

President's Message

As I write this letter I am still catching up on work after a very enjoyable trip to the Association's 23rd Symposium, organized in Oviedo Northern Spain. The meeting was a great success thanks to all the hard work and effort put into the event by Professor Jorge Loredo and his local organizing committee. The success was also testament to the superb set of talks and posters presented at the meeting. In particular I would like to highlight the high quality of research presented in the posters, many of them by research students. Not only was the technical program of a very high standard but the social program was excellent. Eion Cameron seemed greatly pleased to hear bag pipes whilst enjoying a decent Rioja! When asked what he thought he had gained from the meeting, Bob Eppinger had an immediate response that I am sure will resonate with many of us who attended, "WEIGHT!" Yes the meeting certainly introduced us to the excellent wine and seafood of northern Spain and I am sure most people became familiar with the local "sida".

I was also very pleased that Professor Xie Xuejing was able to be present at the meeting to receive the Association's gold medal for his significant contributions to applied geochemistry globally and particularly in China. Elsewhere in this issue we would ask that you spend a few moments filling in the questionnaire related to future meetings of the Association. We are constantly reviewing how the Association seeks to meet the needs of its membership and as times change so too do the needs of our members. But without your feedback it is impossible to be aware of what changes are desired.

Over the last few weeks the need for education in geochemistry has been very apparent from magazine and newspaper articles. Whilst in South Africa this last week two stories stand out in reflecting the general lack of understanding of chemistry. One story, in a travel magazine provoked scare-mongering by environmental groups that a gold mine in central Africa would pollute rivers by "making" chemicals like cyanide, arsenic and mercury. Whilst it may be true for cyanide, actually making natural elements like arsenic or mercury rather contravenes the first law of thermodynamics! In a second story the issue of cadmium contamination of pineapples through imported fertilizer is threatening the South African pineapple industry. A spokesman seemingly unaware of the toxicity of cadmium claimed it could have been worse, like arsenic or another chemical. As far as I know, cadmium is not particularly good for human

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Rob Bowell

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Newsletter for the Association of Applied Geochemists

Multi-Element Determination of Geological Samples using ICP-OES and ICP-MS – Which Technique . . . and When?

Introduction

The determination of major, minor and trace elements in geological samples has always been of paramount importance in the geochemical exploration of elements of economic interest and the evaluation of potential resources. However, these types of samples pose an unusual set of challenges for the analyst, including: the range of analyte concentrations present; the number of elements required per sample; and the amount of samples produced by a typical exploration survey. These factors are further compounded by the presence of a complex range of matrices and the potential for widely different compositions within an analytical batch of samples (Riddle 1993).

Over the past two decades, inductively coupled plasma optical emission spectrometry (ICP-OES) has proven to be a fast and reliable technique for the quantitative measurement of major, minor and trace level elements in geological samples. By simultaneously measuring all of the elements in a sample digest, the speed and cost-effectiveness of analysis are considerably improved. The advent of inductively coupled plasma mass spectrometry (ICP-MS) has further pushed the boundaries of both detection limits and the range of determinable elements available to the exploration geologist. Using either one, or a combination of these powerful techniques, it is now possible to quantitatively measure most elements in the Periodic Table.

An increasingly sophisticated understanding of elemental behaviour and the development of software for handling multi-element arrays, has gone hand in hand with the development of the multi-element capability of the two techniques, leading to the use of a large suite of elements as pathfinders for mineralization. It is important for the geologist to use the appropriate analytical procedure when evaluating exploration survey samples in order to obtain the pertinent information required to identify ore mineralization. Like most commercial analytical laboratories, SGS Minerals Services has developed a comprehensive range of multi-element techniques depending upon the analytical and budgetary requirements of the client. Table 1 provides a brief summary of the typical analytical packages available, although these are often tailored to specific requests. The choice of digestion type has been discussed elsewhere (Chao et al. 1992) and suffice it to say here that initial reconnaissance samples (or soil samples) generally require a partial or weak extraction – the hydrochloric/nitric

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Multi-Element Determination of Geological Samples... *continued from page 1*

Method	ICP-OES	ICP-MS	ICP-OES+ICP-MS
2 Acid Extraction (HNO ₃ and HCl)	32 elements major and trace levels	36 elements Major, trace and ultratrace levels	52 elements Major, trace and ultratrace levels
4 Acid 'near-total' Digestion (HNO ₃ + HCl + HF + HClO ₄)	32 elements Major and trace levels	—	50 elements Major, trace and ultratrace levels
Sodium Peroxide Fusion	28 elements Major and trace levels	—	54 elements Major, trace and ultratrace levels
Lithium Borate Fusion	16 elements Major and trace levels	31 elements Trace and ultratrace levels	47 elements Major, trace and ultratrace levels
MMI-M Selective Weak Extraction	—	45 elements Major, trace and ultratrace levels	—
Sodium Peroxide Fusion Assay	Co, Cu, Ni, Pb & Zn Major components	—	—

Table 1: Summary of typical multi-element exploration packages offered by SGS Minerals Services, showing the range of digestions and instrumental finishes available. 'Ultratrace' is used to differentiate the lower ICP-MS detection limits for particular elements.

acid extraction or partial weak extraction (MMIM) – followed by an elemental scan to provide information concerning anomalies above background levels. Follow-up drilling, delineation, and rock samples are generally analysed using the four-acid digestion (a combination of nitric, hydrochloric, perchloric and hydrofluoric acids) to provide more complete information about composition. Fusion digestions are used to dissolve refractory phases unaffected by acid dissolution, providing a complete multi-element determination of geological samples and high precision assays for ore level concentrations of base and other metals of interest.

Multi-element scans can thus provide a wealth of elemental data in rapid timeframes for the large volumes of samples required by exploration geology. The aim of this paper is to attempt to answer the question of what

technique is appropriate for different types of sample and concentrations of elements of interest. Following a brief introduction to the concepts behind the techniques will be a discussion on how advances in instrumental design have enabled SGS Minerals Services to improve their application to geological analysis in two specific areas. These are the lack of sensitivity of ICP-OES in measuring low concentrations of many trace elements, and the inability to measure high concentrations of elements by ICP-MS. In overcoming these limitations, both ICP-OES and ICP-MS can provide unparalleled analytical data for the geologist at all stages of the exploration process.

Inductively Coupled Plasma (ICP)

The argon plasma is used in atomic spectroscopy as an efficient energy source for the excitation and ionization of atoms. The plasma is initiated through partial ionization of argon by seeding it with electrons. A Radio Frequency generator provides high frequency current to an induction coil and the resultant magnetic field is used to accelerate the electrons. Adding energy to the electrons in this manner is called *inductive coupling*. A chain reaction is set up whereby the accelerated electrons collide with argon atoms, causing ionization and more electrons that collide with other atoms. This reaction results in a stable plasma held within the induction coil by the magnetic field. The plasma has core temperatures of between 8000 and 10000°C, into which samples can be aspirated, producing the required excitation and ionization of elements in solution.

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

ICP-OES utilizes the ICP as an energy source to excite atoms and ions, which, as they return to ground

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state, emit light at characteristic wavelengths that can be measured using an appropriate detector. Modern instruments differentiate the light into its component wavelengths using an Echelle diffraction grating with efficient light throughput. The diffracted light from the grating is measured using a Charge-Coupled-Device (CCD) detector. By comparing the intensities of the sample with a standard containing known quantities of each element of interest, the concentrations of those elements in the sample can be determined.

The main advantages of the ICP-OES system for the analysis of geological samples include the ability to measure many elements from a single sample digest, in order to provide maximum information about the sample, and a variety of strong acid and fusion matrices can be determined directly, since only light enters the instrument. The large number of spectral emission lines available enables analytically useful wavelengths to be found for most elements. In addition, both major (percentage levels) and trace (parts per million levels) elements can be determined from the same solution, without the need for serial dilutions, which take more time and can impact analytical precision. Finally, the robustness of the technique makes it a powerful tool in the analysis of large numbers of samples.

The limitations of analyzing geological samples by ICP-OES methodologies include: potential compromises

in analytical data due to the digestion procedure involved; the enormous number of emission lines and the nature of the plasma source can lead to significant interferences for some elements; and the sensitivity of the instrument tends to be limited for some elements, (e.g. Sn, W and Sb) due to emission lines in the low UV wavelengths that can be absorbed in the air before entering the spectrometer. Whilst the first two limitations can be overcome by carefully choosing the appropriate digestion (see Table 1) and analytical wavelengths, plus interference correction where necessary, the third problem can be improved by using a dual-view approach.

Lower Detection Limits for ICP-OES – The Dual View Approach

Traditionally, plasmas have been viewed from the side (or *radially*), analogous to atomic absorption spectroscopy where viewing occurs along the line of the burner.

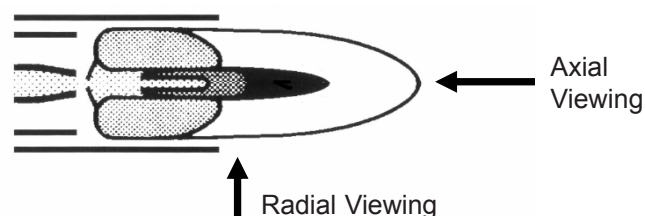


Figure 1. Dual view configuration of the ICP-OES. Axial and dual view instruments have horizontally orientated plasmas and view both across and down the plasma. Radial instruments have a vertically orientated plasma and view through the analytical zone only.

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Multi-Element Determination of Geological Samples... *continued from page 3*

However, the plasma also can be viewed end-on, or *axially* (Fig. 1). The advantage of axial viewing is that more of the light enters the instrument, resulting in greater intensities. Even though backgrounds are also increased and there are potential problems with spectral re-absorption in the plasma tail for easily ionizable elements, axial measurements tend to give sensitivities that are an order of magnitude higher for many trace elements of geological significance. An ICP-OES with a combination of axial and radial viewing is known as a dual view instrument (Nölte 2003).

SGS Minerals Services utilizes the dual view approach (Optima 5300DV ICP-OES, PerkinElmer Life and Analytical Sciences, USA) for the multi-element analyses detailed in Table 1. The radial view is used for high concentrations of elements, and the axial view for trace elements. Thus, measurement of a wide concentration of elements is achieved in a single determination and has the

Element	Radial Detection Limit	Axial Detection Limit
As	7.2	2.7
Bi	5.8	0.5
Co	0.7	0.1
Cr	1.2	0.3
Cu	0.5	0.2
Hg	2.6	0.5
Mo	1	0.2
Ni	1.8	0.2
Pb	8.5	0.6
Sb	4	0.8
Sn	12.1	0.3
W	2	0.6
Zn	4.4	0.1

Table 2: Comparison of two-acid extraction detection limits for ICP-OES using radial and axial modes. Results show the increased sensitivity of the axial mode for many trace elements. Detection limits are determined from three times the standard deviation (3σ) of seven method blank solutions. Detection limits are based upon a 0.25 g sample digestion, diluted to a final volume of 20 mL. All results are in ppm.

advantage of providing lower detection limits for many elements.

Table 2 shows a comparison of detection limits determined using both radial and axial instruments for a two-acid extraction digest. These limits are based upon a 0.25 g sample digestion diluted to a final volume of 20 mL. As can be seen, the axial view produces significant

	Axial		
	ICP-OES	ICP-MS	Difference
Sample-1	15	13.3	2
Sample-2	14	14.8	1
Sample-3	13	11.3	1
Sample-4	15	13.5	1
Sample-5	13	12.2	1
Sample-6	15	14.8	1
Sample-7	15	14.0	1
Sample-8	26	25.2	1
Sample-9	9	9.1	<1
Sample-10	4	2.8	1
Sample-11	9	7.6	1
Sample-12	9	6.7	2
Sample-13	13	11.0	2
Sample-14	10	9.2	1
Sample-15	8	7.8	1
Sample-16	17	16.4	1
Sample-17	15	15.2	<1
Sample-18	16	15.7	<1
Sample-19	14	14.2	<1
Sample-20	22	21.5	<1

Table 3: Comparison of axial ICP-OES recoveries for low-level Pb containing samples with ICP-MS using a four acid digestion. All results are within ± 2 ppm, showing the ability of the axial mode to achieve acceptable precision at low concentration levels. ICP-OES detection limit is 2 ppm for this analysis. Results are based upon a 0.2 g sample digestion, diluted to a final volume of 12 mL. All results in ppm.

improvements for Hg, Pb, Sn, Sb, and other important trace elements compared with the radial approach.

Table 3 shows recoveries for a series of samples containing low concentrations of Pb, determined by axial ICP-OES and ICP-MS, following a multi-acid digestion of 0.2 g

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Multi-Element Determination of Geological Samples... *continued from page 4*

sample diluted to a final volume of 12 mL. The data confirm that acceptable precision (± 2 ppm) can be obtained, even at less than 10 ppm Pb concentration. Table 4 illustrates the recoveries for twelve reference standards analysed using a two-acid extraction (0.25 g sample digested to a final volume of 20 mL) and shows

Standard	Axial ICPOES	Expected Value	% Recovery
LKSD-1	82	84	97
LKSD-2	38	40	95
LKSD-3	25	26	96
LKSD-4	91	93	98
STSD-1	35	34	102
STSD-2	65	66	99
STSD-3	40	39	103
STSD-4	13	13	103
TILL-1	14	12	118
TILL-2	22	21	107
TILL-3	17	16	106
TILL-4	37	36	102

Table 4: Comparison of Pb recoveries for a two acid extraction of reference standards by axial ICP-OES. Acceptable recoveries are obtained for all standards (Lynch 1990 and Lynch 1996). Results are from a 0.25 g sample digestion, diluted to a final volume of 20 mL. All results are averages of five digestions. All results are in ppm.

that accurate values can be measured for Pb at less than 20 ppm in the sample using this technique.

Thus, ICP-OES can be utilized to measure not only high concentrations of elements, but also trace levels by using the dual view instrument. The flexibility of the dual view instrument is shown in other applications as well. For instance, in the determination of low level precious metals by fire-assay, the axial mode provides a detection limit of 1 ppb for Au, compared with the 5 ppb detection limit achievable by radial ICP-OES and atomic absorption spectrometry. Also, for assay quality analysis (<5% for both precision of duplicates and accuracy) on higher concentration samples, the radial mode is used in conjunction with element-specific certified reference materials (CRMs). However, as powerful as the dual view ICP-OES technique has become, there are many cases where even lower detection limits are required for a range of elements and it is then that ICP-MS becomes the technique of choice.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

ICP-MS utilizes the plasma as an efficient heat source for the formation of positive ions. A small portion of the ion beam enters the instrument, is focused using a lens system, and then passed into a quadrupole mass spectrometer, where ions are separated based on their mass to charge ratio. An electron multiplier is used to detect the ions as they pass out of the mass spectrometer.

As with ICP-OES, comparing intensities from known standard solutions to the unknown sample allows the concentration of the analyte to be determined. The instrument measures elements sequentially by scanning up and down the mass range. Hence, the more analytes that are required to be measured, the longer the analysis will take.

The advantages of ICP-MS include: the sensitivity of the technique allows for the determination of many elements in the sub parts per million range (e.g. rare earth elements); the multi-element capabilities of the instrument allow for the analysis of a wide range of elements at different concentration levels in one pass; and the reliability of contemporary instrumentation makes it a powerful tool in the analysis of the large volumes of samples generated in geochemical analysis.

The main limitations of ICP-MS are: the technique cannot directly determine elements in solutions containing high levels of dissolved solids, such as rock digests; the high temperature of the plasma can cause polyatomic interferences (such as argides and oxides of other elements in the sample digest); and the increased sensitivity of the instrument (typically >1000 times that of ICP-OES) makes it inappropriate for measuring high analyte concentrations. Whilst the first two disadvantages can be overcome by diluting the sample prior to analysis, choosing the appropriate isotope and where necessary performing interference corrections, the measurement of increased ion beam intensities from high element concentrations requires a different approach.

ICP-MS was initially developed to measure trace amounts of elements in solutions. However, the proliferation of the technique and its widespread use in many analytical areas, including geological samples, created a requirement for increased dynamic range to allow for the determination of different concentration levels without the need for further dilution. The introduction of the two stage detector enabled the expansion of the use of ICP-MS to measure high concentrations whilst retaining its low detection limit capabilities.

Increased Dynamic Range for ICP-MS – The Dual Stage Detector

Commercially available ICP-MS instruments (SGS Minerals Services uses the Elan 9000, PerkinElmer SCIEX, Canada) contain a two-stage, or dual detector, for the measurement of low (sub parts per billion) and high (parts per million) solution concentrations in a single pass. High intensity ion signals emanating from the quadrupole mass spectrometer are measured by the *analogue* stage, whereas low concentrations pass into the electron multiplier for amplification and measurement – the *pulse* stage (Fig. 2). It is this amplification that enables the ICP-MS to detect very low concentrations of elements in solution.

By measuring solutions containing significant concentrations of an analyte using both stages of the detector, a cross calibration coefficient can be determined (Fig. 2). This determination can be repeated for all

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Multi-Element Determination of Geological Samples... continued from page 5

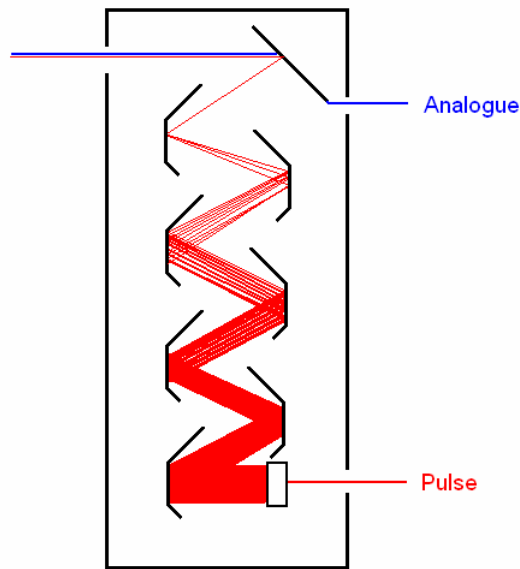


Figure 2. The Dual Stage Detector. A high concentration of the element (in blue) is detected by the analogue stage, whereas the low concentration signal enters the electron multiplier (red). By measuring appropriate concentrations using both stages of the detector, a graph can be drawn and the coefficient between the two stages calculated. This can be used to convert the analogue result to the pulse calibration, creating an extended linear range.

elements of interest to create a dual detector calibration. Due to the linear nature of the detector, sample digests containing a wide range of concentrations can now be determined in a single pass, without the need for further dilution. Table 5 illustrates the measurement of high levels of Pb in a four-acid digestion of geological samples. The results are from a 0.2 g sample digestion, diluted to 12

Sample	ICPMS	ICP	%Recovery	Expected
Unknown-1	S	38200	n/a	
Unknown-2	12270	12500	98	
Unknown-3	325	320	101	
Unknown-4	12600	12700	99	
Unknown-5	2440	2270	107	
Unknown-6	4090	3960	103	
Unknown-7	1660	1640	101	
Unknown-8	36	35	101	
Unknown-9	3660	3390	108	
Unknown-10	4770	4470	107	
SRM2710	5480	5385	99*	5532
SRM2711	1180	1170	102*	1162

Table 5: Comparison of Pb results using ICP-MS and ICP-OES for a range of unknown samples and two reference twenty times was made before analysis. S = signal saturated and could not be measured. Reference Standard results are averages from five replicate digestions. * Based on ICPMS comparison to reference values (Govindaraju 1994). All results in ppm.

mL for ICP-OES, and then diluted a further twenty times for analysis by ICPMS. As can be seen, there are acceptable correlations between the ICP-MS, ICP-OES and expected values for the standards. The ICP-MS results are within 10% of the expected value for both the reference standards and the results obtained by ICP-OES for unknown samples. The calibration for this determination has a maximum point of 60ppm in the sample, enabling the quantitative determination of Pb up to over 200 times higher, with saturation only occurring at much greater than 10 000ppm (or 1%). This confirms that ICP-MS can be used to measure not only trace levels of elements, but also significantly higher concentrations as well. A two-acid partial extraction can be measured by ICP-MS for a range of major components (such as Fe, Ca, Al, Mg), percentage levels of base and other metals, as well as trace levels of other elements. However, ICP-OES is still preferred for major component analysis of geological samples in multi-acid and fusion digests, due to its ability to measure the considerably higher amounts of

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Multi-Element Determination of Geological Samples... *continued from page 6*

major elements extracted into solution using these digestion techniques.

Partial weak extractions, such as Mobile Metal Ion Geochemistry (MMI), have become a significant tool in geochemical exploration over the past decade (Mann et al. 1998). Earlier versions of these leaches measured four or five indicator elements at low concentrations using ICP-MS in order to determine locations of buried gold and base metal mineralization. The recent introduction of MMI-M, a pH-neutral extraction that aids in the identification of polymetallic targets and multi-element associations for specific styles of mineralization, has placed the emphasis on ICP-MS to deliver quantitative results for a much larger range of elements. As well as measuring base and precious metals, the package has been used in the determination of rare earth elements (significant in the geochemistry of nickel sulphide deposits) and major elements such as Fe, Ca and Mg. In total, ICP-MS can be used to measure over 45 elements present in concentrations ranging from sub parts per billion (e.g. Au), through to hundreds of parts per million (e.g. Mg and Ca), further illustrating the major advantage of the dual detector approach to analysis by ICP-MS.

Conclusion

There is an increasing need for high precision analyses of geological samples for a greater range of elements and concentrations. Modern exploration methods involve statistical analysis of data from a set of elements in order to assess possible ore deposits. The challenge for analytical techniques is to evolve to meet these ever-changing demands. These can include: the addition of more elements, lower detection limits, and increased ranges of concentrations. ICP-OES and ICP-MS have been shown to provide reliable results for geological samples and the constant improvement in both techniques, such as use of the dual view for ICP-OES to lower detection limits, and the dual detector in ICP-MS to increase dynamic ranges only strengthens their applicability in a wide variety of situations for both now and the future.

The answer to the question of which technique to use is always contentious, as a number of factors have to be taken into consideration that may be unique to a particular situation. However, the following are some general guidelines:

- For initial reconnaissance work using a weak extraction, use either ICP-OES or ICP-MS to obtain information about a set of elements. For two-acid extraction, use a combination of the two techniques to determine additional elements (such as S, Hg, REE, Th and U).
- For drill cores and rock samples, use either a multi-acid or fusion digestion, followed by ICP-OES for major components and trace elements. Combine this with ICPMS to determine additional elements (such as REE, Rb, Nb, Th, U).

- For individual elements required in high-grade samples (percentage levels), a specialized assay technique is used, combining a larger weight sodium peroxide fusion with ICP-OES determination, and utilizing appropriate element-specific certified reference standards. Note: classical chemistry is used for the settlement quality analyses of payable elements in very high-grade materials

The use of both ICP-OES and ICP-MS provides the most comprehensive sample analysis for the greatest range of elements in exploration situations. The two techniques have become synonymous with geological sample analysis and their unrivalled ability to produce high quality data in a timely and cost-effective manner makes them a powerful tool for the exploration industry.

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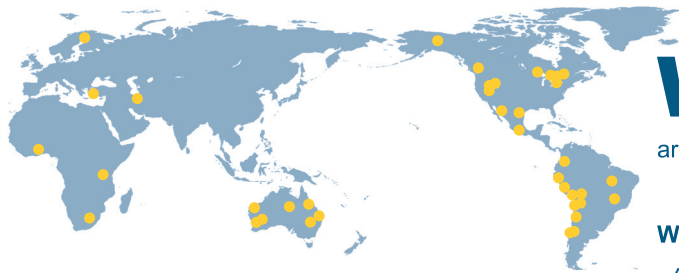
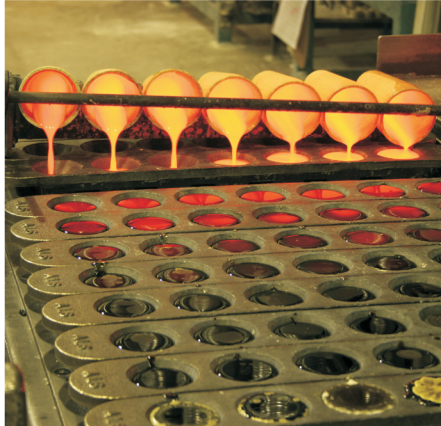


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What is the preferred upper end of concentration for base metals and pathfinder elements in ICP-MS? When should the geochemist switch to routine ICP-AES?

Commercial ICP-MS multi-element packages are now widely used as an exploration tool, providing pathfinder elements at trace concentration levels. The detection limits achievable by ICP-MS for low level concentrations of elements and elements with low crustal abundances have proved their worth in determining new targets and increased information about geological sites.

The combination of multi-element analytical packages and computing power to manage data from for multiple elements over a project has made the collection of these data common place. The range of concentrations in geochemical analysis that needs to be addressed is wide. Major elements are present at % levels. Trace elements such as Se and Te have crustal abundances in the sub-ppm level and require the sensitivity of ICP-MS. Important trace element indicator elements such as arsenic can range from a few ppm to hundreds of ppm. Levels of other elements may cover wide ranges: Cu at sub-ppm in a selective leach, 10's of ppm in grass roots exploration and % levels for on-site and target drilling. These are challenging demands but are being met by current analytical options.

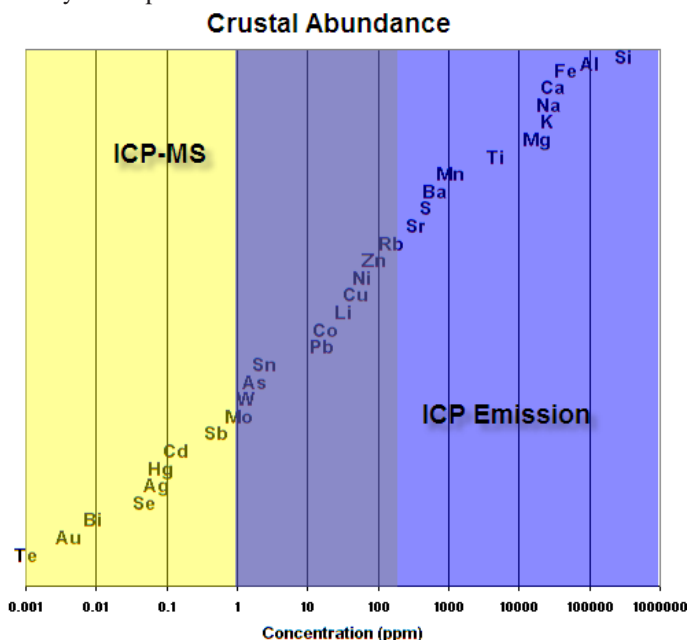


Figure 1: Crustal abundances of various elements versus analytical ranges for ICP-MS and ICP-AES.

Range of ICP-MS and ICP Emission Instruments

Most samples are now analyzed by either ICP emission spectrometry or ICP-MS, or a combination of these two instruments. The range of ICP-MS for most elements is 10 ppb to 250 ppm (in the sample itself). Extension of this upper limit can be done by using the

Analog detector on the mass spectrometer. ICP emission range is from ppm to % levels. The extended range is due to the ability to access emission lines of different sensitivities that allow extension of linear range for any element. It is common within one project to have requirements for different elements that cross all these ranges.

The need for a combination of instruments to cover the range in geological samples is illustrated in Figure 1. Typical crustal abundances for a number of elements are plotted in increasing order against the instrumental ranges for ICP-MS and ICP-AES. ICP-MS optimum analytical range is shown in yellow and ICP-AES optimum range in blue. Gray represents where the two techniques overlap. The instrumental ranges are estimated based on a 1 g to 25 ml digestion.

Best Methods to Provide Results across the Ranges

The standard ICP-MS packages at ALS Chemex have always included both an ICP-AES *and* an ICP-MS ion determination on each sample to provide optimum precision and accuracy for each element over a wide concentration range. Trace elements and low level concentrations are reported from the ICP-MS determination. Major element concentrations are reported from the ICP-AES determination. Elements with concentrations in the mid-range between the techniques are measured by both procedures. Results are cross-checked for correctness to ensure there are not interferences in one technique and to ensure linearity of the reporting between the two techniques. Within a submission, an element such as arsenic or copper might be reported from the ICP-MS, from the ICP-AES, or from a comparison of both instruments at the mid-range. This ensures that from the two determinations, the data reported are the best available between the two instruments. This was established at the early stages of implementation at ALS Chemex owing to concern for quality data for each element reported, and recognition that some elements at certain ranges are better reported from ICP-AES rather than ICP-MS, whereas other elements can only be measured by ICP-MS.

The procedures and calculations for the instruments and reporting protocols are the same across the company globally. The global protocol includes primary isotope and emission line selection for each elements, secondary

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Shea Clark Smith

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What is the preferred upper end of concentration for base metals...

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choices, rules for when secondary choices are used, switching points between techniques, plus calibration protocols and levels. The software for calculation of results from instrument data, and review and approval of data are centrally controlled. In addition to this, all choices made by the analysts during the data review process are captured in an audit trail and are available. Within the procedure, a number of the inserted Quality Control reference standards are used by all laboratories globally. Review of control charts and long-term trends are monitored to ensure consistency amongst all the labs. The outcome is that comparable results are obtained by these methods from all our sites.

Over Limits – when to switch to another method

When the concentration of an element exceeds the range of the procedure, another method is used to determine the element abundance. For instance, fire assay with an AA finish may indicate high-grade samples which are repeated by a fire assay Au with gravimetric finish. We might determine the Cu by a 'geochem'

method up to 1% (with a precision of +/- 10%) and then, at the 1% level, use an assay procedure with an appropriate digestion and finish to determine the concentrations with better precision (i.e. +/- 5%).

The concept of range is important. Range for a procedure is the concentration upper and lower limits which meet the precision, accuracy and performance requirements for the method. Range includes not just the analytical finish but also the appropriateness of the digestion, the volume controls and accuracy, calibration checks and precision of method control.

When a sample exceeds that standard calibration for a method, there are choices to be made in order to determine the content. Oftentimes, a lab will be asked to simply 'dilute' the sample and report a value. In most cases, this is not appropriate. The digestion for the original determination, typically 0.5 g to 25 mL in a test-tube, is not likely appropriate for a highly mineralized sample. The volume control in the test-tube volume (+/- 3%), added to the dilution error (too often done as 1 ml with an Eppendorf pipette plus 9 ml with an autodispenser) now has a volume error of greater than 5%. When this is coupled with other errors in the procedure, the error limits of the 'diluted' value can make the results misleading and not of value.

When an element exceeds the limits of an ICP-MS range, the sample can be determined directly on the ICP emission instrument without dilution. If the sample exceeds the calibration range of the primary emission line,

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What is the preferred upper end of concentration for base metals...

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in many cases, there are secondary emission lines that can be used to extend the calibration without resorting to dilution. This is a case of using the 'right' instrument to analyze the sample. However, at a certain level, all are better served by using the 'right' method – i.e. a specific digestion with volumetric flasks – to provide an appropriate result that is fit for purpose.

In our opinion, it is not responsible to use dilution and a single digestion and instrumental procedure cover the range for sub-ppm up to tens of percent for one element.

Under Limits

In many cases, the detection limits from the ICP-MS packages are not required and, for most of the samples in the project or for most elements, ICP emission detection limits are adequate. This cost-effective choice can be made. Generally though there are a few elements or a few samples where the concentrations are below emission detection limits and an ICP-MS determination would be of value. These can be managed by "Under Limits". Just as you might specify, 'if Cu > 5000 ppm, do an assay', the specification of 'if Bi < 20 ppm, use ICP-MS' can be requested and automatically managed in the system.

A project can have ICP-MS done for a few key elements on every sample, or it can be done by having ICP-MS done only when the concentration for the element is below a trigger level.

ICP-MS to Determine Trace Elements in Ore-Grade Samples and Concentrates

Trace element characterization of a highly mineralized sample or concentrate can add value to a project. ALS Chemex has developed ICP-MS packages for concentrates and high-grade samples. It is not appropriate to have these samples analyzed by the routine exploration package. These packages use slightly higher sample/volume ratios but include a number of other features. In the case of a copper concentrate, high-concentration copper samples are run to correct for any interferences on other elements that may not be corrected for during routine determinations. Separate equipment is used to prevent any carry-over contamination to other work. The commodity element will not be reported by this procedure but trace characterization can be very useful.

Summary

Knowledge of the nature of your project and expected concentration ranges will allow you, in discussion with the analysts at the laboratory, to select the best analytical protocols for your project. This may include a number of methods, for instance both ICP-MS and ICP emission spectrometry, and selected 'over limits' and 'under limits' to give data quality that is suitable for the needs of the project. The procedures established at ALS Chemex are designed to provide quality determinations

for elements at a variety of ranges, with the system set up to automatically provide the optimal procedures if the initial determination is not optimal. The geochemist is not required to request these because they are part of the ALS system. However, collaboration between the geochemist and analyst at the start of the project is always preferable to establish the optimum analytical methods to match the sample concentrations, matrix, and mineralogy.

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Preparation of Indicator Minerals for Electron Microprobe Analysis

Indicator minerals are used in the exploration for a variety of mineral deposits as each mineral type can uniquely identify a particular type of deposit or rock lithology. Generally, indicator minerals have a relatively high density, are silt to sand sized and are physically and chemically resistant (Averill 2001; McClenaghan 2005). These properties help to isolate the indicators in mineral concentrates, created through a variety of size, gravity and magnetic separation techniques so they can be selected with relative ease using a binocular microscope. Examples include scheelite and wolframite as indicators for tungsten deposits, cinnabar for gold or mercury deposits and cassiterite as an indicator for tin deposits (McClenaghan 2005).

Section preparation can be applied to any type of indicator mineral or to any type of sample that is limited in the number of grains for analysis or in grain size. For example, kimberlite indicator minerals are invaluable tools for diamond exploration. As few as five Cr-pyrope grains in a single 10 kg till sample may provide sufficient evidence that further sampling in a region is warranted as they indicate the presence of a kimberlite (McClenaghan et al. 2000; Averill 2001). However, it is the mineral chemistry of that grain that is most revealing, as it can provide information about the upper mantle environment where this mineral formed and the potential for association with a diamond-bearing host (Fipke et al., 1995; Schulze, 2005; McClenaghan and Kjarsgaard, 2007).

Mineral chemistry data obtained by electron microprobe analysis of selected indicator minerals are used to classify indicator minerals and to determine the

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Preparation of Indicator Minerals for Electron Microprobe Analysis...

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diamond prospectivity of an area (McClenaghan & Kjarsgaard 2007). Pyrope garnets are sorted according to their composition; G10 garnets contain less Ca than G9 garnets, with G10 garnets being generally the target in diamond exploration (Fipke et al. 1995; Averill, 2001; Schulze 2005). This garnet classification scheme is not always possible solely using information from a binocular microscope examination. As well, minerals such as crustal garnet and eclogitic garnet are difficult to distinguish optically and can only be distinguished from each other with confidence using electron microprobe analysis. The Fe content of the minerals, determined by electron microprobe analysis, is used to classify these garnets, with crustal garnets typically containing more Fe (Schulze 2005). Similarly, kimberlitic ilmenite is distinguished chemically from crustal ilmenite as kimberlitic ilmenite will contain a minimum of 4% MgO (McClenaghan & Kjarsgaard 2007).

In generating high quality microanalytical data using the electron microprobe, a flat highly polished surface on each mineral grain is a key pre-requisite. Surface roughness impacts data quality in two ways. Firstly, the geometry of x-ray measurement in the wavelength dispersive x-ray spectrometer requires the sample surface, the refracting crystal and the detector to lie on a single plane known as the Rowland circle. Departures from this plane cause defocusing of the characteristic x-rays at the detector resulting in a loss of precision. This loss is less of a concern where the spectrometers are mounted at an inclined plane to the specimen holder, but most electron microprobes are equipped with vertically mounted spectrometers. The second problem caused by surface roughness is that of variable absorption in the x-ray path to the spectrometer. This problem has more of an impact on lower energy x-rays which are characteristic of light elements such as Na, Mg, Al, and Si and also results in a loss of precision. To ensure greater precision and accuracy of microprobe data, the grains must be properly prepared in polished sections with a mirror like finish. The most common problems encountered in preparing indicator minerals for microprobe analysis is the variable hardness among indicator minerals and occasionally, the indicator minerals being quite small. The following section illustrates some ways of overcoming these problems.

How are indicator minerals prepared for electron microprobe analysis?

Two types of sections are commonly used to mount indicator minerals for electron microprobe analysis: 1) bird's eye polished sections and 2) polished grain mounts. Bird's eye polished sections, so-named for their appearance, are used for very fine indicator minerals (<0.25 mm grains). Polished grain mounts are the more common method for mounting coarse indicator minerals (>0.25 mm grains). Typically sections can be 25 mm or 30 mm in diameter and grains are exposed across the entire surface of the section, however polished sections of any diameter or thickness can

be produced to accommodate any microprobe.

- 1) A bird's eye section is used if there is at least 0.5 g of sample. For example, in mineralogical studies of gold ore, the gold is separated from the gangue using a series of gravity separation techniques (Zhou et al. 2004). This procedure will result in a very small, often fine-grained, concentrate. Bird's eye polished sections are often produced from these concentrates for mineralogical examination. When used for indicator mineral analysis, each bird's eye polished section will only contain one indicator mineral species from one sample. The section is made from a 25 mm diameter resin plug. A small diameter, shallow indent is created in the centre of the plug using a lathe (Fig. 1). This indent can be of any diameter, and will depend on the

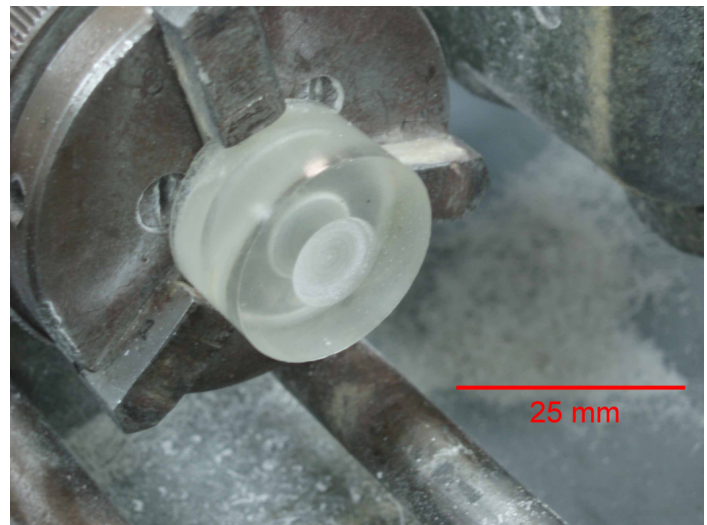


Figure 1: A 25 mm diameter resin plug is used for preparation of a bird's eye polished section. A lathe is used to make a small diameter divot in the surface of the plug.

amount of material available for mounting. The indicator mineral grains are placed in this divot and a small amount of resin is added. The sample is mixed with resin in the divot using a toothpick and the section is placed in a pressure vessel to cure. Once hardened, the section is prepared for polishing, beginning with light grinding to level out the surface and expose the grains. After grinding, the sections are finished on a polishing machine where progressively finer diamond pastes are used to ensure a mirror finish, free of scratches (Fig. 2). In preparing a bird's eye polished section, some indicator mineral grains will be lost during grinding and polishing and some grains will not be exposed at the surface for electron microprobe analysis.

- 2) The more common specimen mounts used for indicator minerals are polished grain mounts. This method is used for grains >0.25 mm in size. Different types of indicator minerals from many different samples are placed on the same mount, making organization during mounting essential. For kimberlite indicator minerals, oxides are separated from silicates as different analytical routines are utilized by the microprobe software for

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Preparation of Indicator Minerals for Electron Microprobe Analysis...

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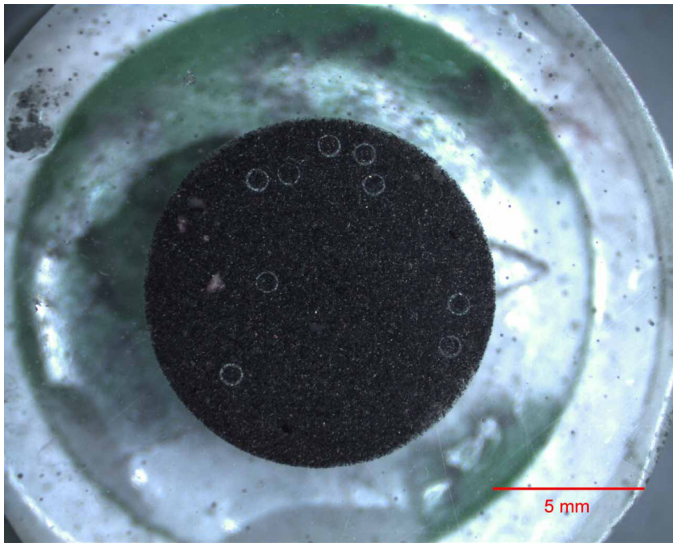


Figure 2: Completed bird's eye polished section prepared from a heavy mineral concentrate. Grains of interest have been scribed with a circle.

these mineral groups. Grains are separated by hardness as well as by size, with a separate mount for 0.25 to 0.5 mm, 0.5 mm to 1.0 mm and 1.0 to 2.0 mm sized grains. This separation helps prevent grain loss due to plucking during the final grinding and polishing process. Following indicator mineral selection, recovered grains are placed in vials or on grain cards. Each grain is manually placed on a glass slide, covered with transparent double-sided tape and their location is recorded on a spreadsheet for easy reference. The slide is placed over a fine grid to ensure the grains are arranged in straight lines (Fig. 3). Binary grains can be specifically arranged so that both phases will be exposed

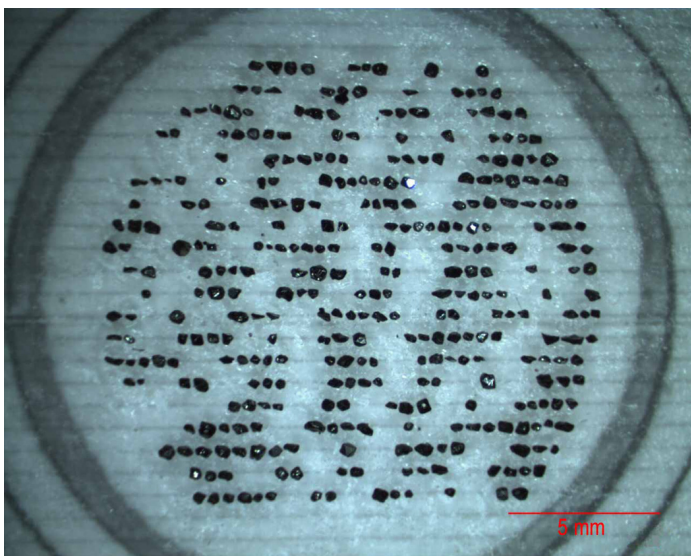


Figure 3: For a polished grain mount, grains are initially arranged right to left on a glass slide. The completed section will be the mirror image, with grains running from left to right.

for electron microprobe analysis. Grains are placed in each row moving from right to left, keeping in mind that the tape represents the surface of the polished section. The finished section will be the mirror image, with grains running from left to right. A significant space is left between grains from different samples. When all grains have been mounted on the slide, a 25 mm diameter mold is placed on the slide. Resin is poured into the mold; low viscosity resin is used to ensure all grains are completely covered. The section is placed in a pressure vessel to cure. When the resin has hardened, the mold is removed and the glass slide is heated so the resin mount can be removed. The surface of the section is examined for holes. Pits must be backfilled before grinding to prevent grains from being plucked from the section. Minerals in the mount are carefully exposed by grinding prior to polishing. Similar to bird's eye polished sections, a polishing machine is used with progressively finer polishing compounds to produce a smooth surface (Fig. 4). The section is checked often during grinding and polishing using a reflected light microscope to prevent grain loss. The under side of the grain mount can also be polished so the general morphology of the minerals can be observed.

Why is careful section preparation important?

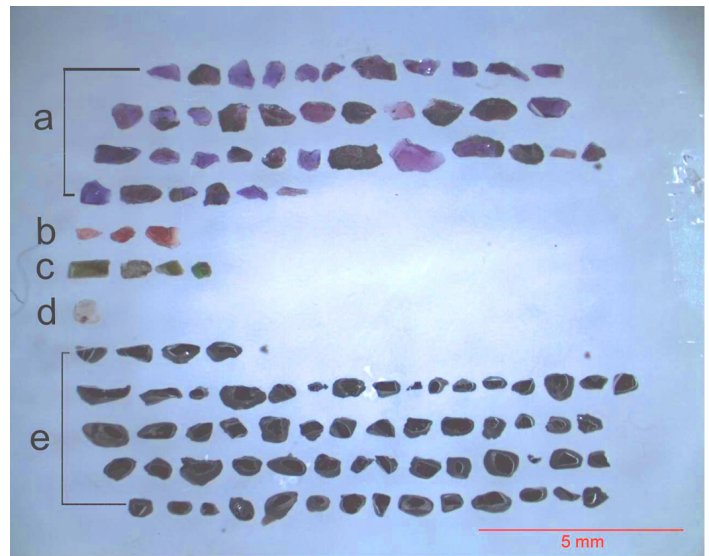


Figure 4: Completed 25 mm diameter polished grain mount. Labeled grains are: a. pyrope garnet; b. eclogitic garnet; c. chrome diopside; d. olivine; e. oxides.

A great deal of effort and expense goes into an indicator mineral sampling program and while section preparation may seem a very small part of the process, it is nonetheless very important, as the loss or misidentification of a few grains could mean that considerable funds will have been wasted and anomalies missed. Organization is essential to the successful preparation of indicator minerals for electron microprobe analysis and it is not a task that should be completed by someone who lacks this skill. Care must be taken at all steps in the preparation process to ensure grains are not mislabeled or lost during polishing or the entire

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Preparation of Indicator Minerals for Electron Microprobe Analysis...

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exploration program may be set back.

Improper section preparation may also yield inaccurate microprobe results which may in turn lead to erroneous exploration decisions. For example, in diamond exploration, thermobarometric data calculated from inaccurate electron microprobe data might indicate a favorable geotherm for diamond preservation, plotting just inside the diamond stability field when in reality, the grains are not. Further exploration including drilling and microdiamond analysis may be undertaken when, in fact, accurate data may not have warranted additional work. Conversely, properties may be relinquished because electron microprobe analysis may incorrectly suggest that the mineral chemistry of grains recovered from the mineral concentrates is not sufficiently indicative of diamond content. Both situations are possible if the electron microprobe data are obtained from the surface of a poorly prepared grain mount. Similarly, the loss or misidentification of a single indicator mineral during section preparation can have very detrimental effects.

While section preparation is a simple step in part of a complex exploration program, it must be carried out properly and consistently over time to ensure reliable geochemical data are obtained from each indicator mineral.

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President's Message... continued from page 1

health and certainly no better than arsenic. These examples illustrate the need for education across society in some basic principles of chemistry.

Within the geological community there is still a need for education in geochemistry as well. A short column in a financial magazine in the last month highlighted problems associated with over estimation of mineral resources for a gold company due to "poor analysis". Presumably such a statement would potentially have a negative impact on share price and could have easily been avoided had a qualified geochemist been involved in the acquisition and verification of the geochemical data. Perhaps here we need a greater push to improve the awareness of financial institutes in recognizing those qualified as geochemists as being essential in confirming geochemical results published in support of resource estimates. Do we need to seek greater recognition through other institutes as a separate professional entity? I would welcome your thoughts on the matter.

The Association continues in the development of the Distinguished Applied Geochemists Fund and hope we are able to finalize arrangements by the end of the year. In order to establish a self-sustaining fund we will have to make some minor changes to our Articles of Association and hope to publish these in **EXPLORE** in the near future for all members to review.

Rob Bowell

President, Association of Applied Geochemists



AAG Gold Medal Award Citation

Professor Xie is truly the father of exploration geochemistry in China. He conducted the first geochemical exploration in that country in 1951 in Anhui Province with the systematic sampling and chemical analysis of rocks, soils, and stream sediments in the search for copper mineralization. He has also been instrumental in developing techniques to aid in the search for buried ore deposits. This began in the late 1950s with the study of element zonation in primary halos during which he developed methods that were widely applied in China. These methods successfully identified extensions to some known deposits. For example, the Qingchengzi lead-zinc deposit in Liaoing Province, northeastern China, was discovered in the 1940s and closed in the 1960s. Prof. Xie and his colleagues identified a large new ore body at depth and the mine is once again in operation. This discovery is described in Chinese-language textbooks on geochemical exploration. His work on exploration for concealed deposits has continued with his supervision of the more recent development of two techniques that have been used for wide-spaced geochemical mapping in concealed terrains. One method, called Nanoscale Metals in EarthGas (NAMEG), involves analysis of soil gases collected onto polyurethane foam. The second method, called Mobile Forms of Metals in Overburden (MOMEQ), involves sequential extractions of overburden material to look at 1) ions from water-soluble salts, 2) metals sorbed on clay minerals, 3) metals bound to organic matter, and 4) metals absorbed by oxides.

One of Prof. Xie's most remarkable achievements has been his planning and technical supervision of China's Regional Geochemistry – National Reconnaissance (RGNR) Project. Over the past 28 years, this national-scale geochemical mapping effort has covered more than six million square kilometers of China. About six million samples (primarily stream sediments) have been collected throughout the country and approximately 1.5 million composite samples analyzed. His work has led to China's having the most remarkable national-scale geochemical database in the world. According to statistics from the former Ministry of Geology and Mineral Resources, 517 mineral deposits have been discovered as a direct result of follow-up studies of anomalies identified by the RGNR Project. The most exciting of these are perhaps two world-class gold deposits (each containing gold reserves in excess of 500 metric tons). One occurs in the greenstone belt located at Xiaoqinling near the border between Henan and Shanxi provinces. The other is the Lannigou gold deposit, which occurs in carbonate terrain of Guizhou Province, southwestern China. This region has become the second largest province for Carlin-type deposits in the world (next only to those near Carlin, Nevada, USA). As a leader in national-scale geochemical mapping, Prof. Xie played an important role in establishing the protocols for geochemical mapping at the global scale as a member of the steering board for the International Geological Correlation Program's Project

259 (International Geochemical Mapping). He continues to be an active member of the International Union of Geological Sciences Working Group on Global Geochemical Baselines.

Prof. Xie received one of the highest scientific honors in China when, in 1980, he was elected as a member of Academia Sinica. He is an adjunct Professor at both Changchun Geological College in Jilin Province and at Beijing University. As such, he has mentored seventeen Master's and PhD students who are currently taking on leadership roles in both geochemical exploration and environmental geochemistry in China.

Prof. Xie Xuejing please accept the Gold Medal Award of the Association of Applied Geochemists in recognition for your achievements in the field of applied geochemistry over a remarkable career that exceeds 55 years and is still going strong.

Professor Xie acceptance speech

Dear Mr. President and Colleagues,

I feel deeply honoured today, to receive this Gold Medal from the Association of Applied Geochemists. The medal is a great honour for me-and not only for me. It is also an honour for those colleagues and students in China, who have worked with me in geochemical mapping for more than 30 years. I am also thankful to my colleagues all over the world for their acknowledgement of China's contributions in the field of geochemical mapping, from the regional to the global level.

In the 21st century, applied geochemistry will continue its development as an indispensable tool in solving crucial problems that confront humanity-In finding new resources and in remedying environmental degradation. It is my belief, that geochemical mapping, from regional to global, will be increasingly fundamental in meeting these challenges.

In so many of the earth sciences- In geography, geology, pedology, hydrology, oceanography, meteorology and others beside-multi-level spatial mapping is vital. But there is one striking hold-out: Geochemical mapping has not been closely correlated with the modern PURE geochemistry as founded by Clarke and Goldschmidt. Geochemical mapping arose mainly as a result of the activities of exploration geochemists.

In April of this year, I was in Guilin to attend the symposium on eco-geochemical mapping and assessment of Eco-geochemical systems. It was there that I heard the sad news of the passing away of John Webb and Arthur Darnley. As you know, these men were pioneers in the development of geochemical mapping.

Immediately a verse by Huang Tingjian, a famous poet of the 11th century sprang to mind:

“Li Cheng is no more, Guo Xi is dead.
What now of the hundred cliffs and thousand summits”

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Acceptance Speech... continued from page 15

When he wrote those lines, Huang was visiting Guilin and Lijiang. With their scenery of winding rivers and soaring karst pillars. Li Cheng and Guo Xi were famous painters but they had died and Huang wonders who now will be able to paint the beautiful landscape of Lijiang.

That day at the symposium, I found myself refashioning the lines:

“Darnley is no more, Webb is dead,
What now will become of geochemical mapping”

It is a joy to see that the geochemical mapping pioneered by such men as Hawkes, Webb, Boyle and Barnley is now being pressed forward by so many successors. This progress has been warmly endorsed in the first day key note address. A whole days general session of this symposium is being devoted to it. I also rejoice to see that both the government and the general public in China and many other countries are finally aware of the importance of geochemical mapping for resources and environmental issues.

I am now an old man in Body. I have, rather to my astonishment, reached the age of 84. But I believe that I still have some time left; to join you in working for the advancement of the young science of applied geochemistry; to help in working out the global picture of mineral resource potential and environmental conditions.

I look forward with great excitement to new efforts to use geochemical mapping to make fundamental contributions to the study of the earth system as a whole.

Many thanks to you all!

Prof. Xie Xuejing



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2006 Student Prize Paper



SGS

JAMIL A. SADER

The Association of Applied Geochemists and SGS are pleased to announce Jamil A. Sader as the winner of the 2006 Student Paper Prize. This prize is awarded for the best paper in a refereed scientific journal by a student, on work performed as a student and published within five years of graduation, which addresses an aspect of exploration geochemistry or environmental geochemistry related to the mining industry. His winning paper is based on research that Jamil undertook for his MSc at the University of Texas at Dallas.

His award-winning paper is entitled ‘*Low-temperature serpentinization processes and kimberlite groundwater signatures in the Kirkland Lake and Lake Timiskaming kimberlite fields, Ontario, Canada: implications for diamond exploration*’ which was published in *Geochemistry, Exploration, Environment, Analysis*, 7:3–22, and was co-authored by Matt Leybourne, Beth McClenaghan and Stew Hamilton. The abstract of the paper is:

“Groundwaters from diamondiferous kimberlite pipes in the Kirkland Lake and Lake Timiskaming regions display unusual geochemical characteristics and signatures compared with groundwaters from the surrounding host rock. Reaction modelling was used to better constrain water/rock ratios, alteration mineralogy and groundwater geochemistry. A soil-zone Ca-HCO₃ water from glacial till was reacted, using a reaction-modelling program, with three different suites of minerals: a kimberlite suite, a felsic intrusive suite and a mafic intrusive suite. Decreasing pH and alkalinity with increasing water/rock ratios in model reactions with the kimberlite suite suggest that sampled groundwaters are from both the hypabyssal facies (high pH and alkalinity; low water/rock ratios) and the diatreme facies (low pH and alkalinity; high water/rock ratios). Geochemical

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Student Paper Prize... continued from page 16

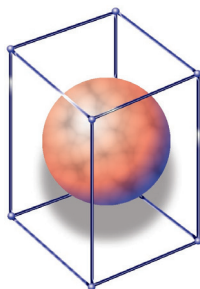
concentrations of sampled groundwaters from kimberlites were compared to modelled waters; results indicate that these waters are different from those flowing through local felsic or mafic intrusive rocks. The kimberlitic groundwaters, and modelled waters, contain low concentrations of Mg and Fe, high concentrations of K and Ca, have elevated pH (up to 12.45), and are defined as a Ca-OH water for the A4 and B30 kimberlites. In contrast, the C14, Diamond Lake and 95-2 kimberlites contain groundwaters that have higher Mg and Fe, lower Ca and K concentrations, and relatively low pH (8.5–10). The reaction model suggests that different minerals precipitate where the water interacts with different kimberlite facies and/or where a different water/rock ratio exists. More hydroxide phases form where pH and hydroxide alkalinity are high. Where kimberlite waters interact with host-rock waters, minerals not likely to be found otherwise, such as magnesite, brucite and magnetite, may be detected along fractures, or near seeps or springs where groundwater comes to surface. Exploration for kimberlites can benefit from the use of groundwater. Groundwater interaction with kimberlitic rocks produces characteristic aqueous geochemical anomalies due to low-temperature serpentinization reactions. The identification of geochemical anomalies in the groundwaters down-flow of a kimberlite and the unusual mineral assemblages that may precipitate from these groundwaters may aid in the location of undiscovered kimberlites."

Jamil completed his undergraduate studies at the University of Saskatchewan and is currently undertaking a PhD at the University of Ottawa. He receives a \$1000 cash prize from SGS, a two-year membership of the Association of Applied Geochemists, together with our journal, *Geochemistry: Exploration, Environment, Analysis* and newsletter, *Explore*, and a certificate of recognition.

The Association of Applied Geochemists would like to thank SGS for, once again, supporting this prize. For the 2007-2008 competition, papers **must have** been published (or accepted for publication) in GEEA.

D.R. Cohen

Chair, Student Paper Competition Committee



Robert G. Jackson

Consulting Geochemist

**3D Zonation Modeling and Vectoring
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Survey Designs and Data Interpretation**

*Seeking new target possibilities through
3D visualization*

3 Leamont Terrace
Dartmouth, N.S., Canada B2Y 1V1
rgjackson@eastlink.ca 902-463-6910

Student Presentation Awards – 23rd International Applied Geochemistry Symposium, Oviedo, Spain

Once again, the Association of Applied Geochemists conducted student presentation competitions at its biennial meeting in Oviedo, Spain in June, 2007. More than 100 talks and posters were presented by students of applied geochemistry at the meeting, and nine judges had a difficult time selecting between the large numbers of excellent presentations. The local organizing committee and the AAG would like to thank the judges of these competitions (Brenda Caughlin, Steve Cook, Bob Eppinger, John Gravel, Paul Morris, Ryan Noble, Mark Pirlo, Paul Polito, and Nigel Radford) for their efforts at identifying two sets of very deserving presentations, and to thank Bob Eppinger for scrutinizing the results.

The winners of the student oral competition were:

First Place: Bigelow, J., Stanley, C.R. Geochemical controls on very coarse-grained Au mineralization within sheeted quartz veins at the mesothermal Callie Au deposit, Dead Bullock Soak, Northern Territory, Australia. Dept. of Geology, Acadia University, Nova Scotia, Canada.

Honorable Mention: Reid, N., Hill, S., Lewis, D. Mineral expression and plant species differences at the Titania prospect: Biogeochemical sampling in the Tanami region, Northern Territory, Australia. CRC-LEME, University of Adelaide, South Australia.

Honorable Mention: Holley, E.A., Craw, D., Kim, J.P. Gold-mining related and natural mercury in an orogenic greywacke terrane, South Island, New Zealand. Dept. of Geology, University of Otago, Dunedin, New Zealand

The winners of the student poster competition were:

First Place: Mohktari, A., Cohen, D., Gatehouse, S. Detection of deeply buried mineralization using ground conductivity and pH measurements. School of Biological, Earth and Environmental Sciences, University of New South Wales, Australia.

Honorable Mention: Chider, K., McQueen, K. Gold dispersion in the calcrete zone, Girilambone Region, Western New South Wales, Australia. Australia National University, Canberra, Australia.

Honorable Mention: Garban, G., Martinez, M. Rare earth, major, and trace element composition profiling of bedded chert and siliceous shale sequences across the Santonian-Campanian boundary in Western Venezuela. Instituto de Ciencias de la Tierra, Universidad Central de Venezuela, Caracas, Venezuela.

Congratulations to all of the student presenters for their excellent efforts at conveying their thesis research to the scientific community!

Clifford R. Stanley,

Associate Professor of Applied Geochemistry & Economic Geology

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Acadia University, Wolfville, NS B4P 2R6, Canada
cliff.stanley@acadiau.ca



Memorial

John Stuart Webb (1920-2007)



John Stuart Webb, the British “father of applied geochemistry”, died at Redhill, England, on 2 April 2007. He was born at Balham, London on 28 August 1920, eldest child of George Stewart Webb and his wife Caroline Rabjohns, née Pengelly. His sister, Mona Audrey, was born in 1924. He attended St. Mary’s school, Balham (1925-30) and Westminster City School, London (1930-38). Having tossed a coin to decide whether he should read medicine or geology, Webb enrolled at the Royal School of Mines, Imperial College (IC), London. He graduated BSc (First Class Honours) in Mining Geology in 1941, having received the Murchison medal and prize (1939), the Brough medal and prize (1940), the Clement le Neve Foster prize (1941) and Cullis Testimonial Fund (1941).

Following a short period as assistant mining geologist to the Government’s Non-Ferrous Metallic Ores Committee, he joined the Royal Engineers in 1941, but was transferred in 1943 to the Geological Survey of Nigeria, as an economic mineralogist, to look for tantalum (then needed for the Manhattan Project and in radar development). He was invalided back to England in 1944, having caught both tick-typhus and malaria. On recovery, he was awarded a Beit Scientific Research Fellowship at Imperial College and began his thesis on *The origin and mineral paragenesis of the tin lodes of Cornwall*, for which he was awarded the Judd Prize in 1946 and his PhD (Mining Geology) and Diploma of Imperial College in 1947. Appointed Lecturer in Mining Geology, he remained at Imperial College, becoming Reader in Applied Geochemistry (1955), Professor of Applied Geochemistry (1961), and retired in 1979 as Emeritus Professor and Senior Research Fellow (1979-89). He was awarded his DSc (London University) in 1967.

In 1948, Webb was awarded the Daniel Pidgeon Fund of The Geological Society, London, to investigate tin-tungsten mineralisation in the Hercynian of Western Europe. However, aware of work on the application of geochemistry to the search for mineral deposits in Scandinavia, he began research in applied geochemistry (then an unknown subject in Britain) in 1949. Together with a colleague, Anthony Millman, Webb used a colorimetric analysis method developed by the US Geological Survey’s Geochemical Prospecting Unit (GPU, established in 1946) and emission spectrography to measure lead and zinc concentrations in natural waters and vegetation close to known mineralization in Ebonyi State, southern Nigeria. They next analysed the metal contents of plants and soil over mineralisation in Cornwall and Derbyshire, England. They concluded that soil might

be better than vegetation as a sampling medium for mineral exploration.

Mutual interest in mineral exploration in Ebonyi brought Herbert Hawkes (1912-1996) of the GPU into contact with Webb. In 1952, Webb visited the GPU and he and Hawkes toured academic and commercial geochemical projects across North America together. On his return, in *A review of American progress in geochemical prospecting* (1953), Webb proposed the establishment of a British programme to examine the application of geochemical methods to mineral exploration, with a view to providing its mining industry “with additional methods of exploration particularly applicable to difficult tropical terrain.”

Meanwhile, with finance secured by Webb from London, Hawkes, together with his GPU colleague Harold Bloom (1913-2001) and John Riddell of the Quebec Department of Mines, began a pioneering regional survey for base metal deposits over 27,000 mi² (69,000 km²) of eastern Canada, based on colorimetric analysis of the fine-grained (<0.177 mm) sieved fraction of samples of stream sediments, an altogether new sampling medium. Webb joined the survey in the summers of 1954 and 1955. They located several promising geochemical anomalies and a metallogenic province subsequently found to contain practically all of the significant base-metal deposits. This discovery established the effectiveness of the technique as a low-cost tool for regional mineral exploration, complimenting the use of soil surveys as a follow-up method. Webb also realised that multi-element drainage surveys might eventually be used to produce cost-effective regional geochemical maps “analogous to geological maps ... to give additional information not only to the economic geologist but also to geologists concerned with the fundamental aspects of geology.”

In 1954, the Geochemical Prospecting Research Centre (GPRC) was established in the Mining Geology section of IC with Webb as its Research Director, assisted (1953-1972) by John Tooms, his first doctoral student. It gradually enlarged with the addition of Ronald Stanton (d. 1979), chemist, 1955-1968; geologist/geochemists: John Fortescue, 1960-1963; Clifford James (1931-2003), 1961-1963; and Ian Nichol, 1961-1969; and agricultural scientist, Iain Thornton, 1964-1988; plus additional staff and numerous research students. Initial studies were concerned with mineral prospecting using soil and drainage sampling in Zambia, Uganda, Sierra Leone, Botswana, Tanzania, North Borneo, Burma and Malaya, and extended in the 1960s to Zimbabwe, the Philippine Republic, Borneo, Fiji, Malaya, East Africa, Australia, and Britain. During the early years, Webb spent considerable time in the field with students. An excellent leader and teacher, his knowledge of geology proved invaluable to his assessment of a field problem and the critical geochemical information necessary to solve it.

Webb’s multi-element regional mapping concept was first tested in 1960 in Zambia, using drainage samples

continued on page 19

John Stuart Webb... *continued from page 18*

collected over 3,000 mi² (7,800 km²) of the metamorphic and intrusive rocks of the Namwala-Livingstone area. The results confirmed that the maps delineated favourable areas for further prospecting but also yielded information relating to fundamental regional geology. By 1966, it was proven an equally effective tool in Sierra Leone. In 1963, Webb initiated studies to investigate the relationship between regional geochemistry and agricultural problems in livestock in Eire and, in 1965, the counties of Devonshire, Denbighshire and Derbyshire in England (the first regional-scale drainage surveys in Britain). Application of geochemistry to marine mineral exploration began in 1964.

To reflect the increasing scope of its applications, in 1965 the GPRC was renamed the Applied Geochemistry Research Group (AGRG). By 1966, its work had established that regional geochemical surveys could aid recognition of areas of potential disease in cattle and sheep at both clinical and sub-clinical levels and, in 1970, Webb proposed that regional geochemistry should form part of any study of trace elements and human health, particularly in developing countries. In the following year, AGRG began a broad programme to identify the severity of contamination in the urban, agricultural and marine environments as a result of both air-borne and water-borne pollution related to human activity. Follow-up studies to the pioneering stream sediment geochemical atlases of Northern Ireland (1973) and England and Wales (1978) amply confirmed Webb's vision that regional geochemistry could provide an invaluable multi-purpose adjunct to conventional geological mapping.

Webb pursued his vision for applied geochemistry over the years with a single-minded tenacity, often in the face of considerable scepticism. By the time of his retirement in 1979, technology-transfer to the mineral exploration industry of field and laboratory methods developed by the Group had been wholly successful; studies related to human health and pollution had been added to the Group's portfolio; and marine geochemistry had been extended to the study of undersea phosphatic deposits, precipitates from metal-rich brines, and manganese nodules in the deep oceans. Over 100 students had been trained, many of whom went on to leading positions world-wide in the mining industry, geological surveys, or academia. Webb's farsighted vision of geochemical atlases as a strategic national requirement eventually became fully realised in the UNESCO Division of Earth Sciences International Geochemical Mapping Project, inaugurated in 1988. Nevertheless, while acclaimed abroad, recognition by many of his peers in Britain of the importance of the work undertaken under his guidance by GPRC and AGRG over the years was lacking.

Webb authored or co-authored 80 publications, including his classic textbook with Hawkes, *Geochemistry in Mineral Exploration* (1962; second edition, 1979), the *Provisional Geochemical Atlas of Northern Ireland* (1973) and *The Wolfson Geochemical Atlas of England and Wales*

(1978), whose like was not to be equalled for some twenty years. His honours include the Consolidated Gold Fields of South Africa Gold Medal, Institution of Mining and Metallurgy, 1953; election as: Honorary Member, Association of Exploration Geochemists, 1977; Fellow, Royal Academy of Engineering, 1979; and Honorary Fellow, Institution of Mining and Metallurgy, 1980. He was dubbed "Father of English Geochemical Mapping" on his award of the William Smith Medal by The Geological Society in 1981.

Webb's interest in medicine never left him, and he was Honorary Secretary, 1975 to c. 1982, of the Rowhook Medical Society (a campaigning-group founded by the pathologist and virologist, George Dick). He married Jean Millicent Dyer (1920-1997) in 1946. Their son, Stuart, was born in 1950.

Donations honouring John Webb can be made to: The Distinguished Applied Geochemists Fund (J. Webb), Association of Applied Geochemists, c/o Betty Arseneault, AAG Business Office, P.O. Box 26099, 72 Robertson Rd., Nepean, ON, Canada K2H 9R0 (Tel: (613) 828 0199; Fax: (613) 828 9288). Donations can be made by: (1) wire transfer to: Canadian Imperial Bank of Commerce, Bells Corners Branch Transit No. 00586, Account no. 02-05117 Robertson Road, Bells Corners, Nepean, Ontario Canada, K2H 5Z1 (Tel: (613) 820 0300; Fax: (613) 820 3203); (2) A cheque made payable to: The Association of Applied Geochemists; or (3) by credit card: Please contact Betty Arseneault (office@appliedgeochemists.org) to make arrangements.

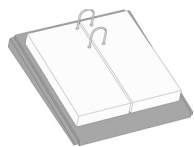
Richard J. Howarth

*Department of Earth Sciences
University College London*

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CALENDAR OF EVENTS

International, national, and regional meetings of interest to colleagues working in exploration, environmental and other areas of applied geochemistry. These events also appear on the AAG web page at:

www.appliedgeochemists.org

2007

- September 9-12, 2007. **Exploration 07** Toronto, Canada. Website: <http://www.exploration07.com/>
- September 24-30, 2007. **AGS-2007 Symposium: Ores and Orogenesis-CircumPacific Tectonics, Geologic Evolution and Ore Deposits.** Tucson, Arizona, USA Website: <http://www.agssymposium.org>
- October 22-24, 2007. **World Gold 2007** Cairns, Australia. Website: <http://www.ausimm.com/gold2007>
- October 28-31, 2007. **GSA Annual Meeting, Earth Sciences for Society – Beginning of the International Year of Planet Earth,** Denver, Colorado, USA website: <http://www.geosociety.org/meetings/2007/>
- November 11-16, 2007. **18th International Symposium on Environmental Biogeochemistry (ISEB-18).** Taupo, New Zealand. Website: www.isebiogeochemistry.com/ISEBXVIII.htm
- November 27-29, 2007. **6th Fennoscandian Exploration & Mining FEM 2007** Lappia Hall, Rovaniemi, Finland. Website: www.lapinliitto.fi/fem2007/index.htm

2008

- January 28-31, 2008. **Mineral Exploration Roundup 2008,** Vancouver, Canada. Website: <http://www.amebc.ca/roundupoverview.htm>
- March 2-5, 2008. **Prospectors & Developers International Convention, Trade Show and Investors Exchange, Toronto, Canada** Website: <http://www.pdac.ca/pdac/conv/index.html>
- May 26 - 28, 2008. **Geological Association of Canada/ Mineralogical Association of Canada Annual Meeting** Quebec City, Canada. Web site: <http://www.quebec2008.net>
- June 29 - July 4, 2008. **Geochemistry of Mineral Deposits,** Il Ciocco Lucca (Barga), Italy. <http://www.grc.org> programs.aspx?year=2008&program=geochem

■ July, 2008. **SEG-GSSA 2008 Resurgence of Economic Geology and the Minerals Industry in Africa,** Joint Conference of the Geological Society of South Africa and SEG Incorporating GEOFORUM 2008. Johannesburg, South Africa. Website: <http://www.seg-gssa2008.org/>

■ July 13 -18, 2008. **Goldschmidt 2008,** Vancouver, BC, Canada. Web site: www.goldschmidt2008.org

■ August 5-14, 2008. **33rd International Geological Congress,** Oslo, Norway. Website: <http://www.33igc.org>.

■ August 10-15, 2008. **9th International Kimberlite Conference (9IKC)** Frankfurt, Germany. Website: <http://www.9ikc.uni-frankfurt.de/>

■ August 18-22, 2008. **Geochemistry of the Earth's Surface 8:** Joint Meeting of the IAGC, Minsoc and Natural History Museum, London, UK. Contact: M.E. Hodson, m.e.hodson@reading.ac.uk

■ October 5-8, 2008. **Geological Society of America Annual Meeting,** Houston, Texas, USA. Website: www.geosociety.org/meetings/index.htm

2009

- July 5-10, 2009. **Goldschmidt 2009.** Davos, Switzerland. Website: <http://www.goldschmidt2009.org/>
- September 2009. **Geoanalysis 2009.** Drakensberg Region, South Africa. Website: <http://geoanalysis2009.org.za/>

Please let this column know of your events by sending details to:

Beth McClenaghan
Geological Survey of Canada
 601 Booth Street
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Questionnaire Regarding the Biennial International Applied Geochemistry Symposia

This questionnaire is provided to gather information for future International Applied Geochemistry Symposia (IAGS). The information will be used by AAG Council and local organizing committees in planning future symposia. If you have attended one of the last three symposia (Oviedo in 2007, Perth in 2005, or Dublin in 2003) please fill out the form and mail it to:

Paul Morris
AAG Symposium Committee
Geochemistry Section
Geological Survey of Western Australia
100 Plain Street, East Perth 6004
WESTERN AUSTRALIA
Fax: 61 8 9 222 3633
Email: paul.morris@doir.wa.gov.au

1. *What preference do you have for symposium format?*

single track (plenary sessions) only parallel technical sessions based on topic area
 no preference

2. *Technical sessions for this meeting typically run for 3 to 4 days in length. Do you prefer no gap in the sessions, a 1-day gap (such as a Wednesday), or a 2-day gap (such as a weekend)?*

no gap 1-day gap 2-day gap no preference

3. *If the above gaps are included between sessions, should the gap day(s) be free of planned activities or should they have planned geology/geochemistry tours or short courses?*

free of planned activities geology/geochemistry tours
 short courses no preference

4. *Do you prefer the conference to be held in upscale hotel/conference centres such as at Oviedo/Perth or lower-cost university-type venues such as at Dublin?*

upscale facility lower-cost facility

5. *How important are pre- and post-symposium fieldtrips to you?*

very moderately minimally no preference

6. *Should all fieldtrips include strong geology and geochemistry components?*

yes no no preference

7. *Are accompanying spouse programs important to you?*

yes no no preference

8. *Should AAG subsidize students for the meeting and associated fieldtrips?*

yes no no preference

9. *What is your affiliation?*

industry academia government private

10. *Do you usually present your work as:* oral poster both

11. *Are you usually interested in attending workshops at Symposia?* yes no

12. *What is your principal area of interest in geochemistry?*

exploration environment both analysis data management.

OVER.....

13. *Freeform area.*

For you, what was the highlight of the last symposium you attended?

How can the symposium be improved?

Other comments or ideas?

New Members and Fellows

Fellows (Voting members)

Prof. Theophilus Davies
University of Jos
Dept. of Geology & Mining
University of Jos, Bauchi Rd.
Jos, Plateau State
NIGERIA PMB 2084
AAG Membership # 3910

Mr. Stewart M. Hamilton
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Mr. Steve Ludington
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Student

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Membership # 3949



AAG Member News

David Kelley has accepted the position of Manager - Americas Exploration for Zinifex Ltd. and will be based in Denver, Colorado, USA. Zinifex is a major zinc and lead producer headquartered in Melbourne, Australia. David was previously with Newmont Mining Corporation. His contact details are as follows:

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RECENT PAPERS

This list comprises titles that have appeared in major publications since the compilation in **EXPLORE** Number 135. 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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RECENT PAPERS

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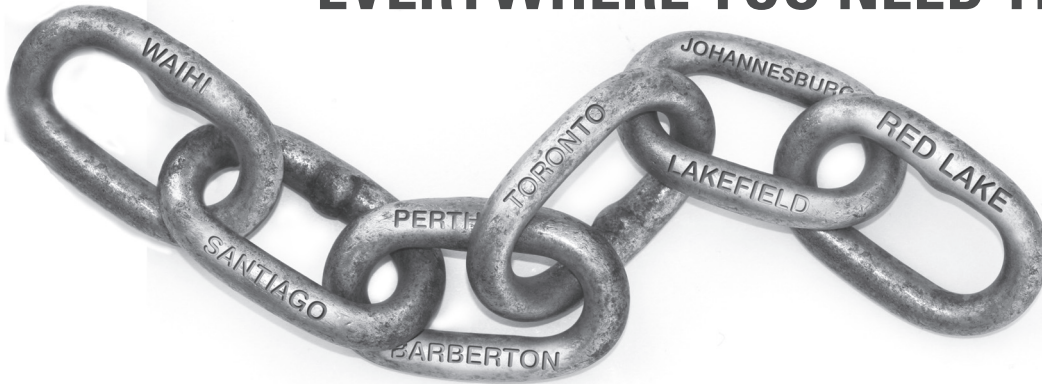
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