measurement technology permits new kinds of measurements to be made, new types of geochemical questions to be posed and answered. New techniques are introduced before the last "new" technique has been perfected, to the point that between-laboratories discrepancies are eliminated from analytical data. Results are accurate enough for most applications without necessarily becoming accurate enough for certification purposes, which aim to achieve uncertainties in reference values that are a factor of three to ten smaller than those of routine laboratory measurements.

In some cases, the initial reference values can be updated successfully as new measurements are made on existing RMs.

But care must be taken in this process. If, for example, a considerable body of the data for a given RM is collected using a leach specific to the dissolution of sulfides, it cannot be merged with data from a whole rock technique, or with data obtained using a leach specific to some other mineral species. Unfortunately, this error has appeared repeatedly in the literature over the years.

Since my own expertise lies with silicate rock RMs, I am perhaps in a poor position to identify the RMs best suited to the needs of the exploration geochemist. However, there are several CRMs that have been developed in support of environmental monitoring that might provide a suitable matrix match and an appropriately-enriched concentration level of key elements to be valuable. These include the NIST soils, SRMs 2710 and 2711, and the industrial sludge, SRM 2782, all of which have been certified for many elements important to exploration. The NIST water SRM 1643e provides the elements of interest at close to detection limit concentrations, and so is less suitable.

CANMET is a principal producer of CRMs for the mining industry. The materials are generally ores and concentrates, so that critical elements occur at much higher concentrations than they exist at in soils and stream sediments, and the CRMs will not necessarily provide a matrix match to exploration program samples. Thus, they are potentially less useful to the exploration geochemist than the GXR materials or the NIST soils and sludges might be. However, their certified values are generally known with great certainty, and the certification is documented in a way that is fully compliant with the latest International Organization of Standardization (ISO) guidance. Thus, these CRMs are of the highest metrological value to the laboratory.

There are specific instances in which it will be difficult to find appropriate RMs. For example, the halogens Cl, Br, and F are being used as indicators of buried Cu-Zn bodies. Yet reference values, especially Cl and Br, are established for far fewer of the many RMs in existence than is the case for ore metals and other pathfinders to them. Similarly, the pathfinders for disseminated Au mineralization, e.g., As, Hg, Sb, W, are not established in many RMs with uncertainties in the reference values that are appropriate to support modern measurements. Other pathfinders for which reference values are either absent or inadequate are Ag, B, Ge, Mo, Sn, and Tl.

Thus, the need exists to continue the development of RMs for exploration purposes, as well as for all other geoanalytical purposes. The effort involved in thorough RM certification, however, is very detailed and laborious, and demand always exceeds availability. Laboratories must endeavor to use existing materials to best possible advantage, while remaining aware of their limitations. Only in this way will laboratory data quality continue to improve as new methods of measurement come into use, and new approaches to exploration continue to be employed.

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…THE MORE THEY REMAIN THE SAME

There is a well-known French expression to the effect that the more things change, the more they remain the same. So it is with sample preparation and assaying, which have been investigated in depth and correctly published on, widely, for more than a hundred years. Yet the literature is more often ignored than appreciated, leading to unnecessary but horrifying losses.

Here is just one example: In February 2003 a small group in Australia expressed concern that mineable grade gold assays from samples of material being treated with adequate recovery, were far too low. A reputable laboratory – part of a major group – using a standard sample preparation protocol and a standard analytical technique, was carrying out sample preparation and assaying. Both protocol and technique were, and are, routinely checked and subjected to in-house and external standards and round-robins with no indication of any problems.

Questioning revealed that the clients did not realize that the protocol and the technique were causing the problem, nor that it was their responsibility to call for different or additional procedures, and not the laboratory's responsibility to flag the problems.

In a nutshell, there was enough gold as free and composite particles to cause a loss of about 20% of value in preparation, by gravity segregation and the scooping of sub-samples. A further 30% to 40% of the remaining gold would have been left soaked into the top of each cupel as there was enough tellurium in the ore to "wet" the surface of each prill in cupellation. Both problems were well recognised by 1915, yet the clients and their associates were unaware that, combined, these would usually result in assays reported at 50% of the true tenor of their samples. The laboratory, of course, had no idea that a problem existed. Hellman (1999), discussed the first part of the problem on p. 6, while Lenahan and Murray-Smith,(1986), mention the second on p. 228. Both are discussed at some length in Brooks,(1999). The clients were unaware of any of those publications.

The world's major mining companies, too, have been falling into the same sorts of traps for years. This has left the gate open on a number of occasions for scams such as Busang, highlighting the need for all exploration and mining people to be aware of the history of understanding such situations.

From the 1890s to the early 1920s many people were concerned with sampling, sample preparation, sub-sampling and assaying. Reading of section 29 of Peele, Volume II (my copy is a 1941 edition) should be part of all exploration geoscientists' training. Gradually, since that time, interest at an executive level and at senior technical levels has waned, with occasional spikes caused by defalcations due to in part, or wholly, to poor and/or fraudulent practice. One can only guess that at all other times these areas of activities are regarded as low level, trivial or non-professional activities by too many educators and managements, very few of whom have learned the tricks and traps of these trades.

Fortunately there is a resurgence of interest and a renewal of serious teaching in these fields, lead by such well-known figures as Francis Pitard, Pierre Gy, and Dominique François-Bongarçon as individuals and bodies such as the Association of Exploration Geochemists, the Australian Institute of Geoscientists, the Australasian Institute of Mining and Metallurgy and the Canadian Institute of Mining, Metallurgy and Petroleum. Despite their valiant efforts, many of the old verities have yet to be re-discovered by modern youth.

Pitard's Chapter 26 of his Volume II, published in 1989, is a four-page effort to change management thinking which should be taken to heart by all in this field. Peele's Mining Engineers' Handbook, first published in 1918, is still irreplaceable as a reference for geologists, mining engineers and primary metallurgists working in these fields, yet neither author's works are common in university courses for explorationists or mine company management. Hoffman and Dunn continue to sound warnings, in their 2002 paper (q.v.).

Perhaps this parlous state of affairs has arisen because the huge financial costs of poor practices are commonly not revealed to those executives responsible, much less to the public (or shareholders). In this field some more recent examples should suffice to illustrate the importance of the point.

During 1999 a porphyry copper deposit, relatively newly in production, had 11% of its near-surface in-pit ore reserves deleted by identification in an infill drilling campaign. No statement was issued about the sample preparation, and analytical techniques used nor about the major part of the problem: pattern drilling of too few metres, parallel to a dominant mineralized fracture pattern. The mineralization, however, was of a type identified, inter alia, by Hoffman (1992) p. 302, as likely to cause problems if analysed by accepted fire assay followed by AA or ICP-AES finish for gold, which is an important element in this mine's production.

Investigation of another porphyry copper mine's sample preparation system during 2000 A.D. revealed that just between comminution and sub-sampling two major potential error sources existed. The ore was chiefly chalcocite, which has an accepted S.G. of 5.5-5.8 (Anthony et al., 1990 vol. I, p. 88). Grades varied from 0.5% by weight to over 1% by weight, but, being a secondary ore, the S.G. of the host rock was about 2 or even a little less, therefore the percentage by volume of chalcocite was of the order of 0.25% to over 0.5%. It was comminuted to nominally 100% minus 150-mesh before being subsampled. Chalcocite is relatively soft and quite brittle, resulting in a minus 150-mesh pulp containing a disproportionate amount of chalcocite in its finer grain sized particles. Metallurgical studies of chalcocite concentrates show clearly that grades increase dramatically as grain sizes of portions selected for assay fall. A good example is given in Duyvesteyn (1995), who showed (p. 35) that three size fractions of concentrate from minus-100 mesh to plus-200 mesh, from minus-200 mesh to plus-400 mesh and minus-400 mesh assayed 28% Cu for the coarsest fraction but 45% Cu for the finest fraction.

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The subsampling consisted of mat rolling the pulp from a puck and ring mill grinder and then scooping 250-300g from the mat. The rolling enhanced the gravity segregation caused by dumping the pulp onto the mat. So-called homogenisation techniques were used to "show" that the protocol was valid. Many so-called homogenisation procedures applied to such material are psychologically good but physically self-defeating because inappropriate sub-sampling is then carried out.

When such a sample is mat rolled the finest fraction behaves in the same manner as fine gold in a pulp – it goes straight to the top surface of the mat (Brooks, 1999, p. 6). Even a slight jolt will cause significant segregation of such material. When a sample is scooped from this kind of mat-rolled pile, the highest grade fraction remains on the mat surface. The leading edge of any sample scoop has a thickness far too great to catch a thin layer of even minus-200 mesh particles, much less the minus-400 mesh particles coating the mat surface (and occupying probably less than 0.1% of the bulk of the sample by volume). So the scooped sample cannot be other than well below the average copper grade of a comminuted pile of chalcocite-bearing material.

This particular mine's practice relating to many of its ore grade samples then involved significant vibration in transport of the 250g Kraft sub-sample packets before they were opened for extraction of small aliquots for copper analysis. Naturally the remaining fine grained chalcocite particles were jiggled to the base or bottom of each packet in transport, and yet aliquots were scooped from the tops of the packets for analysis!

The net result was significant under-reporting of grade resulting in miss-classification of much marginal ore as waste and mill grade ore as leach material. This, of course, raised the waste-to-ore ratio in addition to other obvious problems of mill and leach recoveries being over reported because input rock grades were being consistently under-reported. This took attention away from improving recoveries.

The cash flow loss was estimated by staff as many millions of dollars per annum, after they had been briefed on the realities of the weakness of the process. Similar situations, with similar results, are almost as common in operating gold mines as they are in secondary copper sulphide mines. A very clear example of this is given by Helman in his 1999 paper in which, on p. 6, he states inter alia: "Scoops taken from the top of the mound of pulp in the pulverizer (no tipping it out, much less mat-rolling it) for conventional fire assaying having an average 30% less than gold assays determined by either cyanidation of the whole pulp or by screen fire assaying of a large sample". As he continues: "The low bias of the fire assays, however, has not arisen due to their low (50 grams) sample weights, per se, compared to the larger weights employed by the other techniques but rather because the samples were intrinsically biased due to segregation of gold particles in the pulveriser."

This problem was also noted by this writer during inspection of gold mines in Western Australia during the 1980s when the significant down-grading of head grade values resulted in such unbelievably overstated mill recoveries that the writer immediately mounted investigation of the head grade sample-comminution protocols. The cost of accepting poor metallurgical recoveries, reported as good ones, was not on management's horizon. Sample preparation errors had deluded the metallurgists into not correcting recovery problems in their plants.

Tacit recognition of the problems outlined in the first few paragraphs of this article was given to the writer in 1955, during a visit underground to the Great Boulder mine at Kalgoorlie, Western Australia where telluride-rich ores were being mined. Staff advised that any exploratory-drill intercept of half mineable grade-width or more had to be drifted onto, by management directive, and that even lower grade-widths often warranted this action, to be followed by bulk sampling and metallurgical testing. Of course, sample preparation and assaying of telluride ores was only a minor part of that equation then. Too often discrepancies between reserve grades, mill heads and recoveries have given rise to conflict between geologists and mining engineers when the problems belong elsewhere.

Exploration samples, grossly under-reported because of similar characteristics, quite often have never been followed up. As PGE samples are usually comminuted to 95% minus-200 mesh before sub-sampling (Hoffman and Dunn, p. cit. p. 2) the situation with them is even more acute.

The additional expense incurred in modifying a sample preparation protocol from standard should always be considered when a target element is contained in a mineral or minerals of SG greatly contrasting with that of the matrix. Not only gold, but also primary uranium, secondary-sulphide copper, lead and the PGM's for example, always mandate such a modification.

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Assignment of 'mean' or 'recommended' values for standard materials

Certified reference materials (CRM) supplied by Government-funded projects such as the National Institute of Standards and Technology (NIST) and Canadian Certified Reference Material Project (CCRMP) are intended for multiple purposes, including use as primary standards to test new analytical methods or laboratories as well as for insertion as controls in day-to-day analytical quality assurance. However, because of their relatively high cost, it has become accepted practice in large surveys conducted by exploration companies to use less expensive control samples or secondary standards provided in bulk by commercial companies such as Ore Research and Exploration (http://www.ore.com.au) of Australia. They provide elemental values for such samples by first carrying out a round-robin amongst geochemical laboratories worldwide. For certain elements such as gold, a method (e.g. based on fire assay or aqua regia decomposition) will be requested by the initiator of the round-robin but for base metals the method is often left to the discretion of the lab manager. Ore Research and Exploration distinguishes between results obtained using partial digestions such as perchloric, aqua regia, or hydrochloric /nitric/perchloric acids and those by 'total' methods such as those based on the hydrofluoric/nitric/perchloric/hydrochloric digestion (commonly known as the 'four-acid' digest), fusion, XRF or INAA. However, some commercial suppliers of these standard samples do not, and instead pool all data received in the round-robin, regardless of digestion. For example, one compilation (pers. comm., Steve Cook, 2002) of results for a low-level ultramafic control sample lists 44 data points from various laboratories worldwide. The data are not grouped by method, or into 'partial' and 'total' categories of analysis. A frequency graph of these results for this standard sample is shown in Figure 1. A mean of 165 ppm Ni with a standard deviation of 43 ppm is provided in the summary statistics of the company report and is based on all the data except for the three results in the 300-330 ppm range which are much higher than the
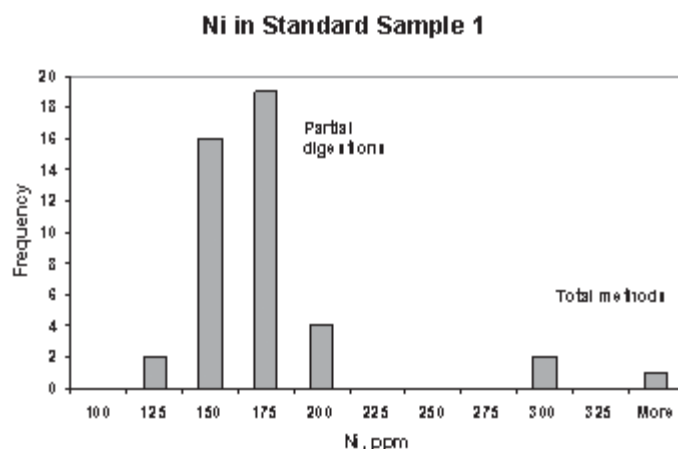
Figure 1: Round-robin data for Ni in standard sample 1 (pers. comm., Steve Cook, 2002)

others and therefore were discarded. However, it is probable that these higher results were obtained by an HF-based acid digestion or a direct total method and are much closer to the 'truth' than the bulk of the analyses which are likely based on aqua regia or an HNO_3/$HClO_4$ type of digestion (without HF). Results for this control sample obtained by Acme Laboratories (Vancouver), and the Geological Survey of Canada (GSC), agree well: 145-155 ppm Ni by aqua regia and 280-300 ppm by HF/$HClO_4$/HNO_3/HCl digestion. Clearly, HF is needed to release all Ni from its mineral assemblages in this sample.

Another example is afforded by Pb data in another standard sample that formed part of a recent round-robin exercise (pers. comm., John Gravel, 2002). The data from a total of 54 laboratories are used in the frequency diagram in Figure 2. Two populations are again evident, one centered on *ca*. 45 ppm Pb and the other on *ca*. 85 ppm. Two of these
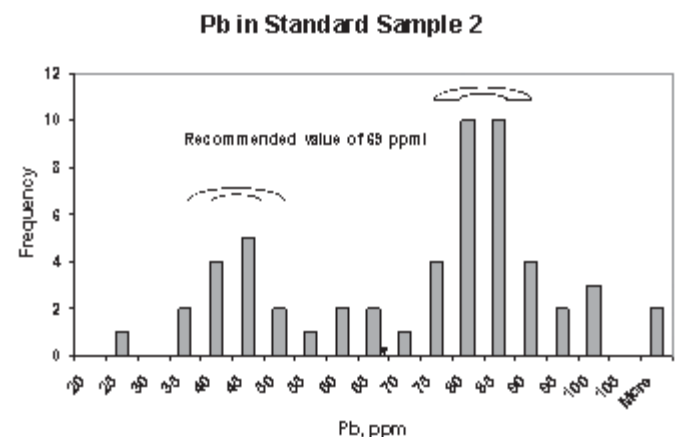
Figure 2: Round-robin data for Pb in standard sample 2 (pers. comm., John Gravel, 2002)

results were provided by Acme Laboratories. Their Chilean laboratory used an HF-based 'four acid' digestion and reported a value of 80 ppm Pb whereas the Vancouver laboratory used aqua regia and reported 43 ppm Pb, two values coincident with the centres of the two population means, in Figure 2. The mean value reported by the company is 69 ppm Pb, between the two populations and is not the true value. It neither represents a total value, nor one expected by an aqua regia digestion.

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Laboratories participate in these round-robins free of charge and use them as a proficiency test. However, some laboratories are no longer participating as they may be deemed 'inaccurate' or their performance 'unacceptable' because their data have been produced by a total method whereas the majority used a partial digestion (or vice versa). This problem of mixing data produced by partial and total extractions has been encountered by proficiency testing programs such as that run by the 'Canadian Association for Environmental Analytical Laboratories' (CAEAL) and the 'Proficiency Testing Program of Mineral Analysis Laboratories' (PTP-MAL) of the CCRMP. The 2002 report by PTP-MAL discusses the differences in results for Co and Ni for one of the test samples and identifies this as being due to laboratories using aqua regia and HF-based digestions. The report calls for suggestions to remedy this situation: one can choose either of two approaches to do so. Firstly, the testing program can stipulate the *type* of result required: total element determination or partial (and if partial, the specific decomposition). Currently, for example under the PTP-MAL, a laboratory is asked to use "one method of its choice in a manner identical, to the greatest extent possible, to that applied to client samples". Clearly, for many geochemistry laboratories, this method would be based on aqua regia decomposition rather than on HF/$HClO_4$/HNO_3. Secondly, the laboratory can be free to implement their commonly used method but results would then be divided into categories that make sense with respect to their chemistry.

The publications by John Lynch (1990, 1996, 1999) of data for his 12 sediment and till CRMs (LKSD 1-4, STSD 1-4, TILL 1-4, marketed under the CCRMP) provide an example of categorising results. He divided data into three groups (Lynch, 1999): 'total', obtained by variants on the HF-based digestion; 'concentrated HCl-concentrated HNO_3' digestion; and 'dilute HCl-dilute HNO_3'. It is interesting that, given all the different conditions employed within the latter two groups, only results for P and Ba differed. However, there were many differences in results between the aqua regia and HF-based data-sets, as demonstrated in an earlier Explore article (Hall, 1999). For example, across the 12 CRMs, the elements Al, Ba, Ca, Cr, Sr, Ti and V showed markedly low recoveries by the aqua regia decomposition, as would be expected from their mineralogy. To a lesser degree, distinctly low results were found also for Mg, Fe, Mn, As, Pb (in tills only) and Sb (in stream sediments). Recoveries by aqua regia across the different sample types were generally better than 75% for Ag, Cd, Co, Cu, Mo, Ni, P and Zn. It should also be remembered that an HF/$HClO_4$/HNO_3/HCl digestion is not total for refractory minerals, so results for elements such as Cr, Ba, Zr, Hf and Ta will be lower than those by XRF or INAA. Therefore, it would be unwise to include HF-based data with results by these total methods for a significant number of elements.

In summary, it is highly recommended that (1) the suppliers of control samples provide adequate information on the methods used to obtain each data point listed and (2) the purchasers of control samples carefully examine these data and select those pertinent to the purchasers' intended use of the samples. This will not only avoid wasted time and expenditures in tracing an analytical laboratory's supposed error when they are not in agreement with a mean value assumed to be true, but will also offer a solid base upon which to judge a laboratory's performance in a round-robin. The current practice of pooling data across partial and total methods does not allow for accurate analysis of a laboratory's performance and generates a meaningless 'mean' value with an erroneously high standard deviation.

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THE ROUND-ROBIN AS A MODEL OF A MEASUREMENT SYSTEM ANALYSIS FOR ASSAYS

The objective of mineral resource estimation is to provide mineral inventories that are both accurate and precise. The quality of such estimates ultimately based on the measurement systems used in the estimates, of which assays are probably the most important and complex. From a Six Sigma perspective, any acceptable measurement system must satisfy five criteria: resolution, linearity, stability, accuracy, and precision. A round-robin survey provides a convenient model for the analysis of precision using the Gage R&R procedure. This method allows the variance of assaying to be split into its individual components and provides an estimate of the proportion of the total variance of the sampling and assaying process consumed by the

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assaying itself and how well it performs against the tolerances specified by the customer. The round-robin data set for the El Morro project in Chile is used as an example of the method.

Introduction

Success or failure in mining investments is dependent on many factors but one of the most important is the reliability of mineral resource estimates. Such estimates are, in turn, dependant on many factors, one of the most important being the quality of assays.

Much work has been published that describes how to obtain quality assays which are defined as analyses that are both accurate and precise (see Vallée and Sinclair, 1998 and references therein). Various texts are available that explain how to collect, prepare, and analyze samples in order to avoid bias (high accuracy) and keep variance within reasonable limits (high precision) (Gy, 1982). This communication presents assays as viewed from the perspective of the Six Sigma quality system and what this general philosophy can tell us about mineral resource estimates when assays are considered as a measurement system.

A round-robin survey of pulp standards provides an excellent model to evaluate assaying as a measurement system. This is because a formal measurement system of analysis requires parts (the individual pulp standards), operators (the participating laboratories) and multiple measurements (multiple replicates of the standards) and provides information on repeatability and reproducibility of the measurement system. This information in turn allows us to determine whether assays are acceptable for the stated purpose of measuring a mineral resource.

Six Sigma and measurement systems

The Six Sigma philosophy is all about processes and outputs but mostly about the needs of customers. Organizations prosper by creating and maintaining processes that provide products and services to customers. Processes however do not produce uniform outputs and as a result a certain proportion of the outputs typically exceed the limits that are acceptable to the customer resulting in defective products or services. A customer-centric definition of defects leads naturally to product improvement in the direction of enhanced customer satisfaction and therefore customer preference (Pande et. al, 2000). Moreover, reduction in the proportion of defective outputs leads to reduced cost since re-work and scrapped work are reduced, all the while increasing production for the same quantities of inputs. The motto of Six Sigma "do it right the first time" is a simple idea that has helped numerous organizations improve their bottom line.

A process output will often have a normal distribution whose parameters will be a mean and a standard deviation. The focus of Six Sigma is to obtain process outputs that are within the tolerances defined by the customer. In a process that is measured by a continuous variable, such as an assay

for example, the specification would be the deviation from the true value of the assay. The performance of the process is measured as follows:

$$Z = (Spec - Mean) / Std \qquad (1)$$

In practice this reads as the number of standard deviations between the mean of the process and the customer's specification limit (the value beyond which the output is considered unacceptable). For a process operating under short-term[1] conditions the ultimate objective is often to operate at a Z of 6 or more thus the origin of the term Six Sigma. With processes that have a discrete output, performance is measured in defects per million opportunities (DPMO) and this translates to 3.4 defects per million for a Six Sigma process.

For any process the objective is to identify a measure of output that is important to the customer, obtain from the customer the acceptable tolerance of the process and, with measurements of the output, determine the present performance level of the process. This is the starting point for improving the process as this provides the way of comparing improvements to the baseline level.

The observed output of any process is equal to the output of the process plus the error in the measurement. It is the measuring device (Kiemele et. al, 1997), more generally known as the measurement system, that causes this accuracy error or bias. Likewise, the observed variation of any process is the sum of the variance of the process itself plus the variance of the measurement system. It can be shown therefore that reductions in process variance will eventually be limited by the quality of the measurement system. As a consequence, a poor measurement system will not permit a process to reach high sigma values. For this reason all Six Sigma projects need to undertake one or more measurement system analyses to ensure that the performance objective (i.e. the target Z or DPMO) can in fact be met using the proposed measurement system.

A measurement system analysis comprises five components, namely, resolution, linearity, stability, accuracy, and precision (Kiemele et. al, 1997). Resolution is the number of individual subdivisions or values that the measurement system can adopt over the range of variation of the process. This number is subject to the "10 bucket rule" in that the size of the increment of the measurement system should be no more than one-tenth the tolerance (the range of acceptable values) set by the customer. For example, a weight scale that is subdivided in tenths of a tonne is suitable to measure differences in truck weights that are at least a tonne. However it cannot be used to measure differences in the order of 100 Kg. For this a gauge

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1. Conditions are considered short term (Zst) when all conditions that affect the output can be considered constant. This is generally the case over short intervals of time when the same operator uses the same equipment. On average a Z shift of 1.5 is found to occur for many processes over the long-term when conditions cannot be considered constant (Noranda, 2001). Hence a process running at a Zst of 6.0 would be expected to run at Zlt = 4.5 over the long-term. By convention, defects per million opportunity (DPMO's) always translate to an equivalent Zlt and vice versa.

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(3) sample preparation, (4) chemical analysis, and (5) data presentation and interpretation. This series of steps is both sequential and iterative. Overall success of the programs is dependent upon the strength of the weakest link in this sequence of activities.

The fundamental approach to exploration geochemistry is based upon a few "simple" assumptions or premises: (1) geological materials can be considered to be composite in nature, and (2) it is possible to recognize, isolate and enhance the individual "signals" from various components of the sample. The weakest link concept controls overall success. Understanding of local geochemical dispersion processes is necessary to identify and sample appropriate geological materials that may reflect mineralization. Physical segregation during sample preparation, selective chemical extraction during analytical measurement, and geochemical signatures revealed via multi-element analysis and data interpretation, together provide the bases for recognizing sample components related to dispersion processes associated with mineralization. The basic questions to be addressed by survey results are: (1) is there an imprint of a mineralization process; and (2) for the subset of "anomalous" samples, are there any trends in the data that vector to the causative mineralization source? While the exploration geochemistry survey process is straightforward, its execution, involving all the aspects described above, can be challenging. Examples of these are provided in all the presentations that follow.

Concerning data analysis and interpretation, Graham noted that the wealth of high quality, low cost, multi-element data available today present both opportunity and challenge. It is essential to maintain connection between the geological environment being explored and the computer-based tools intended to better portray the data. Review of survey quality control, single element data assessment and correlation analysis remain essential first steps in data evaluation. The primary issue is whether or not an individual sample represents an anomalous sample related to mineralization or a background sample related to some other cause. Techniques such as discriminant analysis and cluster analysis may be useful aids in sample classification. Data analysis techniques such as principal components or factor analysis may be useful in apportioning these mixed geochemical signals into their component parts, after which classification approaches can be employed to identify samples related to mineralization. Plotting of data as maps or cross sections, with contouring, coloring and shading are traditional means of both illuminating and masking relationships in the data. Geographic Information Systems (GIS) and evolving interpretive and 3D visualization techniques have considerable potential to aid in data interpretation through enhancement of both magnitude and spatial properties of data. A caution to these data evaluation methods is appropriate: the old saying, "garbage in – garbage out" holds for these approaches. Given the increasing sophistication of all the components of exploration geochemistry, and exploration in general, a team approach seems essential.

Isn't that integration? *(L. Graham Closs, Colorado School of Mines, lcloss@mines.edu)*

Quality Control in geochemical program implementation was discussed by Lynda Bloom. In order to make sound exploration decisions, reliable survey results are essential. Without quality information we risk making the two cardinal mistakes, prematurely abandoning a truly anomalous area, or wasting time and resources pursuing a "false anomaly". Lynda focused on potential errors associated with the chemical analysis component of the overall exploration process – sample handling, preparation and laboratory procedures.

Quality control (QC) is important as a means of establishing the quality of our data, and, more recently, there are regulatory requirements. Lynda recommended approaching the design and implementation of a QC program scientifically with clear objectives and outcomes. She detailed the adaptation of the Six Sigma quality system to sampling and analytical procedures (Define, Measure, Analyze, Improve, Control) in order to identify cost effective procedures for successful programs. She provided coverage of the concepts underlying QC, illustrated with practical examples (Figure 1). *(Lynda Bloom, Analytical Solutions Ltd, asl@idirect.com)*
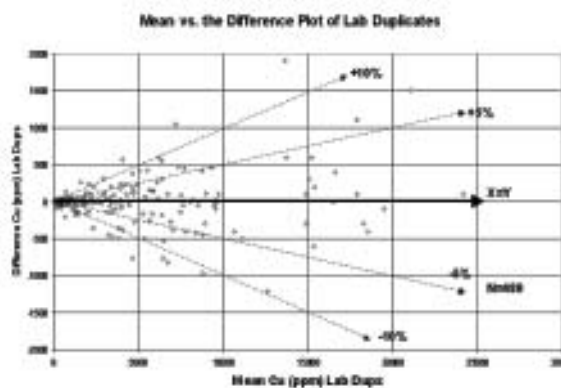
Figure 1: Measurement of QC duplicate analyses by comparing the difference of measurements to mean concentration.

Landscape Geochemistry in geochemical surveys, discussed by David Kelley, considers the totality of the surface environment. The earth contains a complex variety of landscape geomorphology and chemistry reflecting changing climatic conditions over time. Concurrent with global plate tectonics, a complex and extremely varied set of landscapes occur globally, through which we attempt to interpret geochemical data. Dave emphasized that understanding the landscape is essential to (1) understand geochemical dispersion processes, (2) design effective geochemical surveys, and (3) provide for a meaningful interpretation of both regional and follow-up geochemical surveys.

Orientations surveys help to increase our knowledge about the region and ensure that the correct geochemical approach is applied. Through a series of illustrations, Dave reviewed the variety of physical and chemical variations encountered in the global landscape. Several examples were provided that demonstrated the value of incorporating

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landscape mapping into the geochemical data interpretation (Figure 2 - see page 1). *(David Kelley, WMC Exploration Ltd, Dave.Kelley@wmc.com)*

Selective Extraction Geochemistry was reviewed by Mary Doherty. These extractions are partial analysis of a geochemical sample to selectively dissolve specific component(s) of the sample. Selective extractions may be used in exploration in covered areas (i.e. gravel, till, sand, lateritic residuum), for several purposes: (1) to isolate a particular fraction of the sample (i.e.. carbonate, Mn oxide, Fe oxide, Al oxide, clay, organic oxide films); (2) to measure chemically transported elements rather than mechanically transported sample components; or (3) to separate a later chemical signature from composition of the parent sample material. Application of selective extractions has been renewed in recent years by the commercial laboratory implementation of ICP-MS instrumentation which provides part per billion concentrations levels for a wide range of elements, allowing for more in-depth multi-element geochemical data interpretation.

Three core messages were conveyed in Mary's talk. Firstly, a variety of selective extractions are available from which the method utilized for a given geochemical survey should be dependent upon soil type and exploration environment. As discussed by David Kelley in his landscape geochemistry presentation, each landscape environment may require a different type of selective extraction. Secondly, Mary presented case histories which document that selective extractions can be highly effective in detecting concealed mineralization in a variety of geologic settings and landscape environments (Figure 3).   Finally, examples presented demonstrated that selective extractions are sensitive to variations in soil composition, soil pH, and possible seasonal variations (Figure 4). Analytical methods are still under development and vigilant quality control is a requirement for appropriate data interpretation. With effective quality control in place, selective extraction geochemistry can provide additional insight to exploration in complex or covered terrain. *(Mary Doherty, International Geochemical Consultants, LLC, MaryEDoherty@earthlink.net)*
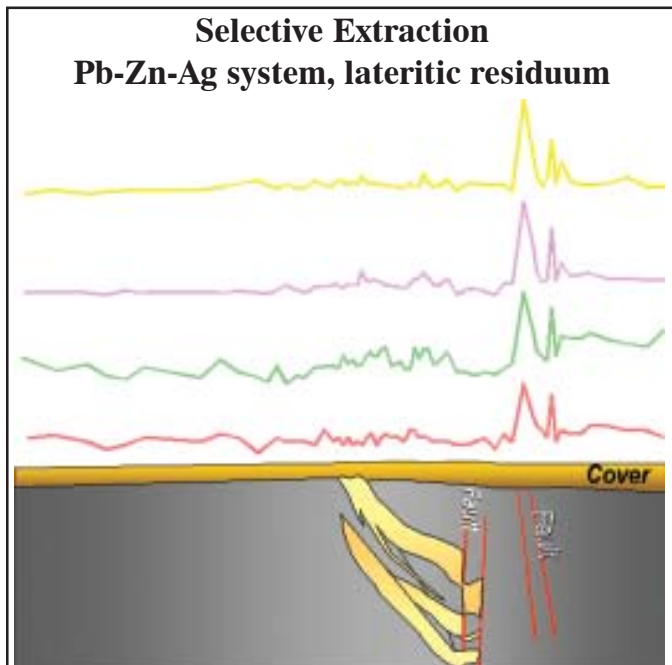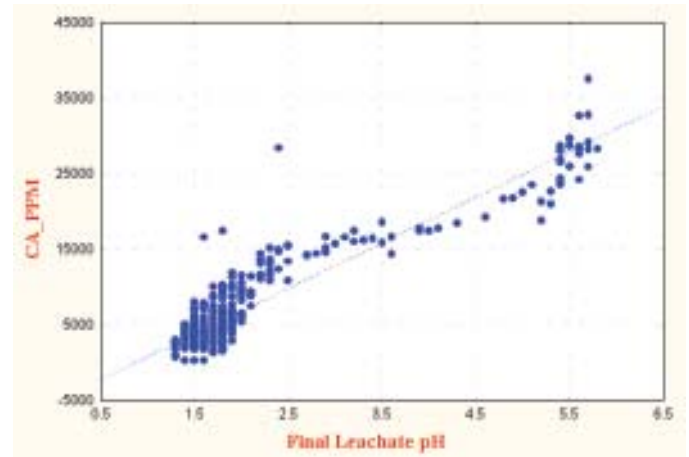
Figure 4: Plot comparing Ca concentration of soils with the leach final pH. In this environment of variable oxide-clay-caliche soils, leach effectiveness was controlled by carbonate content of the soils.

Aqueous Geochemistry principles were reviewed by Paul Taufen in his presentation: "Water: The Great Unrecognized Exploration Sampling Medium". Of all the sampling media in use in exploration, water has benefited the most from lower detection limits and an expanded suite of elements available through the development and ongoing improvement in ICP-MS analytical procedures. Three basic concepts in aqueous geochemistry were emphasized: element speciation, element adsorption, and precipitation from solution. A number of examples in minerals exploration were provided, including examples of exploration for Au and porphyry Cu deposits from western Australia (Figure 5), Southwestern U.S., Great Basin Nevada, and the Philippines.

Paul reminded us of the broad range of application of aqueous geochemistry to minerals exploration, geo-

Selective Extraction
Pb-Zn-Ag system, lateritic residuum

Figure 3. This selective extraction survey provided a multi-element response over faults associated with mineralization in an environment in which stronger digestions (aqua regia) did not reveal either the mineralization or faults.

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Golden Delicious - Dissolved Au
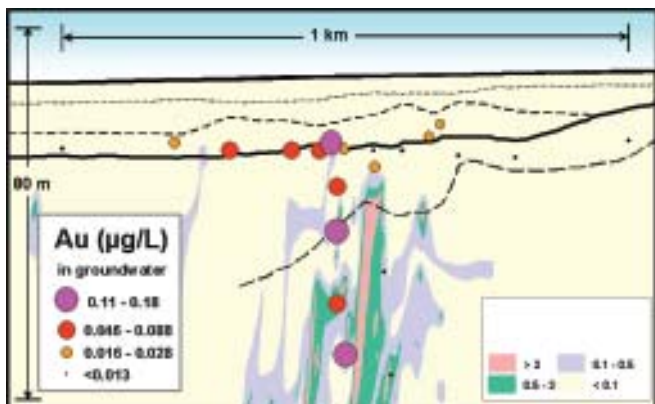
Figure 5: Surface and well water Au concentrations at the Golden Delicious deposit (CRC Leme, 2000).

environmental management, geothermal resource development, solution mining, metal recovery from brines, industrial applications (ie. hot water scaling), and management of public water supply. He further noted that hydrogeochemical minerals exploration and environmental stewardship are really the flip sides of the same coin, and that integration of these approaches can lead to effective discovery, and responsible "cradle to grave" management and sustainable development. *(Paul Taufen, Geochemistry Solutions, taufen@msn.com)*

Biogeochemistry in miner-als exploration was reviewed by Shea Clark Smith, who has applied the method in the western U.S. for over 20 years. Clark is assembling a database from knowledge of his and other's work in Au and porphyry Cu districts of the southwestern U.S. He reported that in Nevada and Arizona alone, over 300,000 vegetation samples for mineral exploration were collected from the mid-1980's to the mid-1990's (Figure 6).
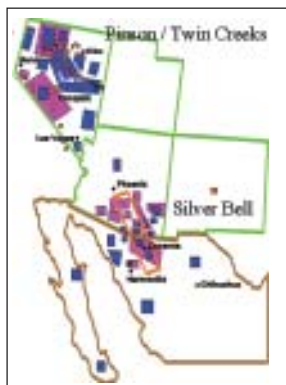
Figure 6: Biogeochemistry surveys in the western U.S.

Clark reminded us that, in so many words, biogeochemistry does find ore (Figure 7). He emphasized
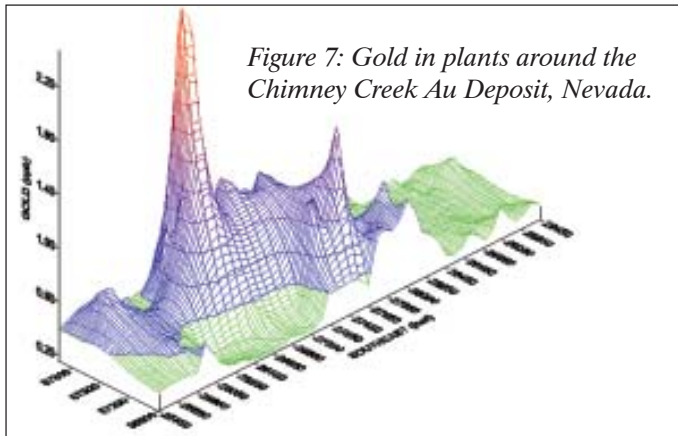
Figure 7: Gold in plants around the Chimney Creek Au Deposit, Nevada.

through numerous detailed examples, that (1) plant trace metal chemistry is linked to ground water chemistry, (2) desert shrubs and trees draw from deep ground water, (3) there is a link between trace metal uptake by plants and weakly bound ions determined by the selective extraction of soils, and (4) most metal absorption and translocation important to exploration occurs from plant roots. Sample uniformity and consistency are extremely important, whereas species effects are of subsidiary importance. *(Shea Clark Smith, Minerals Exploration and Environmental Geochemistry, SheaClarkSmith@compuserve.com)*

Soil Gas Geochemistry was reviewed by Patrick Highsmith, including discussion of various methods to collect and measure gases. Soil gases are produced by the oxidation of sulfides, oxidation of a reduced column in overburden, metabolic processes of microbes colonizing soil or mineral deposits, the off-gassing of entrained organic compounds or volatile species, and/or by micro-particulate or low vapor pressure compounds transported with another carrier phase (CO_2 or H_2O).

One can directly measure CO_2, O_2, hydrocarbons and sulfur gases in soil pore spaces. Real time analysis and follow-up of soil gas anomalies can enhance the effectiveness and efficiency of field time. Currently available static gas trapping devices include the Petrex / Gore Sorber and the Quicksilver Hg methods. Commercially available soil desorbing and leaching methods include SDP (soil desorption pyrolysis), SGH (soil gas hydrocarbons), and the GAS'm method. New analytical technologies are emerging and research continues into the microbes in the soil gas signature of mineral deposits.

Patrick provided several case histories from exploration programs (Figures 8, 9). He recommended choosing the right soil gas tool for the job and integrating with other geochemical and/or geophysical exploration methods. He

Crandon Soil Gas Profile
Line 0E - Looking West
(Geology Interpolated and Projected ~650' West. Simplified from Lambe and Rowe, 1987.)
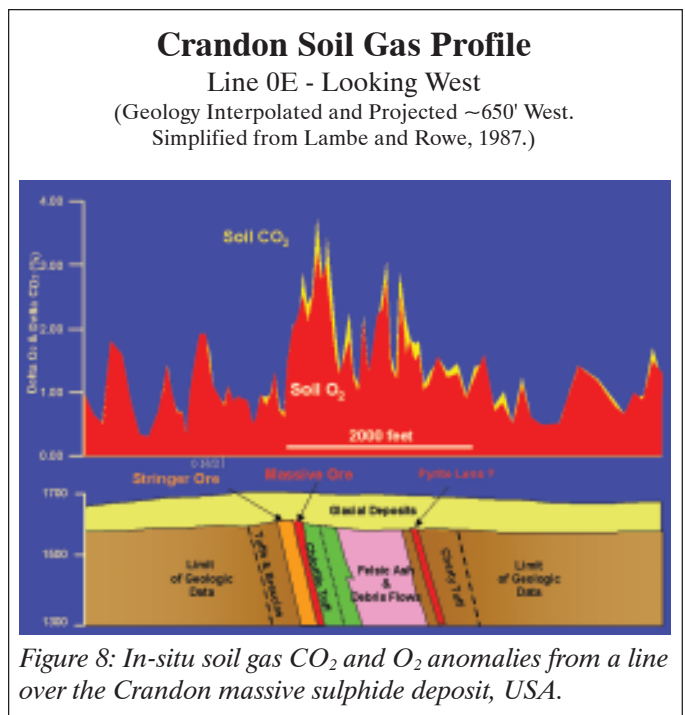
Figure 8: In-situ soil gas CO_2 and O_2 anomalies from a line over the Crandon massive sulphide deposit, USA.

Exploration Technology Workshop...
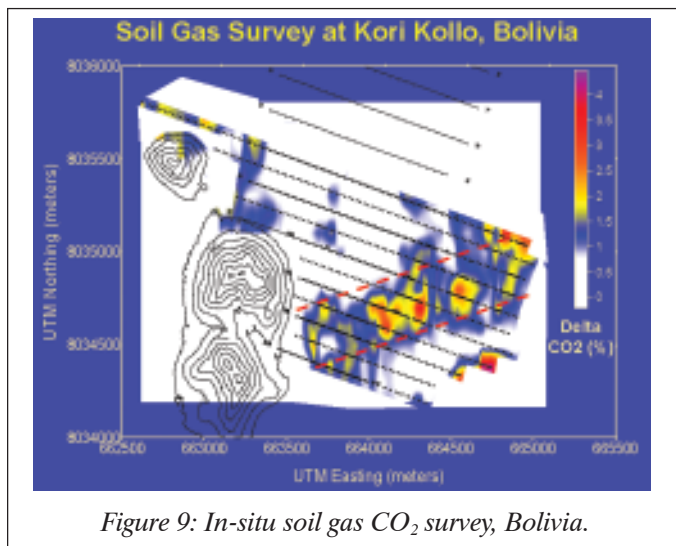
Figure 9: In-situ soil gas CO_2 survey, Bolivia.

noted, that when carefully considered and applied, soil gas geochemistry can add considerable value to exploration programs. *(Patrick Highsmith, ALS Chemex Labs, Patrick.Highsmith@alschemex.com)*

Resistate Indicator Minerals and lithogeochemistry were discussed by Steve Walters, who has specialized in the definition of 'prospective' signatures for potentially economic sources through the use of micro-analytical techniques including automated EMP and laser ablation ICP-MS. Steve reviewed the concepts behind *TerraneChron*, based on collection of regionally distributed zircons to track and interpret the geologic events and metallogenesis of a geologic terrane. The zircon analyses produce three types of data: (1) U-Pb absolute ages, (2) trace elements determination, and (3) Hf isotopes. These are used to establish terrane scale event signatures and crustal evolution histories.

Steve also reviewed potential applications of *QemSCAN* to indicator mineral identification and classification. QemSCAN is an automated SEM system developed by CSIRO based on rapid and highly automated collection of grid-based EDS spectra. The spectra are classified on the basis of user-defined data processing algorithms to produce grain maps based on chemical parameters. The system is used to process large numbers of grains prepared as polished blocks. *(Steve Walters, Advantage Geochemical Solutions, sgw@geodiscovery.com.au)*

Integrated Geochemical Exploration within a broader exploration program was provided by Chris Benn, who reviewed the discovery history of the Antapaccay porphyry Cu-Au deposit, Peru. The deposit was discovered by the BHP exploration group as the result of combined regional geologic assessment, a regional stream sediment geochemical program, regional geophysical surveys, followed by detailed ground magnetics, EM/IP, and a persistent drilling campaign. The discovery hole, hole eight, was drilled in December 1998, intersecting 226 m @ 0.95% Cu as chalcopyrite and bornite. The current total resource estimate is currently quoted as 383 Mt @ 0.85% Cu, 0.13 g/t Au.

The two deposits making up the resource are covered by fluvial-glacial cover up to 80 meters thick (Figure 10). Subsequent to the discovery, a geochemical orientation survey was carried out over the covered resources. A Mn-oxide specific selective extraction anomaly in Cu occurs over both deposits, with a stronger selective extraction Cu anomaly over the southern porphyry. By comparison, the more conventional aqua regia analysis of soils produced a strong geochemical response from mechanically dispersed Cu bearing skarn fragments in the cover materials near outcropping skarn mineralization. *(Chris Benn, BHP Billiton, Chris.J.Benn@bhpbilliton.com)*

Figure 10: Post mineral cover is a serious issue around the Antapaccay mine. Here the 80 meter post mineral Yauri Fm is shown. There are coarse facies variations within this gravel sequence as shown above.

The Processes of Geologic Integration were discussed in a presentation by William Coker, which was followed by additional case histories and a panel discussion of effective mechanisms to integrate the various exploration technologies into effective and productive mineral exploration programs. Bill made a case that true integration of geo-scientific data in mineral exploration involves more than just having geologists, geophysicists, geochemists, and GIS personnel share with or simply pass on their piece of the project data to the others members of the exploration team. Integration should involve intense personal interaction amongst these specialists, from the project planning stage, through implementation to discovery. Bill stated that too often, companies attempt to use and even integrate geochemistry into their exploration without input from a professional geochemist. He queried "How many companies actually have a trained and qualified geochemist, not a retooled geologist, on staff? There is still for many in the exploration community an attitude that every geologist is a geochemist!"

Bill expressed concern that the classic cycle in geochemistry is that a new technique comes along, everyone runs out and tries it, with unskilled people (i.e. no trained geochemists involved), it bombs out because it has not been properly implemented, and the conclusion is that

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"geochemistry" doesn't work. We are starting to see some of this with "Selective Extraction Geochemistry". He feels the question is not necessarily "what" but in most cases "who" doesn't work! In applying any type of geochemistry, it is critical to understand the nature of the surficial environment, the surficial materials that make it up, what processes they have undergone in their development and subsequent modification, and the impact these have on geochemical dispersion processes, particularly those from mineralization. Questions such as "is the overburden residual or transported; what is the nature of soil profile development - if present at all" need to be addressed up front.

As mineral exploration today is global, explorationists are constantly encountering and having to work in many very different regimes. We need to recognise and understand these differences, and their implications for effective survey design. It is critical to determining the nature of how metals will be released, transported and fixed so that in turn one can decide exactly what one should be collecting as a sample medium and what analytical extraction should be utilized. All geoscientists and related personnel should be involved in the project from the start - helping to plan the survey, from the field stage through to data interpretation. Do not seek the expertise needed to interpret and integrate a specific data layer into a project only when a problem arises with that data layer, as in all likelihood, by that time, it is too late to actually effectively utilize those data. For all disciplines,

be more transparent in the way data are processed and presented.

Exploration Success Pyramid

*Figure 11:
Exploration Success through
Technology and Integration.*
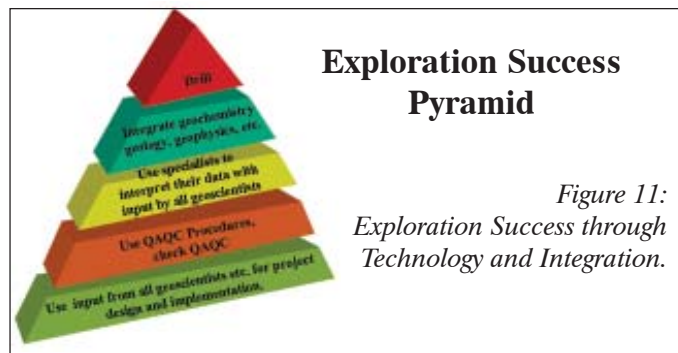
Bill and the Global Geoscience Group of which he is a member outlined the following questions to determine whether an exploration project is truly integrated:

1. Have the geologist, geochemist, and geophysicist etc. actually worked on, or at least visited, the field site together?
2. Has the team produced a map of overburden types?
3. Has the geologist produced a cross section (or several alternative sections) that the geophysicist is using as a starting point for modeling?
4. Is the team seen working together over a light table or at a computer workstation, or are they working separately in their offices?
5. Are the field geologists and core loggers measuring susceptibility and taking samples for physical property measurements?
6. Do all three disciplines participate in project reviews, peer review discussions and all major project decisions?
7. Do you hear conversations like "Hey, could you take a look at this and tell me if it makes geological, geophysical and/or geochemical sense?"
8. At the end of the project, is the joint result better than any one person could produce working alone?

*(William Coker, BHP-Billiton,
William.B.Coker@bhpbilliton.com)*

This **EXPLORE** review has naturally focused upon the exploration geochemistry part of the workshop. Of perhaps greater benefit was the opportunity of learning about innovations in allied exploration geoscience fields from specialists in those fields. It brought to our collective attention that we all have both a common purpose and common challenge: to increase discovery success and decrease discovery cost. This appreciation of and the opportunity to discuss issues of mutual interest with fellow explorationists will likely provide the greatest incentive to take advantage of the synergies available through integration and true teamwork. Let's hope that this type of integrated workshop becomes a standard at our future professional meetings!

Geochemistry Workshop Organizers:
*L. Graham Closs, Colorado School of Mines
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Mary E. Doherty, International Geochemical
Consultants, LLC MaryEDoherty@earthlink.net*
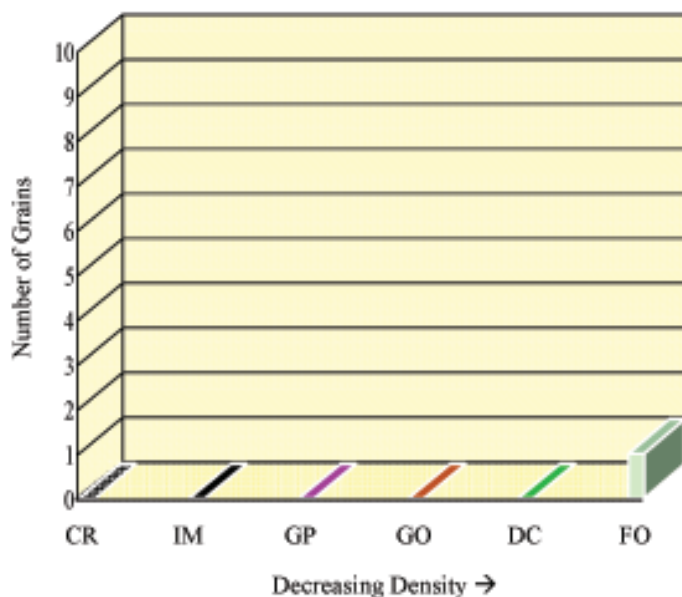
Quality of KIM Processing...

Figure 2: Distribution of lost 0.25-0.5 mm KIM grains. The table tails and heavy liquid lights were each reprocessed once in seach of the missing grains.

Figure 3: Summary of KIM recovery rates for the 0.5-1.0 mm fraction.

10. This extraneous IM loss lowered the overall 0.25-0.5 mm KIM recovery for Sample 10 to 48 percent, well below the 67 to 100 percent level achieved for the other nine samples (Table 4).

Of the 155 KIMs of 0.5-1.0 mm size that were added to the samples, 117 were recovered and 38 were lost (Table 4), including the 4 extraneous IM grains lost from Sample 10. Reprocessing the table tails produced 25 grains encompassing all six species (Table 5, Fig. 4a) whereas reprocessing the heavy liquid lights produced only 1 grain – a low-density FO (Table 5, Fig. 4b).

Discussion and Conclusions

Aside from the extraneous 0.25-0.5 mm CR and 0.5-1.0 mm IM loss from Sample 10 that occurred during gold grain micropanning, the average recovery of the three heaviest KIM species, CR, IM and GP, was in the desired 80-90 percent range for both grain size fractions. Recovery of slightly less dense GO averaged 75 percent, while that of the least dense KIMs, DC and FO, fell to the 50-65 percent range. KIM loss from the fine, 0.25-0.5 mm fraction is mainly to the heavy liquid lights rather than the table tails. This reflects Stokes' Law wherein particle settling rates in a fluid diminish exponentially with decreasing particle size. In contrast, KIM loss from the coarser, 0.5-1.0 mm fraction is mainly to the table tails. This reflects the fact that large grains project further upward into the wet, agitated sand bed

Quality of KIM Processing...
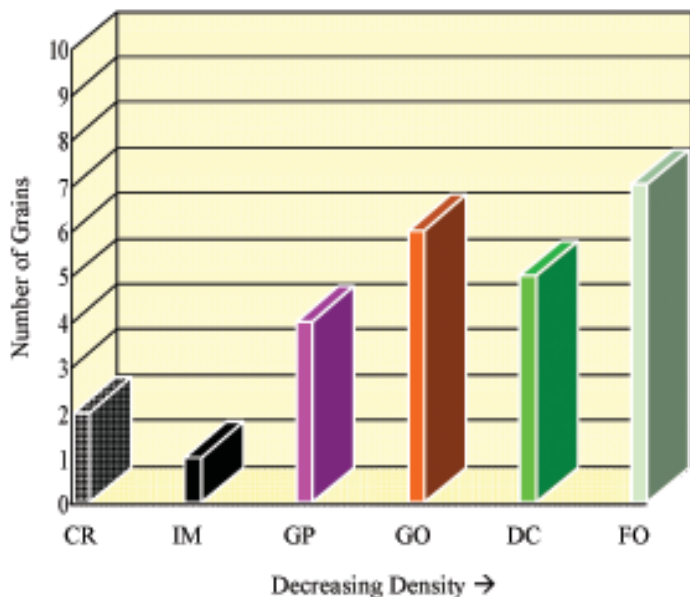
4a - Table Tails

4b - Heavy Liquid Lights

Figure 4: Distribution of lost 0.5-1.0 mm KIM grains. The table tails and heavy liquid lights were each reprocessed once in seach of the missing grains.

Sample	Number of Grains by Species												Total Grains		Total %
	CR		IM		GP		GO		DC		FO				
No	Spiked	Recovered	Spiked	Recovered	Spiked	Recovered	Spiked	Recovered	Spiked	Recovered	Spiked	Recovered	Spiked	Recovered	Recovered
1	0	0	3	3	0	0	0	0	0	0	0	0	3	3	100
2	0	0	3	3	0	0	0	0	0	0	0	0	3	3	100
3	12	12	6	6	7	4	0	0	0	0	0	0	25	22	88
4	0	0	8	8	4	4	4	1	3	1	2	0	21	14	67
5	6	5	0	0	6	6	0	0	0	0	0	0	11	11	92
6	0	0	0	0	5	4	3	2	3	2	0	0	11	8	73
7	0	0	0	0	0	0	8	6	3	3	8	4	19	13	68
8	0	0	0	0	0	0	8	8	5	4	8	4	21	16	76
9	10	9	0	0	6	6	3	2	0	0	0	0	19	17	89
10	0	0	7	2	0	0	0	0	8	4	6	4	21	10	48
Total Grains	28	26	27	22	28	24	26	19	22	14	24	12	155	117	
Total % Recovered	93		81		86		73		64		50		75		

Table 4: Summary of KIM recovery rates for the 0.5-1.0 mm fraction.

Quality of KIM Processing...

	Lost							Found in Table							TailsFound in Heavy Liquid Lights							Unaccounted						
Spike No.	CR	IM	GP	GO	DC	FO	Total	CR	IM	GP	GO	DC	FO	Total	CR	IM	GP	GO	DC	FO	Total	CR	IM	GP	GO	DC	FO	Total
1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
3	0	0	3	0	0	0	3	0	0	3	0	0	0	3	0	0	0	0	0	0	0	0	0	0	0	0	0	0
4	0	0	0	3	2	2	7	0	0	0	2	2	0	4	0	0	0	0	0	0	0	0	0	0	1	0	2	3
5	1	0	0	0	0	0	1	1	0	0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
6	0	0	1	1	1	0	3	0	0	1	1	1	0	3	0	0	0	0	0	0	0	0	0	0	0	0	0	0
7	0	0	0	2	0	4	6	0	0	0	2	0	4	6	0	0	0	0	0	0	0	0	0	0	0	0	0	0
8	0	0	0	0	1	4	5	0	0	0	0	1	3	4	0	0	0	0	0	1	1	0	0	0	0	0	0	0
9	1	0	0	1	0	0	2	1	0	0	1	0	0	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0
10	0	5	0	0	4	2	11	0	1	0	0	1	0	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Totals	2	5	4	7	8	12	38	2	1	4	6	5	7	25	0	0	0	0	0	1	1	0	4	0	1	3	4	12

Table 5: Distribution of lost 0.5-1.0 mm KIM grains.
The table tails and heavy liquid lights were each reprocessed once in seach of the missing grains.

on the sloping deck of the table and therefore are more prone to being caught in the cross flow that conveys undesirable low-density mineral grains to the table reject. ODM largely compensates for this biased loss of 0.5-1.0 mm KIM grains by retabling the reject on all KIM projects but the test results show that some loss is still occurring.

Clearly, KIM recoveries during both primary table and secondary heavy liquid concentration are limited by the laws of physics. Therefore, the potential for raising recovery of the heaviest KIMs above the present 80-90 percent level, or recovery of the lightest KIMs above the 50-65 percent level, is limited. Consequently, the emphasis should be on ensuring that the laboratory workers are sufficiently well-trained and vigilant to maximize the separation capabilities of the tables and heavy liquids and minimize the potential for extraneous KIM losses such as the loss of CR and IM grains experienced while micropanning Sample 10 for gold grains. With the physics of the KIM separations now being well established, more geological variables such as sample granularity and heavy mineral content can confidently be introduced into future spike tests.

Reference cited

Ontario Geological Survey (OGS), 2001, Results of modern alluvium sampling, Fraserdale Area (OFR 6044), Chapleau Area (OFR 6063), Foleyet Area (OFR 6065) and Coral Rapids Area (OFR 6068), Northeastern Ontario: Operation Treasure Hunt — Kapuskasing Structural Zone

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Geochemical Laboratory Updates... *continued from page 7*

Robotic Mineral Sample Preparation for the 21st Century

Consulting analytical laboratories servicing exploration and mining industries in Western Australia, generally test thousands of samples per day. These samples are collected in remote areas of our state where there might be a possibility of gold and other minerals being found, but many samples also come from work where an inferred deposit is being sampled by infill drilling to confirm the presence and extent

Geochemical Laboratory Updates...
continued from page 22

of an ore body. By using the latest equipment and analytical methods, this work can be done with high quality but at relatively low cost and Ultra Trace strives to be leaders in the adoption of new technology so that we can offer the best service possible to our clients.

Samples typically weigh two to three kilograms, however the analytical staff may only test a small portion of this bulk sample. The first part of the analytical process involves pulverising the sample to a fine powder in a sample preparation mill so that in the laboratory, a number of portions may be taken for analysis that will be reproducible and will truly represent the bulk sample that was delivered to the laboratory.

The pulverising process, however, can also damage the sample. The grinding may not be fine enough, the pulverisers may introduce contaminants, small fractions of a sample left behind in the preparation of one sample may contaminate the next sample prepared in that machine and lastly, because it is a manual task, operators sometimes get samples mixed up. These issues led Ultra Trace to investigate installation of a robotic sample preparation cell for this task.

In the pulverisers we use, a large bowl, 30 cm diameter and 10 cm deep is mounted in a machine that makes the bowl gyrate (or wobble). In the bowl is a steel puck (like a disc) and the sample is placed in this assembly. A lid is placed on the bowl and as the machine gyrates, the heavy steel puck flies around inside the bowl and grinds the sample to a powder between its edges and the sides of the bowl.

These mills have a working capacity of about 3500 grams of sample

After many trials, we were able to modify a sample preparation mill so that the bowl could be easily removed from the mill (they are usually bolted in place). This enabled the bowls to be robotically handled so that the bowl, puck and sample could be handled away from the mill with other specially designed equipment. By removing the bowl from the mill, it can be presented to the operator for the sample to be added. At the same time it is inspected for cleanliness.

Ultra Trace constructed a robotic cell that has two robots and six sample preparation mills (Plate 1). The first robot supplies the operator with clean bowls and pucks for manual addition of the sample whilst the second robot transports the bowls to and from the mills, sample bagging system and washing stations. This has resulted in quite good throughput of samples through the cell with a prepared sample being produced every 50 seconds.

Another feature of the cell is that the sample bowls and pucks are washed with very hot water between each sample. This results in much lower cross contamination between samples than the manual system of cleaning which relies on blowing the residual dust out of the bowl with an air blast.

The cell is equipped with an automatic sample divider and bagging system. From the two to three kilograms of sample received, about 300-400 grams of pulverised material is placed in a "wire tie" paper sample bag for analysis in the laboratory. Usually, this portion of the original sample is returned to the client when analytical work is complete. The

Geochemical Laboratory Updates... *continued from page 23*

remainder of the sample is packaged in a UV-stabilised, heat-sealed plastic bag and depending on our clients preference, is either stored, dumped or returned. The bagging system applies bar-coded labels to both the packet and the excess material in the plastic bag.

This robotic cell has now processed over 160,000 samples and continues to provide many benefits, both to Ultra Trace and to our clients:

1. Operators work in a clean environment. Safety around the robots is ensured because the "Fortress" room isolation system prevents the robotic arms being used when the doors are opened.
2. Operators exposure to airborne dust is virtually eliminated.
3. The possibility of samples being switched is eliminated because samples must be processed in a pre-determined order. The operator must read the sample number from the sample bag to identify the next sample to process and he can only enter the sample into the cell if the number entered is the next one to process.
4. Manpower efficiency.
5. More consistent product because pulverising time is linked to sample weight. ie the bigger the sample, the longer it is pulverised
6. Less cross contamination of samples because the pucks and bowls are washed with hot water (90° C) rather than just air-blast cleaned.
7. The cell operator visually inspects the pulveriser bowl for cleanliness before the sample is placed in it.
8. All samples are bar-coded to ensure sample identity during analytical processing.
9. Residues are heat-sealed in a plastic bag which is UV stabilised to ensure long-term storage integrity.
10. The cell uses pulverising technology that is tried and proven. This robotic cell is an adaptation of existing technology rather than adoption of a new pulverising technique.

Should you require additional information regarding this process, please do not hesitate to contact Ultra Trace (colin@ultratrace.com.au) for a demonstration video or visit to the laboratory.

ROUND ROBINS … *continued from Page 13*

measuring 10's of kilograms would be necessary. The formula to calculate resolution is:

$$DI = 1.41 * (USL – LSL) / UM \qquad (2)$$

where USL and LSL are the upper and lower specification limits, respectively and the unit of measure (UM) is the smallest interval measurable by the measurement system. The value of DI should be greater than five and ideally above ten.

Linearity is a measure of the variation in accuracy over the full range of the gauge. The measure of linearity requires some knowledge of the truth. One way is to submit 5 or 6 repeat samples of different certified reference materials covering the range of the measurement system and calculate a regression of the difference between the results and the published value of the standard versus the published value. The P-value for the regression slope should be less than 0.05 (for an alpha risk of 0.05) and the minimum and maximum B/T% ratio of different standards, calculated as follows:

$$B/T\% = Bias / Tolerance * 100 \qquad (3)$$

where Bias is the bias of the measurement system at a specified grade and Tolerance is equal to USL – LSL, should be less than 10%.

Stability is a measure of the variation of the process accuracy over time. The measurement system must be able to measure the same parts to the same degree of accuracy at different times. In the case of deposit delineation this can be several years. Instabilities in the measurement system (e.g. instrumental drift) may occur over short periods of time and are not necessarily bad as long as the problem is recognized and the calibration frequency adjusted accordingly.

Accuracy is a measure of how well the process output reflects the truth and it is determined by calibration studies. Good calibration can effectively remove almost all bias from the observed process output. In the case of assays a round-robin survey of properly prepared project materials or, alternately, the purchase of commercially available reference materials can ensure that bias is absent from the assay results.

Finally, the precision of the measurement system is a measure of the variations caused by the measuring device. This includes the variation attributable to lack of repeatability or reproducibility. Repeatability is the inherent variability of the measurement system and is measured under constant conditions, whereas reproducibility is the variation in measurement made by different operators combined with the variation caused by the interaction[2] between the operators and the parts. This combination of variance is conveniently analyzed with the "Gage R&R" and defined by equations (4) and (5):

$$Var_{R\&R} = Var_{Repeat} + Var_{Reprod} \qquad (4)$$
$$Var_{R\&R} = Var_{Repeat} + (Var_{Oper} + Var_{Oper.Part}) \qquad (5)$$

The variance between parts is not part of the precision of the measurement system. It is used, however, in the computation of the Gage R&R index, which gives an idea of how good (or bad) the precision of the measurement system is: the precision of the measurement system is, in effect, compared against the variability of the process.

The Gage R&R is therefore the sum of 3 component variances and is usually reported as the percentage of the

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2. The interaction between parts and operators is evaluated by the Two-Way Analysis of Variance (Two-Way ANOVA) which measures whether the choice of operator influences the output from the different parts.

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total variation in the process consumed by one or all components of measurement variation.

Assays as a measurement system

Assays are also a process with outputs (concentration of metals) determined from inputs (rock samples). The process itself typically consists of sampling, sample preparation (crushing, splitting, and pulverizing), the weighing of aliquots, dissolution of components (digestion or fusion) and instrumental analysis. Each of these steps contributes to the total accuracy and precision of the output. The objective of this process is to determine the concentration of metals in a rock sample that is both accurate (not biased) and representative of the rock from which the sample was taken (precise) (Gy, 1982; Riddle, 1993). Unfortunately the rock itself is not homogeneous over short distances and samples of core taken from a drill hole rarely reflect precisely the composition of the parent mass the samples purport to represent.

Assays as a measurement system suffer from several unique problems. The first is that there is no mean for the process. Although the deposit may have an average value, it is understood that zoning exists within the deposit so that the assays do not represent samples from a "unique" distribution but come from a whole range of distributions, each with its own mean. Indeed, different assays each have their own "average" value, so the process output is always a moving target. Secondly, the measurement system must be capable of making accurate and precise measurements over a wide range of concentrations, or at least between the cut-off values up to the highest concentrations found in the deposit. Finally, the destructive nature of assaying implies that it is usually not possible to measure the performance of each assay but only that of the control samples included in the batch. These issues will be addressed below.

Resolution, linearity, stability, accuracy and precision

For assays to be evaluated as a measurement system, each of the elements of resolution (linearity, stability, accuracy and precision) must be assessed in turn. Assay results are usually reported to two or three decimal places and even if the third decimal is generally not significant, it is accepted that assays for major components can be confidently reported to the second decimal place which corresponds to increments of 0.01. For a deposit with an average grade of, say, 2.0% Cu and an analytical method and standards that allow determinations to be done with a tolerance of ± 5.0%, the resolution can be calculated as per equation (1):

$$DI = 1.41 \,(2.1 - 1.9) / 0.01 = 28$$

This indicates that the assaying measurement system has sufficient resolution to be able to detect small process variations. Whether these detected variations are related to the process itself or to the measurement system is determined from a study of precision.

Linearity is measured by comparing the results of the measurement system against the truth across the range of the measurement system. With assays this would mean from well below the cut-off grade to the highest grade encountered in the deposit. This is usually constrained by the standards used, which usually represent the cut-off grade, the average grade, and near the highest grades in the deposit. A second, perhaps less efficient way of measuring linearity is to use the round-robin data itself. By calculating the regression of the difference between the analyzed values and the accepted values vs the accepted values for all the standards, for each lab, it is possible to evaluate linearity. Evidently the fact that the same data is used to estimate the accepted value for the standards will necessarily imply that some labs will be biased high and some low for different parts of the grade distributions. Nonetheless a good performing laboratory can and should have a statistically significant slope on the regression line.

Stability is related to the accuracy of the measurement system over time. It is normally addressed by regular calibration. With assays, stability is a common issue because mineral resources are typically delineated over a long period and by incremental drilling programs. However, by using the same standards or, alternately, by calibrating new control materials along with old ones, it is possible to ensure that successive drilling programs attain the same level of accuracy. In the case of previous programs that were undertaken without adequate controls, the solution is to re-analyze a suite of pulps or rejects of older drilling programs with the same standards being used in the current sampling program to bring the whole assay database to the same level of accuracy.

The accuracy of assays is determined by incorporating into the batches pulp materials with known concentration distributions (i.e. mean and standard deviation). Unfortunately this covers only the digestion and analysis part of the process, albeit the ones most susceptible to cause bias. Additional controls on accuracy come from submitting the pulps to a secondary laboratory, usually one that was part of the round-robin survey if project standards were prepared. Some bias may arise from cross-contamination of samples during sample preparation. This can be prevented by frequent cleaning of the equipment, and can be easily monitored by inserting blank material into the sample-processing stream. Some bias may also arise from the use of deficient crushing or grinding equipment, or from the improper operation of otherwise acceptable equipment, particularly the riffle splitter. These biases are very difficult to detect without detailed studies. Finally the samplers may cause bias when oversampling the core half that contains more mineralization, or the opposite when favoring the less mineralized portion of the core. Fortunately, bias can be prevented here by good sampling protocols and can be detected, albeit with difficulty, by incorporating systematic core-duplicate sampling into the drilling QA/QC program. In the final analysis, every effort must be made to ensure there is no measurable bias in the assays because of its profound impact on the financial value of the project.

Variance in sampling and assaying is additive. It is also cumulative, so that the variance measured from coarse crush

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replicates includes all sources of variance that have been subsequently introduced further downstream in the process. The variance of pulp sub-sampling, digestion, dilution, and instrumental analysis can be conveniently modeled by the round-robin survey. Using equation (4) the parts are represented by the different standards and the participating laboratories represent the operators. From this equation can be obtained the component variances for the parts (repeatability) and the operators plus the operator-part interaction (reproducibility). Repeatability is also defined as the within-lab variance as it measures the average variance of all the parts and across all the operators whereas reproducibility is the between-lab variance since it measures the average variance of the means of all the parts across all the operators (Noranda, 2001). The component variances and total variance can then be used to measure performance against the tolerance of the process. This we will define as the maximum acceptable variance from sampling, sample preparation, and analysis. It is because pulp standards have been homogenized and are therefore free from the natural variance arising from the sampling and sample preparation process that we can determine what proportion of the total variance is being consumed by the assaying itself.

The issue of tolerance in assays is complex because the customer (who defines the defect and hence the tolerance of the process) is normally concerned with the precision of the resource estimation itself which is dependant only in part on the precision of assays. It depends also on other factors such as the geological interpretation, the deposit type, the continuity of the mineralization, and the grade distribution in the deposit. The precision of assays impacts mostly in the case of precious metal deposits, especially those with important nugget effect, where sampling may require special protocols to ensure that sub-sampling variance is controlled. Otherwise the traditional approach is to rely on the large number of samples typically present in a resource database to limit the variance since the precision is proportional to $1/(N)$ square root. This may apply for the overall grade of the deposit but the number of samples that typically contribute to an individual resource block is only a small subset of the database so that the impact of high sampling and assaying variance may not be insignificant.

Consider a tolerance of ±10% of the value of a resource block, half of which could be attributable to the assay results (±5.0%). Furthermore, assume that the resource block is estimated from 100 assays. All else being equal the tolerance of individual assays should be 10 times greater or ±50% because precison is proportional to $1/(N)$ square root. Let us be more demanding and place the tolerance at ±40%. This method of determining process tolerance is admittedly not very elegant but at least relates the tolerance to the customer's expectation. The specifications are then calculated as follows:

$$\text{USL} = \text{Mean} +40\%, \text{LSL} = \text{Mean} -40\%, \text{and}$$
$$\text{Tolerance} = \text{USL} - \text{LSL}$$

The total tolerance is therefore equal to 80%, or 0.8 times the mean of the process which, in the case of assays would be the average grade of the deposit. We can now use this information along with the round-robin data to evaluate assays as a measurement system.

The complete analysis of variance in measurement systems is greatly facilitated by the Gage R&R procedure that is implemented in various statistical software packages, including Minitab. Within Noranda's Six Sigma program a number of measures have been adopted to interpret the Gage R&R. These will be reviewed in the case study presented below.

El Morro Project

An example is presented here of a Gage R&R performed on results of a round-robin survey on pulp standards prepared for the El Morro project in northern Chile. These standards were prepared to provide control materials for the 2002 diamond drilling program undertaken by Noranda and its partner, Metallica Resources.

Site location.

The El Morro project is located in northern Chile approximately 650 km north of the capital city, Santiago and 80 km east of the town of Vallenar. The property is owned by Metallica Resources (MR) and is under option to Noranda Inc. who may acquire up to 70% interest (Figure 1).

The El Morro project occurs within the southern extension of the Chilean Oligocene Porphyry Copper Belt that extends more than 300 kilometers to the north and hosts some of the largest copper deposits in the world. The project may occur along the southern extension of the West Fissure Fault System that is associated with the El Salvador, Escondida, Chuquicamata, and other porphyry-copper deposits but its gold-rich nature also suggests similarities with the deposits found in the Maricunga district (Noranda, 2002).

The property is located within a 16-kilometer wide north-south trending graben structure. Tertiary-aged, mineralized porphyritic stocks intrude contemporaneous volcanic and older, volcanic, sedimentary and intrusive units. In addition, more recent Atacama gravels and ignimbrites unconformably overlie a good part of the property covering and preserving the porphyry mineralized systems from erosion.

A number of mineralized porphyry systems have been identified on the property. The most important is the La Fortuna zone that, as of September 2002, was estimated to contain 590 million tonnes of material grading 0.56% Cu and 0.46 gpt Au at a 0.3% copper cutoff (see http://metal-res.com). La Fortuna is a typical copper and gold-rich porphyry deposit with a primary sulfide zone centered on a diorite to granodiorite porphyry complex and overlain by a supergene enrichment zone.

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Following the 2001 drilling program and success obtained on the La Fortuna mineralized zone a decision was made to manufacture standards for future drilling programs. A total of five pulp standards was produced from coarse rejects of the 2001 diamond drilling program. Reject samples were selected on the basis of mineralization types and range in copper and gold grade. The result is a set of five pulp standards that cover the range of mineralization types found

Standard	Description	Cu (%)		
		Mean	Std	RSD%
EM1	Barren Leached Zone	0.0061	0.0004	5.74
EM2	Low Grade Leached Zone	0.0133	0.0006	4.13
EM3	Supergene Enriched Zone	1.0670	0.0302	2.83
EM4	Primary Zone, cut-off grade	0.3138	0.0076	2.43
EM5	Primary Zone, average grade	0.6220	0.0096	1.54

Table 1: Summary of round-robin results for El Morro pulp standards.

in the La Fortuna deposit and the grades encountered within these types (table 1).

The samples were prepared at ALS-Bondar's Coquimbo laboratory where they were pulverized to –200 mesh and passed through an electric vibrating sieve. The oversize material was discarded. The standards were subsequently homogenized in a pulp mixer for 24 hours. Afterwards the samples were split into 200 g packets and sealed in plastic bags.

The five standards were submitted to a total of seven labs for multiple determinations. Ten packets of each standard were submitted randomly to each lab. Results were received and compiled to obtain the mean value and standard deviation for each standard using the method proposed by Smee (2001) (see Table 1).

The distribution of assay results along with the low relative standard deviation (RSD%) obtained for the copper assays indicate a very high degree of homogeneity for the standards. In particular, the low standard deviation and excellent agreement between laboratories for EM3, EM4, and EM5 (Figure 2) indicate that these standards will provide excellent accuracy control and this was borne out during the 2002 drilling campaign.

For the purposes of the MSA standard EM1 was removed because it is essentially a blank and its very low metal concentration is near the detection limit of the
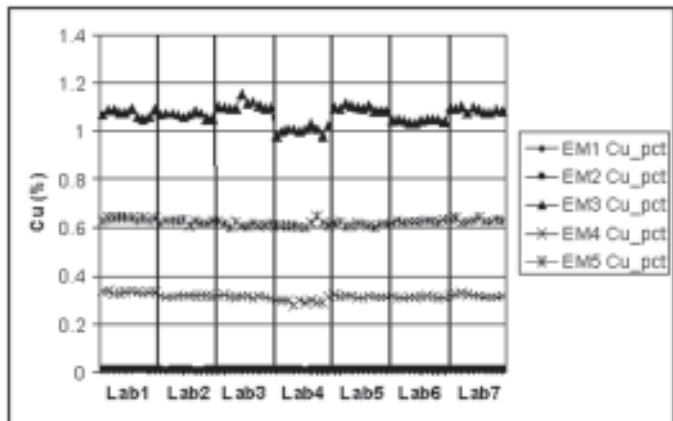
Figure 2: Control chart for El Morro pulp standards.

analytical method and arguably outside the range of the measurement system.

For the standards to be evaluated as a measurement system the five criteria mentioned earlier must be satisfied. In addition, heteroskedasticity is a well-known problem as the variance of assays increases with concentration resulting in possibly lower accuracy in high-grade assays. Fortunately, the grade distribution at El Morro is fairly narrow with concentrations of copper ranging from the cutoff (0.3% Cu) up to about 1.5% in the supergene enriched zone.

The average grade of the standards is 0.506% Cu. If the tolerance is ±40% of the assay value we have the following:

$$USL = 0.506\% + 40\% \quad \text{and} \quad LSL = 0.506\% - 40\%$$
$$Tolerance = (0.708 - 0.305) = 0.4\%Cu$$

As mentioned previously, the resolution of the measurement system is more than adequate and the accuracy and stability can be maintained during the project through insertion of pulp standards and blanks. Additional measures include submitting a fraction of the pulps to a secondary laboratory for check assays and by collecting a certain percentage of core duplicates to test for sampling bias, also called overselection.

Linearity was tested using the method outlined above. The difference between the round-robin assays for each laboratory minus the accepted value for each standard (Diff_Cu) were plotted against the accepted value of the standards (Figure 3). The regression was calculated and five out of the seven labs were found to have a significant P-value (Table 2). However of these only one laboratory was found to have a B/T% ratio greater than 10%. There would be legitimate concern about using this laboratory as the project's primary laboratory.

Coming soon in the AEG **EXPLORE** newsletter:

Technical articles and letters to the editor are encouraged as submissions for discussion within the newsletter. Each issue of **EXPLORE** contains a series of short discussion papers which provide either an update on a particular geochemical topic, or present current debates about issues of interest. Suggestions for future "Focus" topics may be forwarded to the editor, Mary Doherty (Email: MaryEDoherty@earthlink.net).

Issue: **Focus topic and Contact:**

120   3-D Vectoring and Data Integration
       Robert Jackson rgjackson@ctnis.com
Contributor Deadline   May 31, 2003
Publication Date: July 2003

121   Environmental Geochemistry Update
       Rob Bowell rbowell@srk.co.uk/srk003@aol.com
Contributor Deadline   August 31, 2003
Publication Date: October 2003

122   Soil Gas Chemistry
       Patrick Highsmith phighsmith@alschemex.com
Contributor Deadline   November 30, 2003
Publication Date: January 2004

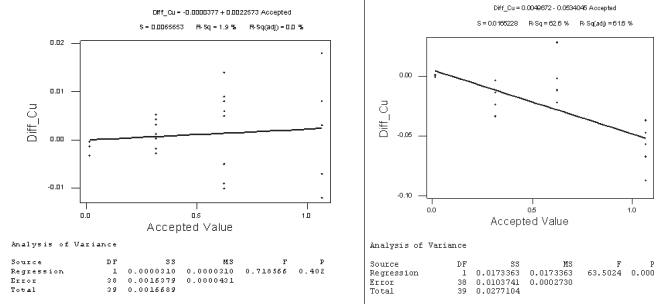# Certified Reference Material
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Figure 3: Examples of regresson of Diff_Cu vs Accepted Values. A) Regression for Lab3. The P-value is equal to 0.402 and therefore the slope is not significantly different from zero. B) Regression for Lab7. The P-value is equal to 0.000 and therefore the laboratory data are not linear. Moreover the maximum bias is -0.052 which translates to a B/T% of -12.88%.

Laboratory	P-value	Min/Max B/T%
Lab 1	0.337	
Lab 2	0.000	4.83%
Lab 3	0.402	
Lab 4	0.000	7.01%
Lab 5	0.000	-4.36%
Lab 6	0.000	4.65%
Lab 7	0.000	-12.88%

Table 2: P Values for regression of Diff_Cu vs Accepted Values and maximum B/T% (bias/tolerance) for the seven laboratories used in the El Morro round-robin survey. P-values less than 0.05 are significant for an alpha risk of 5% which means that the slope of the regression is significantly different from zero, and therefore that the laboratory in not linear. In cases when P is significant B/T% should not exceed 10% which only occurs with Lab 7.

GEOANALYSIS

5th International Conference on the Analysis of

Geological & Environmental Materials

8th–11th June 2003
Arktikum Building
Rovaniemi, Finland
Organised by
Geological Survey of Finland
www.gsf.fi/geoanalysis/geoanalysis2003

GTK

and
International Association of Geoanalysts
www.geoanalyst.org

The final criterion, precision, is studied with the ANOVA method of the Gage R&R. The procedure is available in Version 13.31 of Minitab augmented with Version 2.11 of the Six Sigma Academy module. The data must be organized in a table with columns for the standard (part) and the laboratory (operator) in addition to the elemental assays. The ten replicates performed by each lab for each standard are much larger than the minimum three replicates required, and the number of labs used (seven) is greater than the minimum three required. The rule of thumb is that the product of the parts and the operators should be equal to or greater than 15.

An example output for the Gage R&R is presented in Table 3 and in Figure 4. The top part of the table shows the two-way ANOVA table including the interaction of the operator and the parts. The P-value for the parts is zero and this is to be expected; if it was not below 0.05 we would conclude that all the parts were the same. The P-value for the LabID is not significant, meaning that the different operators do not measure the parts in a significantly different way. Finally, the P-value for the LabID*Standard interaction is equal to 0.000 which indicates that the labs don't measure all the standards in the same way.

Table 3: Summary output table of Gage R&R procedure from Minitab Ver 13.30.

Figure 4: Graphical output from the Gage R&R procedure in Minitab Ver 13.30.
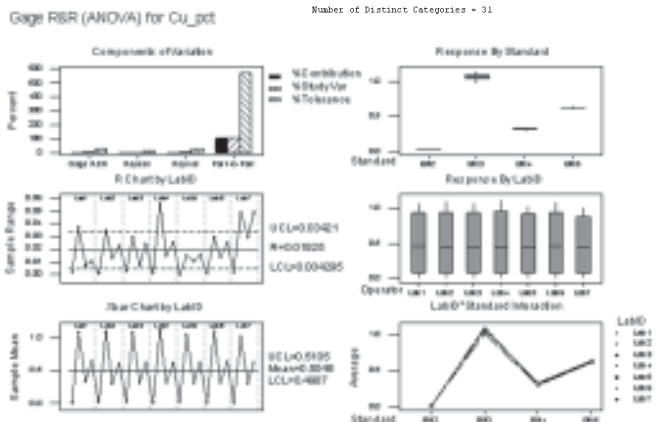
The middle part of Table 3 shows the different component variances and their percent contribution to the total variance. Here, the part-to-part variation is rightfully the largest part of the variation. The total Gage R&R takes up only 0.2% of the total variation, and repeatability is only 0.03% vs 0.17% for reproducibility. The lower part of the table shows the standard deviations in the first column for the same components. This is followed by the Study Variation, which is simply 5.15 times the standard deviation or about 99% of the process. When recalculated as a percentage in the %Study Var, the data show the percentage of the process taken up by each component. Notice that the

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components do not add up to 100% because only variances are additive. The final column, %Tolerance represents the Study Var times 100 and divided by the process tolerance, in this case 0.404. The general rule to interpret these numbers is that the Total Gage R&R should be less than 30% and only if it is less than 10% will the measurement system accompany process improvement to six sigma levels. Here we see that the Total Gage R&R is equal to 25.93 which is acceptable. Closer inspection reveals that the reproducibility is 23.84% and more than twice the repeatability at 10.20%. This means that assay results from a single lab would have better precision than when results from multiple labs are combined.

The final line in Table 3 shows that the discrimination index (DI) is equal to 31, an excellent result and consistent with the fact that the part-to-part variation is the largest component of the system. This index is another method of calculating resolution that takes into account the results of the Gage R&R. The lower the value of the total gage and the higher the DI.

Implications for assays as a measurement system in resource estimation

The evaluation of the round-robin survey of pulp standards shows that assays are an acceptable measurement system but do tend to suffer from lower reproducibility in relation to repeatability. The implication of this is, not surprisingly, that labs tend to agree more with themselves than with other labs. However in some cases the reverse is observed, namely that the reproducibility is better than repeatability and will typically occur when the homogeneity of the standards is poor, a common feature of precious metal standards. In this case the variances of the measurements on the standards are larger than the variances of the averages of the standards for all the labs. The nice thing about the Gage R&R is that it allows us to compare the degree of homogeneity of the standards against the variations caused by the use of different operators. With highly homogeneous standards most of the gauge will be occupied by the operator variance: a good reason to do most of the assays in a single lab. Check-assaying in a secondary lab remains necessary to monitor accuracy, however, and should not be discontinued. Ironically, with less homogenized standards the incentive to concentrate all the assaying in a single lab is not as compelling, at least from the perspective of precision.

Conclusion

Assays can be considered from the Six Sigma perspective as a measurement system used to estimate a mineral resource. The round-robin survey is a convenient model to study the adequacy of the measurement system for this task. A round-robin study of copper standards prepared for the El Morro project in northern Chile shows that assays can allow proper measurement of the deposit. At assay tolerance levels appropriate to produce estimations of resource blocks with better than ±5% error, the assays can be said to be an acceptable measurement system.

The Cu data from the El Morro standards indicate that reproducibility occupies more than twice the %Tolerance than repeatability but that the %Tolerance of the total Gage R&R remains within acceptable limits. This indicates that the standards are very well homogenized and that they will provide good monitoring of the assay batches.

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### Selected Laboratory Web Sites of Interest:

The following column highlights some of the favorite laboratory web-sites of our members. *If we have missed your favorite site and you would like to contribute, please send site addresses with a brief description along to the editor*, and we will include them in our list. This list is of course provided for information only; AEG and the editors do not endorse nor specifically recommend any of the service providers.

Geochemistry Link Sites

www.aeg.org ....Association of Exploration Geochemists

Geochemical Laboratories

Company	Web address
ActLabs	www.actlabs.com
Acme Analytical Laboratories Ltd	www.acmelab.com
ALS-Chemex Labs	www.alschemex.com
Amdel	www.amdel.com
Atoka Geochemical Services Corp.	www.atoka.com
Becquerel Laboratories, Inc	www.becquerellabs.com
Elemental Research, Inc	www.elementalresearch.com
Genalysis	www.genalysis.com.au
Inspectorate	www.inspectorate.com
Overburden	www.odm.ca
Rocklabs	www.rocklabs.com
SGS	www.sgs.com
XRAL Laboratories (SGS)	www.sgs.con/xral
Ultra Trace	www.ultratrace.com.au
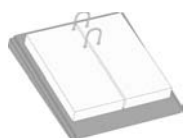
Calendar of Events

International, national, and regional meetings of interest to colleagues working in exploration, environmental and other areas of applied geochemistry.

■ April 1-3, 2003, **Geological Society of America, Cordilleran Section meeting,** Puerto Vallarta, Mexico. INFORMATION: Elena Centeno, National University of Mexico, Ciudad Universitaria, Coyoacan, 04510 Mexico. Phone 525-622-4314, Fax: 525-550-6644. http://www.geosociety.org/meetings/

■ April, 6-9, 2003, **16th Industrial Minerals International, Congress**, Le Centre Sheraton, Montreal Quebec, Canada. Contact: Mike O' Driscoll, Industrial Minerals Magazine, Park House, Park Terrace, Worcester Park, Surrey KT47HY England; Tel. 4420.7827.6444; Fax 4420.7827.6441; Email modriscoll@indmin.com; Web sitewww.indmin.com

■ April 7-11, 2003 **EGS, AGU, and EUG Joint Assembly, Nice Conference Centre**, Nice, France , by the EGS, AGU, EUG. (Meetings Department, 2000 Florida Avenue, NW; Washington, DC 20009 USA, Phone: +1-202-462-6900 FAX: +1-202-328-0566 EMail: meetinginfo@agu.org Web: http:/www.copernicus.org/egsagueug/).

■ May 04 - 07, 2003, **CIM Montreal 2003 - Annual General Meeting**, CIM Geological Society, Montreal, Quebec. INFORMATION: Prof. David Lentz - V.P. CIM Geological Society Dept. of Geology, University of New Brunswick, Box 4400, 2 Bailey Drive, Fredericton, New Brunswick, E3B 5A3 CANADA, Tel: (506) 447-3190, Tel: (506) 453-4803 - main office, FAX: (506) 453-5055, email: dlentz@unb.ca, www.cim.org.

■ May 4 - 7, 2003 **CIM 2003 Conference and Mining Exhibition, Montreal, Quebec Canada**. Organizers: CIM & Tradex. Tel: +1 514 939 2710, Fax: +1 514 939 2714 Email: smajor@cim.org, URL: www.tradex.cim.org.

■ May 7-9, 2003, **Geological Society of America, Rocky Mountain Section meeting,** Durango, CO.

■ May 12-16, 2003, **GeofluidsIV: Fourth international conference on fluid evolution, migration and interaction in sedimentary basins and orogenic belts**, Utrecht University, Utrecht, The Netherlands , by the Netherlands Institute of Applied Geoscience TNO-National Geological Survey. (Ms. J.M. Verweij, PO Box 80015, 3508 TA Utrecht, The Netherlands, Phone: +31 30 256 4600 FAX: +31 30 256 46 05 EMail: j.verweij@nitg.tno.nl Web: http://www.nitg.tno.nl)

■ May 18-24, 2003 39th **Forum on the Geology of Industrial Minerals**, John Ascuaga's Nugget Hotel & Casino, Sparks, Nevada, USA , by the Nevada Bureau of Mines and Geology, Nevada Division of Minerals, and Nevada Mining Association. (Terri Garside, NBMG/MS 178, University of Nevada, Reno, NV 89557-0088, Phone: 775-784-6691 ext 126 FAX: 775-784-1709 EMail: tgarside@unr.edu Web: http://www.nbmg.unr.edu/imf2003.htm)

■ May 25-28, 2003, **GAC/MAC/SEG Joint Annual Meeting**, Sheraton Wall Centre, Vancouver, British Columbia, Canada, by the Geological Association of Canada, Mineralogical Association of Canada, Society of Economic Geologists. Venue West Conference Services, 645-375 Water Street, Vancouver, BC V6B 5C6 Canada, Phone: +1 604, 681-5226 FAX: +1 604 681-2503 EMail: vancouver2003 @nrcan.gc.ca Web: http://www. vancouver2003.com)

■ May 25 - 28, 2003 **Sudbury 2003 Mining and the Environment Conference**, Laurentian University, Sudbury, Ontario, Canada. Organizers: Centre for Environmental Monitoring and Canadian Land Reclamation Association Tel: +1 675 1151 ext 5054; Fax: +1 673 6530 Email: sudbury2003@laurentian.ca URL: www.sudbury2003.ca

■ May 26-28, 2003 **2nd International Symposium on Contaminated Sediments : Characterisation, Evaluation, Mitigation/Restoration, Management Strategy Perfor-mance**, Loews Le Concorde Hotel, Quebec City, Quebec, Canada , by the ASTM (American Society for Testing and materials), CGS (Canadian Geotechnical Society), CSCE (Canadian Society of Civil Engineering). Helene Tremblay, Département de géologie et de génie géologique, Universite Laval, Phone: 1-418-656-2193 FAX: 1-418-656-7339 EMail: 2sisc@ggl.ulaval.ca Web: http://www.scs2003.ggl.ulaval.ca/

■ June 1-5, 2003, **AMERICAN SOCIETY for SURFACE MINING and RECLAMATION (ASSMR) 19th National Meeting and Billings Land Reclamation**, Billings, Montana. INFORMATION: Dennis Newman, dneuman@montana. edu, http://www.ca.uky.edu/assmr/Upcoming_Events.htm

■June 8-10, 2003, **3rd Canadian Conference on Geo-technique and Natural Hazards,** Edmonton, Alberta. INFORMATION: Corey R. Froese, c/o AMEC Earth & Environmental Limited, 4810 - 93 Street, Edmonton, Alberta, T6E 5M4, Fax (780) 435-8425, Email: corey.froese@amec.com.

■June 9 – 11, 2003, **Geoanalysis 2003, 5th International Conference on the Analysis of Geological and Environ-mental Materials, Rovaniemi, Finland.** INFORMATION: Geological Survey of Finland, Geolaboratory/Geoanalysis 2003, PO Box 1237, FIN-
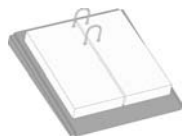
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70211 Kuopio, Finland, Phone: +358 20 550 3670 Fax: +358 20 550 13, E-mail: Lars.Westerberg@gsf.fi Web: http://www.gsf.fi/geoanalysis 2003/

■ July 12-18, 2003, **6ᵗʰ International Conference on Acid Rock Drainage (ICARD),** Cairns, Australia; INFORMA-TION: Clive Bell, c.bell@mailbox.uq.edu.au or website http://www.ausimm.com.au/events/event_writeups/icard.asp

■ August 29 - September 3, 2003, **21ˢᵗ International Geochemical Symposium (IGES), North Atlantic Minerals Symposium (NAMS),** Dublin, Ireland. Information: The Secretary LOC - Eibhlin Doyle (e-mail eibhlindoyle@gsi.ie), http://www.conferencepartners.ie/igesandnams2003.

■ August 31-September 4, 2003, **Emerging Concepts in Organic Petrology and Geochemistry,** The Banff Centre, Banff, Alberta, Canada , by the Canadian Society for Coal Science and Organic Petrology (CSCOP) & The Society for Organic Petrology (TSOP). (Dr. Martin Fowler, Geological Survey of Canada, 3303-33rd St. NW, Calgary, Alberta T2L 2A7 Canada, Phone: 403-292-7038 FAX: 403-292-7159 EMail: Mfowler@nrcan.gc.ca Web: http://www.cscop-tsop2002.com)

■ September 7-11, 2003 **6th International Symposium on Environmental Geochemistry (ISEG)**, Edinburgh, Scotland (Janet Beard, In Conference Ltd. 10b Broughton Street Lane, Edinburgh EH1 3LY, Scotland, UK, Phone: 44-0-131-556-9245 FAX: 44-0-131-556-9638 EMail: janet@in-conference.org.uk Web: http://www.iseg2003.com)

■ September 13, 2003, **6ᵗʰ Intenational Symposium on environmental geochemistry**, Edinburgh, Scotland. Information: Dr. John G. Farmer, Department of Chemistry, University of Edinburgh, phone +44(0)131 65- 4757. E-mail: J.G.Farmer @ed.ac.uk

■ September 16-18, 2003 **International Conference on Tectonics and Metallogeny of Central and Northeast Asia,** Scientific Hall, Russian Academy of Sciences, Academy Town, Novosibirsk, Russia , by the Russian Academy of Sciences and U.S. Geological Survey. (Alexander A. Obolensky, United Institute of Geology, Russian Academy of Sciences, Novosibirsk, Russia 630090, Phone: 7-3832-33-30-28 FAX: 7-3832-35-27-92 EMail: obolensk@uiggm.nsc.ru Web: www.uiggm.nsc.ru/uiggm/geology/admin/)

■ September 22-26, 2003 **7th International Conference on Gas Geochemistry**, Freiberg University - Conference hall "Alte Mensa", FREIBERG, Sachsen, Germany , by the Freiberg University of Mining and Technology and Saxon Academy of Sciences. (Dr. Jens Heinicke, Saechs. Akademie der Wissenschaften /TU-BAF, B-v-Cotta Str. 4, Phone: +49-3731-392212 FAX: +49-3731-392212 EMail: heinicke@ physik.tu-freiberg.de Web: http://www.copernicus.org/ICGG7)

■ October 5-10, 2003, **The XII International Mineral Processing Congress**, Cape Town, South Africa. Information: www.impc2003.org.za.

■ October 12-15, 2003, **Tailings and Mine Waste '03**, Vail Cascade Resort Vail, Colorado. INFORMATION: Linda Hinshaw, Department of Civil Engineering, Colorado State University, Fort Collins, CO 80523-1372, Telephone: 970-491-6081, FAX: 970-491-3584/7727, Email: lhinshaw@ engr.colostate.edu. Web www.tailings.org.

■ November 2–5, 2003, **Annual Meeting of the Geological Society of America**, Seattle, Washington. INFORMATION: TEL 1-800-472-1988, meetings@geosociety.org.

■ February 23-25, 2004, **2004 SME Annual Meeting and Exhibit,** Denver, CO. INFORMATION: Meetings Department at 800-763-3132 or 303-973-9550.http://www.smenet.org/meetings/

index.cfm

■ June 27-July 2, 2004 **11th International Symposium on Water-Rock Interaction**, Saratoga Springs, New York, USA (Dr. Susan Brantley, Secretary General, Dept. of Geosciences, The Pennsylvania State University, 239 Deike Building, University Park PA USA 16802, Phone: 814-863-1739 FAX: 814-863-8724 Web: http://www.outreach.psu.edu/C&I/WRI/)

■ October 10-15, 2004, **SEG International Exposition & 74th Annual Meeting**, Denver, Colorado, US , by the SEG. (Debbi Hyer, 8801 S. Yale, Tulsa OK 74137, Phone: (918) 497-5500 Email: dhyer@seg.org Web: http://meeting.seg.org)

■ November 7-10, 2004, **Annual Meeting of the Geological Society of America**, Seattle, Washington. INFORMATION: TEL 1-800-472-1988, meetings@geosociety.org.

■ February 28-March 2, 2005, 2005 **SME Annual Meeting and Exhibit,** Denver, CO. INFORMATION: Meetings Department at 800-763-3132 or 303-973-9550. http://www.smenet.org/meetings/calendar/event_calendar.cfm

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This list comprises titles that have appeared in major publications since the compilation in **EXPLORE** Number 118. Journals routinely covered and abbreviations used are as follows: Economic Geology (EG); Geochimica et Cosmochimica Acta (GCA); the USGS Circular (USGS Cir); and Open File Report (USGS OFR); Geological Survey of Canada papers (GSC paper) and Open File Report (GSC OFR); Bulletin of the Canadian Institute of Mining and Metallurgy (CIM Bull.): Transactions of Institute of Mining and Metallurgy, Section B: Applied Earth Sciences (Trans. IMM). Publications less frequently cited are identified in full. Compiled by L. Graham Closs, Department of Geology and Geological Engineering, Colorado School of Mines, Golden, CO 80401-1887, Chairman AEG Bibliography Committee. Please send new references to Dr. Closs, not to **EXPLORE**.

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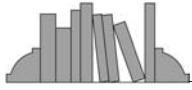
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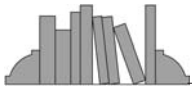
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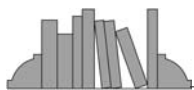
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