SELENIUM IN GEOLOGICAL SAMPLES:
INVESTING IN A MATHEMATICAL MYSTERY

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ABSTRACT

Investigations into the environmental fate and effect of anthropogenic sources of selenium in the water and sediment downstream of some mining areas are ongoing. Complimentary investigations into the sources and mechanisms of selenium release from the host geology are also underway. The existing geochemical data for selenium concentration in rock samples has a reported analytical variability of between 6% and 135% (of the sample mean, 95% confidence limits). The difficulties and implications of utilizing data with this level of variability, to interpret and quantify mechanisms of selenium leaching, are discussed. Many authors subscribe primarily to a sulfate-oxidation release mechanism, as selenium is known to substitute for a variable portion of the sulfur within a rock matrix. No data has been reported that fully supports this hypothesis. Data that would support competing or alternative sources and mechanisms of selenium release are discussed. The industry is currently collecting thousands of selenium samples for each environmental assessment, even though there are no protocols on how and where geological samples should be collected, stored or on how the data should be interpreted. A collaborative research program to develop new analytical techniques, to aid in resolution of this situation, is introduced.

INTRODUCTION

Selenium is an essential trace element in the animal kingdom. There is however a narrow range between minimum required levels and toxic levels (Schrauzer, 2000). Anthropogenic sources are increasing the amount of selenium found in the water and sediment downstream of some mining and other industrial areas, as shown in Table 1.

The potential for environmental effects relating to any aspect of mining is a regulatory, public and industry concern. Studies to improve our understanding of the complex selenium cycle in and near aquatic habitats are under active investigation (EVMEMC, 2003).

A complimentary field of selenium study, discussed in this report, is the quantification of the potential for selenium release from a geological matrix, because of mining activities. Throughout this report, the term "selenium samples" refer to geological samples, not aquatic or biological samples.

The basic requirements for assessing potential selenium leaching are precise data on the original concentration and speciation of selenium in the host geology (Tokunaga et al., 1998; Lemly, 2007). The measurement of the concentration of selenium contained in a sample is as important as the analysis of the portion that leaches, based on some kind of kinetic assessment.

Table 1. Selenium concentration in waterways down stream of selected Canadian and U.S. mining areas (micrograms/litre, parts per billion, mass to volume)

Location	Downstream, μg/L	Reference
Beaverlodge, Sask.	12 - 14	(Palace, 2006)
Key Lake, Sask.	19	
Red Lake, Ont.	18 - 35	
Miramar, NWT	1 - 4	
Hinton, Alberta	1- 60	(Millennium, 2004)
Dry Valley Mine, Idaho	150 - 230	(Lemly, 2007)
Enoch Valley, Idaho	94 - 355	
Sparwood, B.C.	5 - 20	(McDonald and Strosher, 2000)
Lower Elk River, B.C. (above Koocanusa Reservoir)	1.5 - 3.2	(EVMEMC, 2003)

Regulatory concerns and delays in permitting are one consequence of the absence of validated analytical and predictive geochemical information on selenium leaching. Regulators require reasonable assurance that environmental risks can be detected and resolved, prior to issuing mining permits (Chapman, 2005). A lack of information for decision making and the potential for denial of mining permits results in some urgency to address both concentration and speciation information needs for the mining industry.

This paper reviews the inherent variability of the existing measures of selenium concentration in a geological matrix. A research program to address the current analytical limitation is introduced.

BACKGROUND

There is currently a debate within the scientific and mining community on the best approach to assess selenium and the potential for leaching. Should selenium be included as part of an acid rock (AR) assessment of a potential mine site or is a more specific approach needed? There are at least two possible lines of reasoning:

1. Elemental similarities result in selenium replacing a portion of sulfur in sulfidic rock. Therefore oxidation of the sulfide releases the contained selenium to the biotic environment (Oram, 2007; Ryser et al., 2005; Lussier, 2001; Bond,.2000; Munkers et al., 2000) Thus, including selenium as an additional parameter in a conventional AR assessment would seem reasonable; but

2. If sulfide oxidation is the primary mechanism of selenium release then, logically, mine sites with significant AR should experience more profound selenium leaching than alkaline sites. The

reverse has been the case. As well, the chemical properties of selenium are such that it is more soluble under alkaline conditions than acidic. It would seem reasonable therefore to expect non sulfidic selenium sources, with higher or lower concentrations of selenium, and in, as yet, undefined conditions that encourage either dissolution or mobilization of the selenium.

How could alternative selenium sources and mechanisms be discovered, assessed and predicted? Improved screening and measurement techniques for selenium, in situ in a rock sample, would be needed. The analytical variability of existing information is sufficient to obscure much of the needed information. A review of the literature provides support for both of the above arguments. This conundrum in turn leads to an examination of the reported data, used to support these positions.

Information favoring the Sulfur Oxidation Release Mechanism

Lussier (2001) operated humidity cells containing five different seleniferous materials from the southeast coal mining district of British Columbia, for 20 weeks. She observed a strong positive correlation between the amount of selenium and sulfate in the leachate, which led her to suggest that sulfide oxidation is likely the source of selenium released into tributaries of the Elk River.

Three related investigations of selenium leaching were made in the phosphate mining district in southern Idaho, USA. One study related to an area of a waste rock dump (Munkers et al., 2000) and a second to a nearby rock drain (Bond, 2000) at the Smoky Canyon Phosphate mine. The third study investigated sediment speciation in the waterways below the phosphate mines (Oram, 2007). All three reported that selenium leaching was a result of sulfide oxidation. In one case, this conclusion was supported by observations of parallel increases of selenium and sulfate in a stream as well as measurements of selenium within the pyrite lattice structure of the host rock (Bond, 2000). As well, a thin section microprobe was used to confirm that 1.28% of the sulfur in the pyrite, by weight, was substituted for by selenium.

Information Favoring Additional Sources and Release Mechanisms

Sulfur oxidation is likely an important mechanism of release. However, there is evidence to support the existence of other mechanisms. An assessment in the southeastern coal mining district of B.C. reported that, on a mass balance basis, sulfide oxidation could not account for the bulk of selenium that was leaching in this area (Ryan and Dittrick, 2001). Pyritic selenium was low, about 10 ppm and coal seams contained very little pyrite. This lead to the conclusion that pyrite was not contributing much to the total selenium concentration in the samples (Ryan and Dittrick, 2001).

Hypothetical alkaline-release mechanisms for selenium are in better alignment with physical, chemical and observational information than sulfide/acid release mechanisms. Consider:
- Chemical solubility properties of selenium;
  - ☑ more soluble (and therefore mobile) in alkaline conditions than in acidic;
  - ☑ leaching was first identified as a concern downstream of alkaline coal and phosphate mine sites (Berdusco et al., 2000; Munkers et al., 2000) not downstream of acid generating mine sites; and

☑ documented ionic interactions have been shown to enhance selenium mobility (Dhillon and Dhillon, 2000; Tokunaga et al., 1998).

- Expected relationships in the host geology between sulfur and selenium;
  ☑ selenium concentrations in rock and coal do not exhibit large differences even when there are differences in the sulfide content of the two materials (Ryan and Dittrick, 2001).

- Lack of a relationship between the proportion of selenium and sulfur in the host geology and in downstream water. (Day and Sexsmith, 2005).

Discussion of Adsorption and Desorption Mechanisms affecting leachability

The interpretation of selenium leaching data may be complicated by a delay in its expression as a result of repeated adsorption and desorption steps as the leached selenium works its way through the mass of a mined rock pile. Several researchers have commented on potential attenuation or enhancement mechanisms of selenium leaching. These include:
- Co-precipitation:
  ☑ Gypsum, but only if evaporation-concentration occurs (Day and Sexsmith, 2005); or
  ☑ Barite, from silicates and carbonates (Day and Sexsmith, 2005).
- Sorption:
  ☑ Incorporated into precipitated iron hydroxide ($Fe(OH)_3$) (Day and Sexsmith, 2005);
  ☑ Adsorbed by ferric oxides or certain clay particles (Strawn et al., 2002); but
  ☑ The southeastern B.C. coal study found that selenium concentration did not correlate with iron oxides (Fe_2O_3).
- Ion exchange possibilities that may enhance the mobilization of selenium:
  ☑ Chloride (Cl^-), nitrate (NO_2^-), organic carbon (Chapman, 2000) and some metals for example magnesium (Mg) and manganese (Mn) (See et al., 1995; Ryan and Dittrick, 2001).

Sources of Analytical Variability

The limited precision and accuracy of selenium analysis in geological samples has received little attention in the literature. One reference reviewed the lack of data quality for selenium in soils in quite disparaging terms (Fisher, 2000) specifically regarding the lack of standardization in:
- sampling protocol;
- sample storage;
- sample preparation; and
- sub-sampling for analysis.

Examples, in support of Fisher's (2000) position, relating to the diversity of approaches to manage volatilization of selenium, after collection and during analysis, include:
- no information reported;
- "low" temperature drying (35 $^{\circ}$C) (Munkers et al., 2000; Kunli et al., 2004);
- room temperature drying; and

- drying in sealed glass tubes (Schulmann-Choron et al., 2000).

All reports indicated that volatilization was controlled but limited or no supporting data was provided except in Ryan and Dittrick (2001). The trace concentrations of selenium in rock are inherently difficult to analyze and subject to potential contamination from a number of ubiquitous sources. For example, hydride analysis, commonly used to determine selenium concentration in rock and soil samples, requires the use of reagents such as nitric and hydrochloric acid that can contain trace amounts of selenium as a contaminant. Selenium is known to substitute for sulfur in many compounds and *vice versa* (Ryan and Dittrick, 2001). Gains or losses from contamination or volatilization may result in biased, skewed and otherwise random variability in the results of trace level analysis of very small samples.

Fisher (2000) noted that samples of soil and plant material remain biologically active while drying at room temperature. Selenium continues to be metabolized and volatilizes. Fisher (2000) observed that volatilization losses are a significant issue in generating accurate and reliable analytical data.

Possible sources of contamination or losses of selenium were assessed by researchers investigating selenium in the microstructures within a rat brain. Only reagents of an "ultra pure" grade were used and they analyzed all materials that would be in contact with samples before undertaking the main research investigation. This assessment included laboratory air (particulates, gaseous selenium), water, dissecting tools, containers, dust and all reagents (Schulmann-Choron et al., 2000).

RESULTS AND DISCUSSION

Variability of Selenium Concentration Data

There are a substantial number of reports on selenium concentrations in rock, soil and sediment but few of these are supported by variability statistics. An in depth assessment of the four reports that included analytical vigor (Kunli et al., 2004; Zawislanski et al., 2003; Ryan and Dittrick, 2001; Lussier, 2001) indicated the standard error of the mean (SE or SEM) for selenium samples ranges between 6% and 135% at the 95% confidence limit (CL). Variability of this magnitude appears to be inherent in all the available analytical techniques.

It is an accepted practice to report standard deviation (SD) as a measure of the variability of data around the sample mean, as was done in the four reviewed reports. The SD however leaves a heightened impression of the accuracy of the data (Zar, 1974). It is more informative to calculate "how good is our estimate of the mean?" This is done by calculating the standard error (SE) which is then used to calculate the more informative confidence interval (CI) and confidence limits (CL) of the mean. One SD, displayed as a vertical bar on a graph, represents a 68% confidence limit for the sample mean. To represent the 95% confidence limit the bar is roughly twice as long. The relevant statistical relationships are:

(1) Standard Error (SE) = Standard Deviation (SD)/square root of the number of samples (N)
(2) Confidence Interval (CI) = SE multiplied by the critical "t" value (usually taken from a table).

(3) Confidence Limit (CL) = Sample mean +/- CI

Lussier (2001) Coal and rock, southeastern B.C.

Lussier's data (2001) from the coalfields in southeastern B.C. are compiled in Figure 1. The relative variability of the mean ranged between 6% and 135%, using a 95% confidence interval (CI). The concentration of selenium and the relative variability do not have a significant correlation ($r^2 = 0.09$) with high or low relative variability over the full range of concentrations found in this study.

Sixteen samples were examined in detail. The samples were analyzed using the best available analytical technologies and multiple laboratories to assess selenium concentration. This constitutes the largest multiple-analysis selenium data set reported in the literature. The multiple data sets show the variability inherent in selenium sampling and analysis.
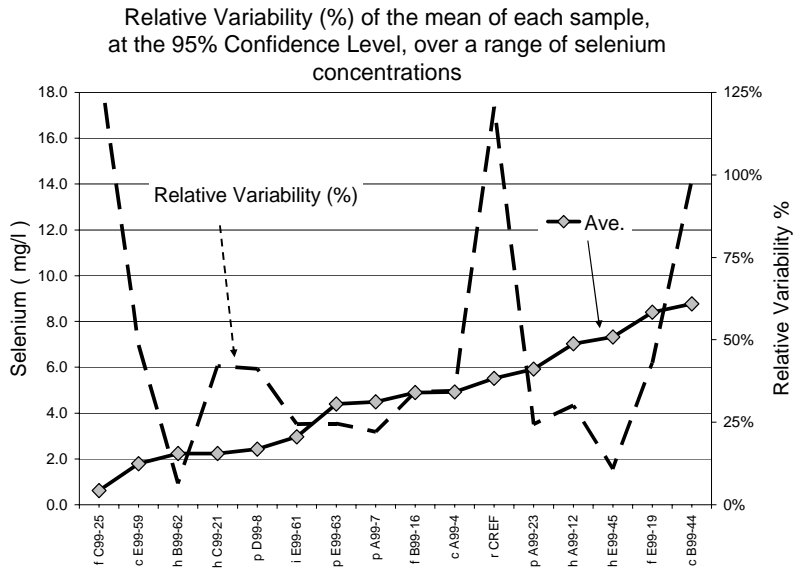

Figure 1: Mean Selenium concentration for sixteen sets of data and the relative confidence interval (percentage) for each (raw data from Lussier, 2001)

Ryan and Dittrick (2001) Coal and rock, southeastern B.C.

The B.C. Ministry of Energy and Mines, analyzed a data set consisting of 375 samples, collected throughout the coal mining area of southeastern B.C. The results of replicate and duplicate quality assurance/quality control data analysis are included in Table 2. Instrumental Neutron Activation Analysis (INAA) was used to assess both the precision and accuracy of the data by repeatedly analyzing one sulfide rock standard (n=5) and one coal standard (n=4), randomly with different batches of samples. A set of duplicate samples was analyzed in a second laboratory as well. It was reported that in the range "between 1 and 5 mg/kg, precision and accuracy were moderate" (Ryan and Dittrick 2001). The calculated 95% confidence interval (CI) was between 23% and 39%.overall.

Zawislanski et al. (2003) Soils in California

This study was in the San Joaquin Valley area of California. Dredged seleniferous sediments, moved to dry land, contained selenium in the 30-50 mg/kg range. The soil samples were analyzed for selenium concentration using synchrotron technology, specifically X-ray absorption near edge structure (XANES) spectroscopy that reportedly agreed quantitatively with the Hydride Generation - Atomic Absorption

spectrometry (HG-AAS) data. The XANES data were not provided and the HG-AAS variability data was presented graphically, as one standard deviation (SD). Using approximations from the graph, estimates of the SE, for concentrations ranging between 0.1 mg/kg to 30 mg/kg, are between 100% and 130% at the 95% confidence interval (CI). This is consistent with HG-AAS data in other studies.

Selenium concentration data from these four reports are summarized in Table 2, to provide an overall context of the magnitude of the analytical variability for selenium in geological samples.

Table 2: Summary statistics on measures of variability in the reported data

Reference	Reported SD mg/kg	Mean mg/kg	Calc. 95% CI of the mean, mg/kg	Calc. range of mean, 95% CL, mg/kg		Calculated relative SE (%)	No. of samples "n"
				From	To		
Ryan and Dittrick (2001) Coal -	0.25	2.35	0.92	2.6	4.1	39%	4
Sulfide -	0.19	3.32	0.79	1.4	3.3	23%	5
	0.33	0.6	0.83	(0.21)	1.44	134%	3
	0.55	1.8	0.87	0.93	2.67	48%	4
	0.06	2.2	0.14	2.09	2.38	6%	3
	0.38	2.2	0.94	1.29	3.17	42%	3
	0.40	2.4	1.00	1.43	3.44	41%	3
Lussier (2001)	0.46	3.0	0.73	2.25	3.70	24%	4
	0.44	4.4	1.08	3.32	5.48	25%	3
16 data sets	0.40	4.5	0.99	3.51	5.49	22%	3
	1.05	4.9	1.67	3.23	6.57	34%	4
	1.07	4.9	1.70	3.22	6.63	35%	4
	4.19	5.5	6.66	(1.14)	12.19	121%	4
	0.91	5.9	1.44	4.48	7.37	24%	4
	0.85	7.0	2.11	4.92	9.15	30%	3
	0.32	7.3	0.80	6.53	8.13	11%	3
	2.27	8.4	3.61	4.79	12.01	43%	4
	5.44	8.8	8.66	0.12	17.43	99%	4
Zawislanski et al., (2003)	1-SD ~0.1 to 20	0.1(lo) 30(hi)	Est. ~ 0.2 Est. ~ 40	0.0 0.0	0.3 70	100% 130%	5
Kunli et al. (2004)	10%	None (est.~1)	1.05	8.9	11.1	11%	None (est. ~6)

SD = Standard Deviation   CI = Confidence Interval   CL = confidence Limits   SE = Standard Error

Kunli et al. (2004) seleniferous soil and rock, China

This study of rock and soil was located in one of the selenosis-endemic areas of China (Shaanxi Province). The data was reported to have a relative standard deviation (SD) of 10%. Unfortunately, there was insufficient detail provided to allow further examination of the data. The number of samples was not provided so the 95% CI was conservatively approximated at 11% (assuming a sample size of 6). This investigation utilized different sampling and analysis protocols from North American reports so there

would likely be little comparability of data between studies. The soil and rock samples were analyzed using hydride generation atomic fluorescence spectrometry (HG-AFS). A solution of nitric and sulfuric acid was used for sample digestion. Sulfuric acid can contain notable amounts of selenium, unless it is an "ultra high purity" grade (Schulmann-Choron et al., 2000). Aqua regia (smoking, freshly mixed, concentrated nitric and hydrochloric acid at a ratio of 1 to 3) is used on this continent. The soil and rock samples were dried at a temperature of 35°C for 6-12 h to "avoid volatilization". The results may be biased high from the sulfuric acid or low due to volatilization at 35^{0}C.

Current Practice: Selenium Assessment

Assessing selenium concentration in geological samples is now undertaken as part of the environmental assessment (EA) and permit approval process for new or expanding mines in western Canada in particular. The B.C. Environmental Assessment Office (EAO) maintains a public database of EA reports. One report, NovaGold Canada's Galore Creek Mine Environmental Assessment (Rescan, 2006) in northwestern B.C. was selected as indicative of the use of "best available technology" for selenium assessment. The work was undertaken by a well-reputed team of consultants and was accepted by the Provincial and Federal EAOs. A summary of this data is shown in Figure 2. (Original data in appendix 5A, Tables 3-3 and 3-4 of the EA.)
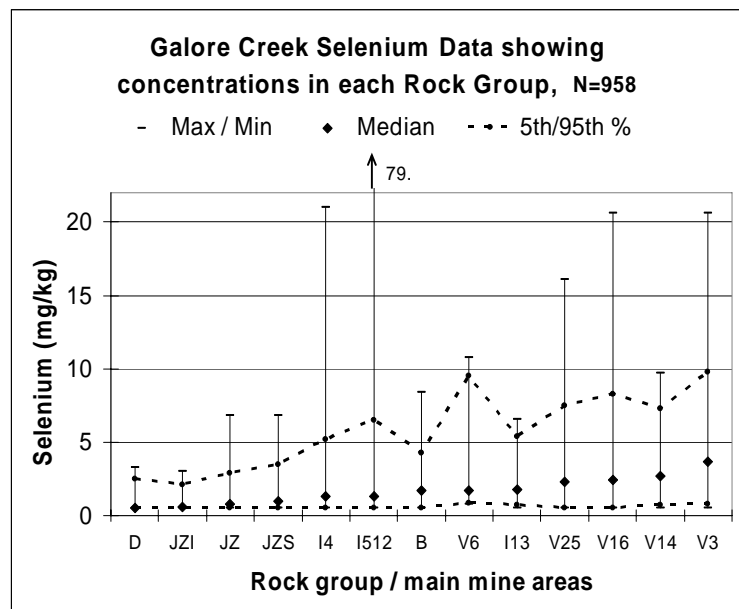

Figure 2: Summary of the selenium data in the Galore Creek environmental assessment, showing the range and median values by rock group.

It was noted in the EA that selenium at this site was broadly correlated with both sulfide-sulfur and copper, suggesting that pyrite and chalcopyrite rocks carry trace, but variable, amounts of selenium (Rescan, 2006). The summary data shown in Figure 2 indicate that the median values for each rock group are similar but the range of values found in each group vary suggesting no correlation between rock group and selenium concentration.

The 5/95% range on the graph represents the values of 90% of the samples in a particular group (Rescan, 2006). The vertical bars should not be mistaken for standard deviations. This is the best available data that can currently be generated. The range of values provides a general impression of the combined analytical and geological variability of selenium within in each rock group. It does not provide any indication of the relative magnitude of each source to the overall variability and no conclusion is possible regarding the potential of the site to leach environmentally significant amounts of the contained selenium.

There are many questions regarding the reliability of currently available analytical methods to determine selenium concentrations in geological samples. The increase in new mine development magnifies the amount of data being generated. At the same time protocols for selenium sampling, sample handling, target geological material, analytical procedures, leach assessment and mechanisms of leaching are unclear or do not exist. As a result, the assembled data has little intrinsic value but a great cost.

Possible Solution

The research hypothesis is that selenium leaching from mine sites is not exclusively sulfate sourced or driven. An investigation into the answers of fundamental questions relating to selenium leaching indicated several data gaps as outlined in this review. Novel investigative tools are more readily available for use in making significant new inroads on understanding selenium sources and leaching. A review of the needed information and the capabilities of the recently commissioned synchrotron facility in Saskatoon, Saskatchewan point to a clear way forward. Synchrotron technology is used to routinely investigate selenium speciation information in a variety of matrices (Christensen et al., 2004; Ryser et al., 2006). The experts at the CLS facility have advised that the analytical procedure envisioned is both feasible and timely (personal communication, Dr. J. Cutler, Associate Director of Research for Industrial Science, CLS). Equipment being installed in the summer of 2007 at the CLS will form a critical component of this work.

The research objectives are to develop:
- a novel synchrotron-based method of measuring trace levels of selenium in geological samples;
- a new understanding of the analytical limitations of conventional analytical techniques; and
- new knowledge on geochemical speciation of selenium in a geological matrix.

Selenium concentrations in selected geological samples will be determined using two conventional methods, inductively coupled plasma mass spectroscopy (ICP-MS) and Instrumental Neutron Activation Analysis (INAA). The results will be compared and contrasted to those generated with the novel synchrotron method. Selenium speciation will be assessed using established synchrotron techniques. The result will be new analytical tools, which in turn will enable further research in this field and a new insight into the potential mechanisms of selenium leaching.

CONCLUSION

A review of the literature indicates insufficient clarity in selenium concentration and leaching data to support sulfur oxidation as the only major source and mechanism of selenium release. Analytical tools with greater precision than those currently in use are needed to undertake basic investigations into selenium leaching. A greater understanding of alkaline leaching mechanisms and the role selenium speciation and ion exchange in the overall process are needed. Novel tools will facilitate new research into predictive assessments of selenium leaching, including both the relative contribution of alkaline and acid leaching mechanisms to downstream water quality.

The new Canadian Light Source Synchrotron in Saskatoon, Saskatchewan, has increased the accessibility of this technology for Canadian industry and researchers. These facilities can be used to provide a new and in-depth look at selenium concentration and speciation in the host geology. No similar investigations appear to have been undertaken to date.

A research project to develop new methods to measure selenium concentration in geological samples, will be undertaken as a collaborative project between Natural Resources Canada, the University of British Columbia (UBC) and the mining industry. Industry input ensures this undertaking is consistent with the identified priorities of mines that may be affected by selenium leaching.

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