



# A robust statistical method for mineralogical analysis in geometallurgical diagnostic leaching



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## ABSTRACT

CSIRO researchers have been involved in the development of predictive metallurgical indices as a tool in the hydro- and geometallurgy fields. Rapid, small-scale, cost effective tests and protocols have been developed for comparative ranking of attributes relevant to leach performance, e.g. leach index (relative indication of leach performance), recovery, impurity deportment, reagent consumption and mineralogy of samples for their relative ranking. Results from these tests can be used for plant design or process optimisation to maximise the commercial value of an ore body and to minimise the social and environmental impact of mining operations. The motivation for development of these tests includes the reduction in use of traditional mineralogical tools, be they for reasons of accessibility, cost, speed or scale-up for processing of large sample numbers. Mineralogical analysis remains essential for validating leach results in the development of test protocols and as a means of quality control. This paper presents an accurate, robust statistical method for QEMSCAN data analysis that has been developed for use in conjunction with the geometallurgical leach tests.

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## 1. Introduction

Geometallurgical information generally consists of large sets of data points and may include information different from what is obtained traditionally from sample characterisation. In the hydro-geometallurgical area specifically, CSIRO researchers have been involved in the development of rapid, small-scale, cost-effective predictive indices for the comparative ranking of attributes relevant to leach performance, including work in the AMIRA International P843A Geometallurgical Mapping and Mine Modelling (GeM<sup>III</sup>) project (Kuhar et al., 2011a,b; McFarlane et al., 2011a,b). The focus of the small-scale leach tests has been to determine the leach index (relative indication of leach performance), recovery, impurity deportment, reagent consumption, effect of mineral grain or particle size and degree of mineral liberation and mineralogy of samples for their relative ranking. Some of this information is obtained from diagnostic or sequential leach tests which can provide information on the deportment characteristics (mineralogical form and associations) of a valuable element of interest. An understanding of the form in which an economic element is present or how it is associated with various host minerals is key to appropriate process design and understanding the geometallurgy of an ore body.

Diagnostic leaching typically involves the use of selected reagents and leach conditions which target specific forms of, or

the minerals associated with the valuable element. The abundance of that mineral form or association is then determined by analysis of that element in the leach solution and of the remaining abundance in the leach residue. Diagnostic leach tests typically involve the successive application of reagents (usually from least to most aggressive) to the residues of previous leach steps, intended to target specific minerals or host phases containing the element of interest. Variants of the diagnostic leaching methodology have been applied to several different ore types, in particular gold (Lorenzen and Tumilty, 1992), copper (Parkinson and Bhappu, 1995), and lateritic nickel ores (Botsis et al., 2011; Swamy et al., 2003). In our research, we have developed tests that are run in parallel rather than in series with the aim of potentially reducing turn-around time and cost.

Validation of the diagnostic or selective leach is necessary to confirm whether the intended mineral leached according to theoretical predictions. However, despite the implementation and use of these leaching techniques, there is an absence in the literature of detailed and robust mineralogical characterisation data to support the proposed methodologies. This may be because of some of the issues associated with data analysis using these techniques. In the gold and copper literature especially, reagent specific mineral solubility information has often been based on leach tests conducted on mostly single or pure mineral samples only, with relatively straightforward characterisation by X-ray diffraction (XRD) (Parkinson and Bhappu, 1995).

Rapid scanning electron microscope-based energy dispersive spectrometer (SEM-EDS) systems such as the FEI Quantitative

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Evaluation of Minerals by SCANning (QEMSCAN) electron microscope and Mineral Liberation Analyser (MLA) have a wide range of uses in the minerals industry, particularly in the field of process mineralogy. The ability to quantify modal mineral abundances in the sample down to approximately 0.01 mass%, and to provide relevant textural information (association and liberation) with a high degree of reproducibility, makes them highly suited to processing applications. Most processing applications utilise these instruments for plant surveys, to supplement mass balance information, and to determine the mineralogical distribution and elemental deportment between various rougher, cleaner and concentrate streams (see for example, the work by Dai et al., 2008). QEMSCAN and MLA are also often applied to investigations of tailings material to determine the liberation (Lotter et al., 2010), or association (Goodall, 2008) characteristics of the unrecovered minerals, or on ore material to determine gold deportment and supplement diagnostic leaching data (Goodall et al., 2005). These studies have used the quantitative data as point estimates for the liberation or modal mineral abundance parameters, without considering sampling (between sample) variability or error.

For the hydrogeometallurgical characterisation of samples, mineralogical analysis could be used initially to verify and calibrate leach recoveries. Thereafter, the leaches could be conducted without further mineralogical analysis. Automated mineralogical techniques such as the QEMSCAN and MLA are capable of producing highly reproducible, quantitative mineralogical results as well as quantifying mineral abundances at low levels and can be used in the leach verification and calibration stage. QEMSCAN data are normalised to 100% (unless mass flow data is entered) and therefore mineral abundances in residues may increase because of mass loss from the dissolution of certain minerals. This makes direct interpretation of mineral abundance changes based on the QEMSCAN data challenging. Furthermore, such a technique would also require the ability to distinguish significant mineralogical changes from background and sampling error.

To address these difficulties, in this paper, a robust statistical methodology is proposed for QEMSCAN analysis, data processing and interpretation of data from leach tests. The methodology is illustrated using results from sulfuric acid, cyanide and aqua regia leaches (commonly used in diagnostic and selective leaching) of a composite copper-containing sample.

## 2. Materials and methods

### 2.1. Samples

A composite sample was created from several flotation concentrates containing mainly sulfide minerals, as well as an iron-oxide and silicate-rich filler material. The concentrates were selected such that the final composite contained copper oxides, secondary copper sulfides (chalcocite and covellite) and primary copper sulfides (chalcopyrite and bornite). The material was ball milled to a  $P_{80}$  of  $-64\ \mu\text{m}$ . The final product was dried, split using a rotary sample divider and stored in a chest freezer. Oven drying times and temperatures were minimised to prevent sulfide mineral oxidation.

Leach residues were riffle split, mixed with graphite powder and de-agglomerated with a fine brush. This mixture was then set in EPOFIX 2 pack epoxy resin, allowed to harden and polished and coated with a thin film of carbon.

### 2.2. Leach tests and analysis

Sulfuric acid (5%) and sodium cyanide (5%) leaches were conducted for 24 h at room temperature with 14 mass% solids and agi-

tated on a mini-bottle roller. Sulfuric leaches were sampled at 0.5, 1, 1.5, 2, 4, 6 and 24 h. Cyanide leach solutions were sampled at 1.5, 3, 6 and 24 h. Sulfuric acid and cyanide titrations were used to determine reagent concentrations throughout the leach and to adjust cyanide concentrations where required. Leach residues were filtered, washed and oven dried at 60 °C before riffle splitting and submission for chemical and mineralogical analysis. Residual copper was determined by aqua regia digest. Leach recoveries were calculated using the extracted grade in the leach liquor (based on solids density and liquor grade) and the residue assay.

Filtered leach solutions were submitted for multi-elemental analysis by ICP-OES. Free acid titrations were also conducted on final samples from acid leach tests. Head samples and leach residues were dissolved by a standard four acid digest before multi-elemental analysis by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Varian VISTA-PRO).

QEMSCAN mounts were prepared and analysed for the head composite sample (six mounts) and sulfuric acid (six mounts), sodium cyanide (three mounts) and aqua regia (two mounts) leach residues.

## 3. Results and discussion

### 3.1. Suitability of the QEMSCAN for mineralogical analysis

The Species Identification Profile (SIP) used for the characterisation work in this study has been developed using the composite sample produced for this work, as well as other copper mineral samples as references. The SIP file is used to classify the elemental compositions of each pixel, as a mineral or phase (Gottlieb et al., 2000). The use of a single SIP file to classify all mineral abundance data, and the analysis of multiple blocks/samples produces highly reproducible results. These characteristics allow for the statistical analyses of QEMSCAN data which are necessary to distinguish between “significant” and “background” changes in mineral abundances (sampling error).

Both QEMSCAN and quantitative XRD (QXRD) are commonly used as mineralogical analytical tools. QEMSCAN was used in preference to QXRD in this analysis as (i) the micronising which is commonly used in QXRD preparation has been found to affect bornite crystallinity (unpublished work), (ii) commonly-used internal standards such as fluorite and corundum tend to exhibit peak overlaps with bornite or chalcocite and (iii) QXRD has a detection limit of approximately 1–2 mass% for most phases and error is introduced by not accounting for these minor phases in the abundance data. QEMSCAN on the other hand allows for the identification and quantification of minerals in a sample at very low abundances down to approximately 0.01 mass%.

The main limitations of the QEMSCAN are that (i) it cannot be used to characterise very fine material (e.g.  $P_{80} < 15\ \mu\text{m}$ ) because of the predominance of mixed X-ray spectra (from particles touching or because of boundaries between phases within a single grain) which cannot be classified into meaningful mineral classes (mounts from coarser material also need to be prepared carefully to prevent particles from touching and yielding a high number of mixed spectra) and (ii) phases are identified in the SIP file based on their chemistry alone and the absence of other information (such as structural information from XRD) means that a unique mineral name cannot be assigned in some cases. (It is possible that a grain size analysis of minerals of interest before and after leaching could also assist in identifying which minerals have been leached, however, errors in size measurements could be incurred when measuring two dimensional sections.)

These unique features of the QEMSCAN technique result in the presence of several phases in QEMSCAN data which are not strictly mineral or phase names, and appear vague in terms of their defini-

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