

# The Use of Systematic Sampling and XRF-XRT Based Scanning to Determine Potential Recovery of Metals from Waste Rock

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## Abstract

It is difficult to evaluate the potential for reprocessing and extraction of minerals from waste rock with valuable and/or harmful elements.

We suggest a new sampling strategy/protocol for waste rock, specifically developed for historic mining sites, in combination with XRF-XRT scanning with a GeoCore X10 instrument.

Håkansboda historical mine site in Sweden was used as a case study to look at the potential for the combination of techniques.

The combination of the suggested randomized sampling strategy/protocol and the dataset from the GX10 scanning enables prediction of amenability for pre-processing with the use of mechanical sorting or if the extraction of valuable minerals only can be achieved through fine grinding, flotation or leaching.

**Keywords:** mining waste, secondary resource, sampling, mineral recovery, tomography

## Introduction

Mining waste is abundant and poses an environmental problem. It is also possible that some of the mining waste are suitable secondary resources for reprocessing. In order to determine both environmental impact and resource potential detailed characterization is required. However, it is usually not practical to excavate the entire area and sometimes also cultural heritage issues pose a hurdle to extensive sampling. It is therefore important to have methods for sampling considering these problems but still provide useful results.

It is often complicated to sample and determine average concentrations of elements in historical waste rock. This makes it hard to evaluate the potential for reprocessing and extraction of minerals with valuable and/or harmful elements.

Early evaluation of the potential for reprocessing also need to consider the concentrations of relevant elements, their

host mineralogy and paragenesis, grain sizes and distribution between different size fractions in the mining rock waste.

In this paper, we present a new sampling strategy/protocol for waste rock, specifically developed for historic mining sites. We also look at the combination of X-ray fluorescence (XRF) and X-ray tomographic (XRT) scanning in order to determine the potential for reprocessing.

## Methods

### *Geological setting*

A very quick estimate of the potential for recovery of valuable minerals from waste rock can be obtained by scanning remains of sample preparation pulp from destructive analysis. Håkansboda historical mining site in Sweden was used as a case study to look at the potential for the combination of techniques (sampling strategy and XRF-XRT scanning). Håkansboda mining site has

primarily been mined for copper, but also some cobalt. The mineralization is sulfide based and contains chalcopyrite ( $\text{CuFeS}_2$ ), pyrite ( $\text{FeS}_2$ ), pyrrhotite ( $\text{Fe}_{1-x}\text{S}$ ), sphalerite ( $\text{ZnS}$ ), galena ( $\text{PbS}$ ), arsenopyrite ( $\text{FeAsS}$ ) and some cobaltite ( $\text{CoAsS}$ ).

### *Sampling strategy and chemical analysis*

Sampling of mining waste is anything but straightforward. Heterogeneity issues, for instance, make the use of classical statistical methods problematic. It is important to understand the creation of mining waste deposits in order to understand how to sample and suggesting an approach for surveying and sampling mining waste.

In order to collect a fair sample from a mining waste deposit it is crucial to know how the waste has been generated, why it is deposited in different areas and how the handling of the waste has influenced its homogeneity and thus the prerequisite for collecting a fair sample.

The concept of using cumulative moving average (CMA) to determine when enough samples have been collected to establish saturation is described and exemplified with some analytical results from the historical mining site Håkansboda in Sweden.

It is also suggested that several seemingly peripheral, parameters regarding the mining site (shape of the deposit, vegetation cover, vegetation type etc) are recorded in order to increase knowledge about the site.

It is, however, important to remember that every mining site is unique and site-specific information is important in order to be able to revise the sampling strategy.

Before samples are collected it is important to define the waste piles/objects that are going to be sampled. Each object should ideally cover an area with the same or similar properties. Pieces (20-75 mm) was chipped from rocks in different waste piles/objects. Only one chip was collected from each primary piece. 25-50 pieces were collected for each sample (2-5 kg). To be as objective as possible when chip was selected favouring by size, colour, accessibility, shiny or dull minerals were avoided. It is important to remember that dense material generally stay close to the point of disposal and large pieces will tumble down to the base of the

slope. Samples should therefore be collected evenly to include the various segregations that may have occurred as a result of dumping.

At vegetated piles vegetation was carefully lifted and samples were collected without doing an excavation.

Collected composite samples were sent to MS Analytical, Stensele, Sweden, for sample preparation (crushed to 70% passing 2 mm). A subsample was split and sent for destructive analysis at MS Analytical in Vancouver. Digestion was performed using aqua regia or a four acid mix for the lithogenic elements while analysis were performed by ICP-OES and ICP-MS.

A compromise between the costs for sampling and, based on empirical evidence from earlier investigations, an estimated absolute confidence error of about 25-50% from the true average. 25% is commonly used as a requirement for pre-feasibility studies.

To find the point of saturation, a study of the cumulative moving average (CMA) value of the EMOI (Element or Mineral of Interest) through a series of samples from the same object (same population) will indicate when saturation has been satisfactorily reached. 36 composite samples were collected from Håkansboda historical mining site. Sample saturation, here defined as the point when additional samples do not significantly change the average obtained by previous samples.

### *XRF-XRT scanning*

Remaining sample pulp after crushing and splitting was wrapped in thin plastic cling film and placed in the drill core sample tube. After scanning, which took less than 20 min, it was possible to study a tomographic image of the scanned sample and get indicative chemistry, measured and mineral stoichiometric calculated bulk density and distribution of the dense particles in the sample.

Drill core scanning by combined XRT (X-ray tomography) and XRF (X-ray fluorescence) was performed using the GeoCore X10 instrument (Orexplore AB, Stockholm, Sweden). For elemental concentrations, the GeoCore X10 utilizes a combination of XRF spectra and attenuation to calculate stoichiometric solutions for mineral compositions and final elemental abundances (in wt. % or mg/kg) per unit volume of drill core. Scanning data was imported into the

Orexlore Insight software, to visualize and analyze the obtained digital drill core.

The rendering is reflecting attenuation expressed as metallic aluminum equivalents with the current instrumental setup. Attenuation can not be directly translated to mineralogy, but as a guidance for some case-relevant non-silicate minerals, the theoretical attenuations are estimated to be about: pyrite 3.88, pyrrhotite 4.00, sphalerite 4.17, arsenopyrite 6.52 and cobaltite 6.68.

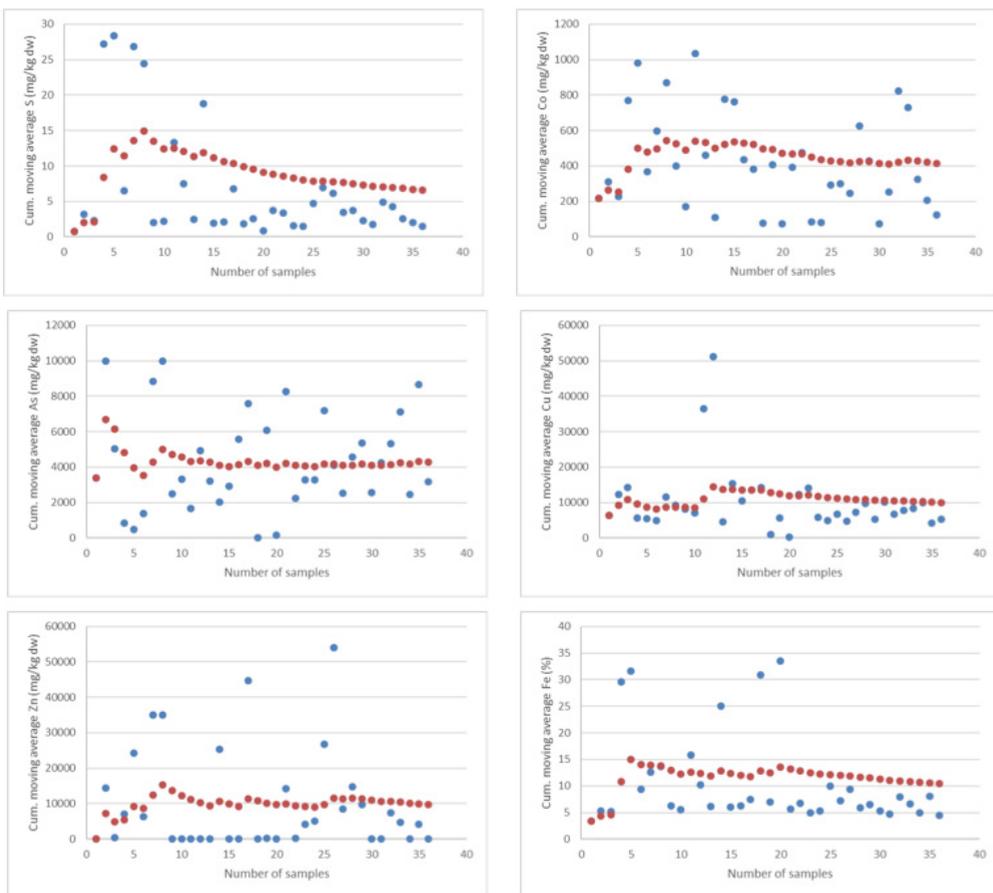
## Results and discussion

### Sampling strategy

From a sample set of 36 waste rock samples from Håkansboda the average concentrations were 6.6% sulfur, 10 000 mg/kg dw copper,

410 mg/kg dw cobalt, 4 300 mg/kg dw arsenic, 9 620 mg/kg dw zinc and 10.4% iron.

Individual concentrations for each sample as well as cumulative moving average concentrations (CMA) are presented in fig. 1 for sulfur, copper, cobalt and arsenic. It is clear that the CMA is getting close to the true average values (within 20-25%) for all presented elements after roughly 15 samples. In practice this means that by collecting and analysing around 15 composite samples from a site you will get a good handle about the average concentrations at the site. It is, however, important to note that even if 15 composite samples are often enough at this site another type of mining waste might require another number in order to reach the true



**Figure 1** Cumulative moving average concentrations (mg/kg dw) for sulfur, cobalt, arsenic, copper, zinc and iron at Håkansboda historical mining site in Sweden. Average concentrations are calculated using increasing numbers of samples (from 1+n until all samples have been included). Calculated average (n 36) concentrations for sulfur, cobalt, arsenic, copper, zinc and iron are 6.6%, 410 mg/kg dw, 4 300 mg/kg dw, 10 000 mg/kg dw, 9 620 mg/kg dw and 10.4%, respectively.

average. Different elements behave differently and have different distribution patterns, some elements are more "nuggety" than others (i.e. compare the distribution of gold versus a rock-forming element like silica).

### XRF-XRT scanning

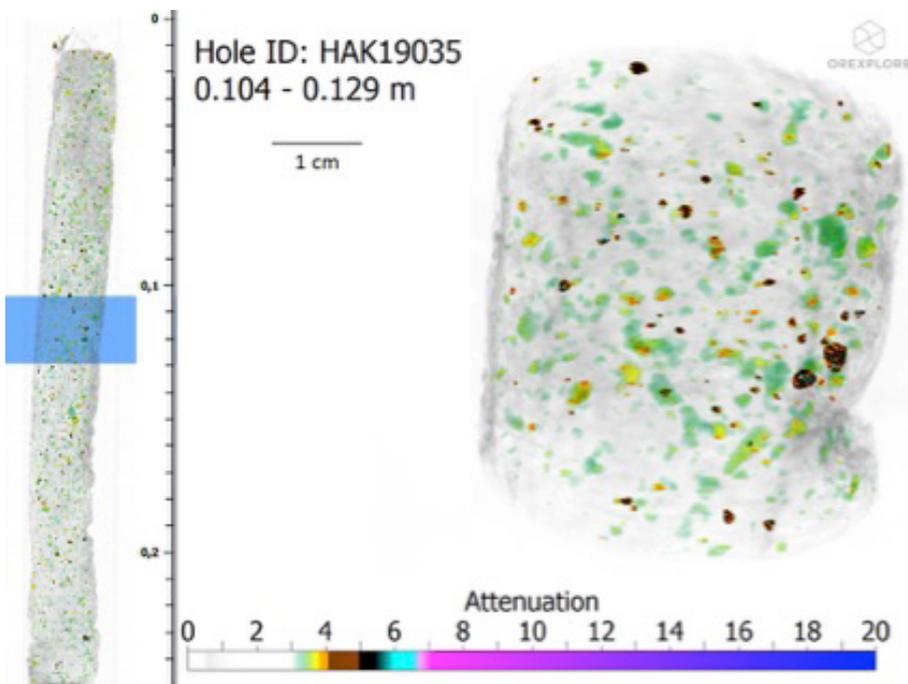
The GeoCore is primarily designed for scanning solid drill cores but has in this experiment been used for small particles. All samples have been scanned, but only results from two samples will be presented. The tomographic image of sample HAK19035 in fig. 2 shows on the left the tomographic image of the total sample length (0.247 m corresponding to 755 g at a measured bulk density of 2.32 g/cm<sup>3</sup>) and on the right a 3D-image of a selected 25 mm (88.2 g) subsample (shown in blue to the left). The subsample is estimated to contain 118 visible non-silicate mineral grains.

Another example is HAK19016 in fig. 3. The entire sample is 0.346 m corresponding to 878 g at a measured bulk density of 2.48 g/cm<sup>3</sup> and the subsample (in blue) is 43 mm (108 g). The subsample is estimated to contain

129 visible non-silicate mineral grains.

It is quite clear from both samples that dense minerals containing trace elements are mainly found as discrete particles. These discrete particles consists of the same mineral and from the results it can be concluded that at this liberation minerals and thus the element of interest can be recovered using physical sorting. There is also information about the sulfide presence in the sample. The data can also be used to determine how much of the potential acid generating sulfide minerals are remaining in the waste after reprocessing.

In tab. 1 below a comparison between element concentrations based on the XRF scans from the GeoCore) and conventional wet chemistry (digestion and ICP analysis) are presented. In general the reported concentrations from the GeoCore are after a ca 20 minute scan, in the same order of magnitude as the conventional analysis but somewhat lower. It is, however, important to remember that the concentrations from the GeoCore are based on the entire section while the concentrations from the conventional analysis are only based on a small fraction



**Figure 2** Attenuation for sample HAK19035. Magnification shows the section 0.104-0.129 m. Minerals with attenuation below 3 has been rendered transparent in the image (carbonates and silicate minerals).

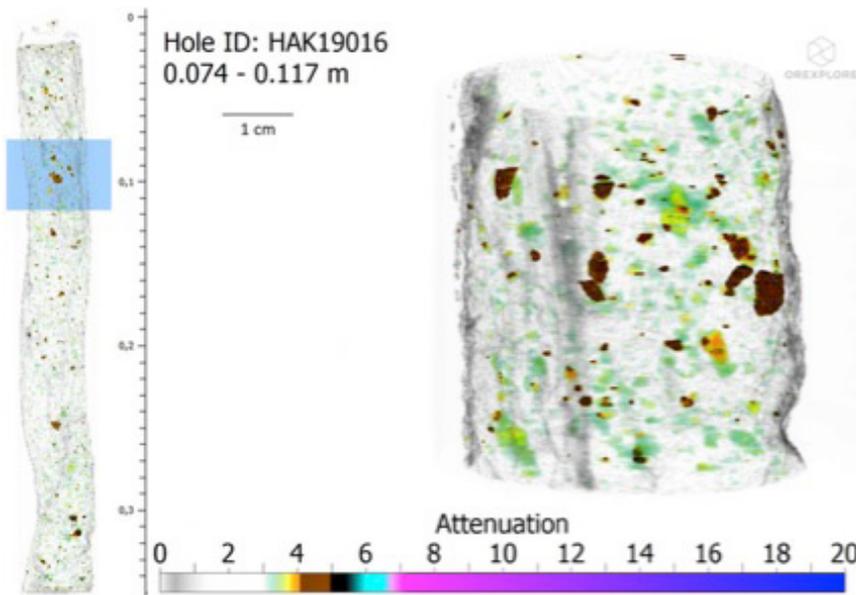


Figure 2 Attenuation for sample HAK19035. Magnification shows the section 0.104-0.129 m. Minerals with attenuation below 3 has been rendered transparent in the image (carbonates and silicate minerals).

of the section. It is thus clear that even if the GeoCore is not calibrated for element concentration determination the results are still a very good indication about the presence of trace elements as well as potentially acid generating sulfur. Since also the mineralogy are determined the acid generating potential can quickly be determined for entire sections of waste rock or exploration drill cores.

**Conclusions**

A quick method has been suggested as an answer to a difficult practical problem. It was found that by using a randomized sampling it is enough to sample approximately 15 composite samples from a site in order to obtain an average within acceptable limits from the "true" average.

Scanning of the crushed waste rock with a XRF-XRT scanner (GX10) provided an indication on elemental concentration, elemental associations and the distribution of grades between particles. This information provided information about the association and clustering of elements within the waste rock and if the mineralization is enough liberated for mechanical sorting at the chosen particle size. The results will also provide an indication about how much of the potentially acid producing sulfides that will be removed from the site when the elements of interest are being recovered.

In summary, the combination of the suggested sampling strategy/protocol and the dataset from the GX10 enables prediction of amenability for pre-processing with the use

Table 1 Comparison between estimated concentrations of sulfur, iron, zinc, copper, cobalt and arsenic based on the GeoCore scans and conventional wet chemistry (MS Analytical, Vancouver).

Element mg/kg dw	HAK19016 Conv.	HAK19016 GeoCore	HAK19035 Conv.	HAK19035 GeoCore
Sulfur	244 000	129 000	188 000	97 500
Iron	136 000	73 900	250 000	81 400
Zinc	350 000	89 500	25 300	8 790
Copper	9 290	4 720	15 300	5 890
Cobalt	825	283	776	378
Arsenic	>10 000	2 090	2 010	809

of mechanical sorting or if the extraction of valuable minerals only can be achieved through fine grinding, flotation or leaching.

Sampling of mining waste is impossible, but it can still be done!

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### **References**

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