A Comparison of Horizon-based Versus Depth-based Soil Sampling

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Introduction

Soil samples are commonly collected and analyzed for various elements by mineral exploration and environmental companies, as well as federal and provincial geological surveys. Interpretations based on the results will potentially direct an exploration company to the bedrock source of the soil anomaly or may result in the recommendation and implementation of a site remediation plan by an environmental company.

The definition of a ‘soil’ sample collected and analyzed for mineral exploration, however, is not the same as a ‘soil’ sample collected for environmental purposes. In mineral exploration, ‘soil’ samples are typically collected from a specific soil horizon (horizon-based sampling), such as a humus layer (H-horizon), a B-horizon soil or a C-horizon soil. A typical Nova Scotia soil profile is illustrated in Figure 1. ‘Soil’ samples collected for environmental studies, such as human health risk assessments, are typically collected from a predetermined depth (depth-based sampling), for example all soil material from 0 to 25 cm of depth.

This small research study, involving a limited field and laboratory component, compares the analytical results of horizon-based soil samples with those obtained from emulated depth-based soil samples.

Defining a Soil

Defining a ‘soil’ is outside the scope of this paper, simply because the discussion surrounding the definition can be lengthy. The layperson’s definition of a ‘soil’ varies widely. For example, a farmer will likely define and describe a soil in different terms than an engineer or a gardener. The same is true for a mineral exploration geologist and an environmental geologist. For the purpose of this paper, all references to soil and soil horizons will be based on The Canadian System of Soil Classification (Agriculture and Agri-Food Canada, 2005).

Overview of Soil Sampling Methods

In mineral exploration, geochemical soil sampling is one of the tools used to identify anomalies for (1) a particular element of economic interest (e.g. gold) or (2) a pathfinder element (e.g. As) that may be an indication of mineralized bedrock.

Consistency in geochemical sampling is stressed in mineral exploration (Levinson, 1980; Dunn, 2007; Rose et al., 1979; Amor et al., 1998; Hoffman, 1986). Dunn (2007) states: “consistency in sampling is of paramount importance.” This is because different soil horizons have different geochemical signatures and physical characteristics; therefore, soil samples must be collected from a consistent soil horizon. If not,
‘soil’ from different horizons may be difficult, if not impossible, to interpret correctly. An example of geochemical variance associated with different soil horizons is presented in Table 1.

**Table 1.** Concentrations of lead and copper highlighting geochemical variance associated with different soil horizons. Modified from Hawkes (1957).

<table>
<thead>
<tr>
<th>Horizon</th>
<th>Depth (cm) (from – to)</th>
<th>Pb (ppm)</th>
<th>Cu (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ah</td>
<td>0 - 1</td>
<td>440</td>
<td>150</td>
</tr>
<tr>
<td>Ae</td>
<td>1 - 5</td>
<td>840</td>
<td>300</td>
</tr>
<tr>
<td>AB</td>
<td>5 – 40</td>
<td>1000</td>
<td>380</td>
</tr>
<tr>
<td>Bf</td>
<td>40 – 75</td>
<td>1300</td>
<td>750</td>
</tr>
<tr>
<td>C</td>
<td>&gt;75</td>
<td>1700</td>
<td>1100</td>
</tr>
</tbody>
</table>

Even in biogeochemical sampling: “intermixing of plant species can generate an unpredictable mixture of unrelated elemental concentrations” (Dunn, 2007). Geochemical variance within an individual plant can also be pronounced (Table 2).

Mineral exploration companies sample specific soil horizons (horizon-based sampling) whether they are performing work in Nova Scotia (Coker et al., 1988; Woodman, 2004), New Brunswick (Lutes, 1985) or any other province or territory in Canada. Once a geochemical anomaly has been identified, more detailed mineralogical studies are typically conducted on the sample to explain the source of the anomaly. These studies may indicate, for example, that gold in a sample occurs in the form of gold-telluride, or gold occluded in sulphide, or that the morphologies of the gold grains suggest a proximal or distal source (DiLabio, 1990; Goodwin, 2005).

Soil sampling is also used in the environmental field. Samples are not collected from a consistent soil horizon, but are collected from surface to a specific sampling depth (depth-based sampling). A standard protocol for collecting soil samples from a specific depth does not exist for preliminary human health risk assessments. In fact, the depth used to collect soil samples at contaminated sites is highly variable and site dependent. A number of factors are taken into consideration and used to determine the preferred sampling depth. Factors that need to be taken into consideration to determine the depth of sampling include the source and type of contaminant(s), the mechanism by which the contaminant entered the soil, the soil porosity/permeability characteristics, the terrain slope, and the potential for humans to be exposed to the contaminant. If a direct pathway of exposure to soil contaminants is deemed to exist, additional soil sampling, including deeper samples, may be required to fully evaluate the contamination issue, particularly in the third dimension.

There are several sources of information that give guidance for collecting soil samples for environmental studies. For example, Federal Contaminated Sites Program at Health Canada indicates that (depending on the definition of surface soil) the depth of sample collection may vary from 0 to 5 cm but can be as deep as 1.5 m (http://www.hc-sc.gc.ca/ewh-smt/pubs/contamsite/part-partie_i/scope-portee_e.html#231). In the Yukon, for example, a maximum sampling depth of 0.5 m below the site surface is recommended, and if deeper samples are required they are to be collected at constant depth intervals such as 0.75 m or 1.0 m intervals (http://www.environmentyukon.gov.yk.ca/pdf/protocol3.pdf). The definition of ‘site surface’ (or the zero reference line) is unsupported in the soil sampling protocol.
Purpose of the Study

The purpose of this study was to emulate a soil survey and compare the analytical results for various elements associated with (1) horizon-based sampling (consistent sample medium) and (2) depth-based sampling (mixed sample media). Samples collected for the horizon-based survey include two distinct soil horizons: (1) a mineral soil horizon, specifically the Bf soil horizon, and (2) an organic soil horizon, specifically the F soil horizon. The depth-based survey emulated soil sampling at various depths by varying the proportion of B-horizon soil (Bf) and organic soil (F) encountered in a given profile.

Methods

Field Sampling

Approximately 6 kg of the Bf soil horizon and an additional 1.5 kg of the organic-rich F soil horizon were collected from a sample site located approximately 500 m north of the main workings associated with the Mooseland Gold District, Halifax County. The sample site was situated in an area of mature softwood growth and was characterized by an undisturbed soil profile. The site was further characterized by the following (simplified) soil profile, from top to bottom: L, F, Bf and C. The H, Ah and Ae soil horizons were absent.

Sample Preparation

All sample material was prepared (and analyzed) at DalTech Minerals Engineering Centre (Dalhousie University) in Halifax, Nova Scotia. The entire 6 kg Bf-horizon soil sample was dried (maximum drying temperature 30°C), rolled, homogenized and sieved, with approximately 200 g of material passing through a 500 microns (<0.5 mm) screen. Similarly, the entire 1.5 kg F-horizon soil sample was also dried (maximum drying temperature 30°C), rolled and homogenized, with approximately 50 g of material passing through a 1000 microns (<1 mm) sieve. A coarser size fraction was used for the humus sample to avoid maceration of the material by mechanical methods.

Where a mixture of mineral soil and organic soil (to emulate a depth-based sample) was required for analysis, the ratio was determined on a volume/volume basis. For example, a soil/organics ratio of 90% soil and 10% organics used 9 units of mineral soil to 1 unit of organic soil. The combined sample was rolled to ensure homogeneity.

Sample Digestion and Analytical Technique

Approximately 1 g of sample was digested for 2 hours in a hot (70°C) aqua regia (3:1 HCl:HNO₃) with 5 drops of HF added. Aliquots were analyzed with a Varian Vista Pro ICP-OES Radial system for 10 elements (As, Co, Cr, Cu, Ni, Pb, S, Sb, V and Zn) and analytical results reported in parts per million (ppm). A separate 0.5 g sample was digested in a 20:1 HNO₃:HCl solution prior to Hg determination by Cold Vapour-Atomic Absorption (CV-AA) using the Bacharach-Coleman Mercury Analyzer System. An antifoaming agent was used with all samples to prevent foaming in the biological oxygen demand bottles due to the presence of organic material in the soil.

No quality control samples were inserted with the soil samples, but numerous certified reference standards were used by the laboratory during analysis.

Results

Results for the five analyses of the Bf-horizon, five analyses of the F-horizon and one analysis of each mixture of 90% Bf and 10% F, 80% Bf and 20% F, 70% Bf and 30% F, 60% Bf and 40% F, 50% Bf and 50% F, 40% Bf and 60% F, 30% Bf and 70% F, 20% Bf and 80% F, and 10% Bf and 90% F are presented for arsenic (As) and mercury (Hg) in Figures 2 and 3, respectively.

Arsenic concentrations range from approximately 10 ppm in the Bf soil horizon to approximately 3 ppm in the F soil horizon and demonstrate a high degree of precision for a specific soil horizon. This analytical precision was repeated for all elements (except Sb) tested in this study.

Arsenic, however, clearly shows a negative correlation with increasing proportions of organic
soil (Fig. 2). This same trend is repeated for Co, Cr, Cu, Ni, Pb and V. Mercury (Fig. 3) and S (and to a lesser degree Zn) show the opposite trend, a strong positive correlation with increasing organic soil. Low concentrations of Sb are present in soil; therefore, most results were at the lower detection limit of 0.5 ppm, regardless of the percentage of Bf or F soil present in the sample.

The graphs clearly demonstrate that a soil geochemical survey designed to sample a consistent soil horizon (e.g. the Bf soil horizon in a horizon-based sampling program) would yield relatively consistent elemental concentrations. If soil horizons are mixed, however, as they would be with a depth-based soil sampling program, then the elemental concentrations will vary simply on the basis of the percentage of Bf or F soil present in the sample. False anomalies or exceedences may be generated simply on the basis of sample inhomogeneity. Mixing soil horizons, therefore, could result in unnecessary funds being spent on (1) a remediation program as the result of an environmental risk assessment or (2) an exploration program chasing normal background concentrations.

Discussion

Soil horizon boundaries are commonly wavy, broken to discontinuous, and rarely maintain a constant thickness even over very short distances. In fact, the upper soil horizons rarely remain static but commonly increase or decrease in thickness to the point where they may even pinch out over several tens of centimetres of lateral distance. Therefore, if a soil sampling protocol required sampling a specific soil horizon (e.g. the commonly sampled B-horizon), the targeted horizon is not likely to occur at a consistent depth but more likely
to occur at variable depths and in some instances may be absent in the soil profile. In a depth-based sampling program, variation in the thickness of soil horizons is ignored. The soil sample is collected from a specified depth regardless of whether the sample site is located in pristine forest, a bog, a manicured yard, a farmer’s field or a flood plain.

This raises many important questions such as:
What is the source of the geochemical response?
What are the geochemical effects of mixing sample horizons? What is the definition of zero (the starting depth of sample acquisition in a depth-based sampling program)? How can soil samples collected from natural, undisturbed sample sites (i.e. forest) be compared with sample sites influenced by anthropogenic effects (e.g. lawn)?

These are critical questions and become increasingly important once analytical results are received and an interpretation of the results is required because recommendations to commit further exploration or remediation expenditures will likely be based on the interpretation.

Why is this important? Take arsenic for an example. The Canadian Council of Ministers of the Environment (CCME) soil quality guideline for arsenic is 12 ppm. Soil concentrations for the Bf soil horizon presented in Figure 2 are at or near the CCME guideline limit for arsenic. Conversely, all samples of the F soil horizon fall well below the guideline limit. If all samples collected contained variable (percentages of) organic material, as would be expected in a depth-based sampling program, it is reasonable to assume that all soil tested would fall below the CCME guideline. Even if some contamination is encountered, it could be significantly ‘diluted’ by the inclusion of organic material in the depth-based soil sample. It is acknowledged that this scenario may be an oversimplification, but it reinforces the argument that sampling a consistent sample medium (as opposed to a consistent sampling depth) is necessary to minimize variance. This also has significant implications for anomaly identification and interpretation for research into long-range transport of pollutants.

Figure 3. Mercury (ppb) results for a Bf soil horizon, emulated mixed Bf/F soil horizons and an F soil horizon.
The greatest source of error occurs at the sampling stage, that is the initial sample collected in the field. Regardless of how many analytical blanks, preparation splits or certified reference standards are blindly inserted in the sample stream, how many times the analytical instrumentation is calibrated or how many samples are sent to a second laboratory as a check, if the wrong (type) of sample is collected, all QA/QC protocols put in place are effectively meaningless.

Conclusions

A limited soil sampling study was undertaken to test the analytical response associated with two types of soil sampling protocols: horizon-based sampling and depth-based sampling. Sample material for the study included the Bf soil horizon and the organic-rich F soil horizon, and samples were collected from an undisturbed site located north of the Mooseland Gold District.

The two soil horizons (B and F) and variably mixed soil samples emulating changing thicknesses of soil horizons were analyzed for As, Co, Cr, Cu, Ni, Pb, Sb, V, Zn and Hg. Results indicate that all elements (except Sb, which occurred at or near the lower detection limit of the instrumentation) demonstrate a relatively high degree of analytical precision for a specific soil horizon. Results for As, Co, Cr, Cu, Ni, Pb and V indicate that for the emulated depth-based sampling there is a negative correlation with increasing organic content. Conversely, an increase in Hg, Sb and Zn show a positive correlation with increasing organic content in soil.

Although simple in design, the results of this study demonstrate the different geochemical responses associated with horizon-based soil sampling versus depth-based soil sampling. As a result, false anomalies or exceedences (in legislated soil quality guidelines) may be generated simply on the basis of sample inhomogeneity. Mixing soil horizons, therefore, could result in unnecessary funds being spent on (1) an exploration program that chases normal background concentrations or (2) a remediation program recommended as the result of a human health risk assessment.

References

