Methodological approach for mineralogical characterization of tailings from a Cu(Au,Ag) skarn type deposit using QEMSCAN (Quantitative Evaluation of Minerals by Scanning Electron Microscopy)

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ABSTRACT

Skarn type deposits are important potential resources for Cu, Au, and Ag as well as other strategic metals, which require accurate characterization of the mineralogy, texture and grade for successful processing and environmental management. The mineralogy of these deposits and of the resulting tailings has traditionally been examined using transmitted light microscopy, cathodoluminescence, X-ray diffraction, scanning electron microscopy, and electron probe microanalysis. In the present study, the Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN®) technology was applied to rapidly acquire spatially resolved mineralogical data from tailings associated with a Cu(Au,Ag) skarn type deposit. The resulting modal and textural data provided relevant additional information on the distribution of the ore minerals, including detail on the trace minerals, grain size distributions, and mineral associations. The following benefits of detailed mineralogical knowledge from this study can be pointed out: it improves the lithotyping of these complex deposit types and will benefit their ore processing strategies; it allows inferences to be made about the environmental behavior of the tailings, namely the acid mine drainage potential; it provides data about deportment of penalty and toxic elements, which are specifically As, Te, and Sb. Thus, particular applications of QEMSCAN include assessments of the acid consumption of the mineral assemblages (mainly assured due to calcite and dolomite) and of the abundance, distribution and mobility of potentially toxic elements, such as As.

1. Introduction

Mine production in the world is facing major operational challenges daily. The most efficient metal extraction processing route is directly related to the inherent mineralogical features of the ore being processed (Coetzee et al., 2011). Mineral deposits threaten to deplete their resources and environmental sustainability of the mining activity requires consideration not only of environmental regulations but also to determine the potentially toxic elements (PTE) and their behavior.

Process mineralogy, which forms part of the field of applied mineralogy, can be considered the practical application of mineralogical knowledge to aid mineral exploration and to predict and optimize mining and ore processing. According to Lotter et al. (2011), it is an integrated discipline which combines quantitative (and qualitative) mineralogical techniques with metallurgical test work. As such, process mineralogy is useful for the optimization of grades and recoveries within a working mine and has been applied to several deposit types, such as Au (e.g., Hausen, 1985; Zhou et al., 2004; Vaughan, 2004; Zhou and Gu, 2008), Pt-group elements (e.g., Cabri et al., 2005; Kraemer et al., 2015), phosphates (e.g., Leal-Filho et al., 1993; Sant'Agostino et al., 2001), and U (e.g., Bowell et al., 2011; Pownceby and Johnson, 2014). It has come into its own as a respected inter-discipline in the fields of mineralogy and metallurgy, and occupies an important place in both research and industry (Zhou et al., 2004).

In turn, mining activities generate a corresponding amount of tailings and other wastes that are considered as sources of environmental contamination. Tailings are mining wastes produced during the metal recovery process, often of very small grain size, composed of polymineralic assemblages, which consist mostly of different silicates, oxides, and sulfides (e.g., frequently abundant pyrite).

If the tailings are exposed to water and oxygen, the sulfides will oxidize and acid mine drainage (AMD) is produced, releasing acidity sulfate, and PTE, such as Cu, Zn, Pb, and As (e.g., Gleisner and Herbert Jr., 2002; Gleisner, 2005; Valente et al., 2013). The geochemical characteristics of these mining wastes, specifically size-dependent metal concentrations and solid-phase speciation, will determine the potential for PTE to migrate offsite and subsequent exposure and bioavailability to humans and biota (Schaider et al., 2007) as well as for biomagnification through the food chain. Thus, the tailings are one of the most typical environmental liabilities associated with mining activity as they represent a source of acidic and metal or metalloids contamination. They dictate landscape properties and, in general, all other environmental conditions of the mining sites. Moreover, sometimes disruptive processes may promote sudden and critical environmental impacts even at great distances (Azcue and Nriagu, 1995; Hatje et al., 2017). Often, the contamination associated with the evolution of tailings ends with the complete degradation of the ecosystems, since AMD is a multifocus type of pollution (Gray, 1998). Such environmental consequences justify an exhaustive characterization of tailing wastes, including physical, chemical and mineralogical attributes, in order to prevent their pernicious evolution and to plan monitoring and remediation strategies.

In the last years a variety of modern analytical techniques have been developed and/or applied to the characterization of tailings, including X-ray absorption fine structure (XAFS) spectroscopy, polarized energy-dispersive X-ray fluorescence (pED-XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM), electron probe microanalysis (EPMA), and inductively coupled plasma-mass spectrometry (ICP-MS) (e.g., Ostergren et al., 1999; Morin et al., 1999; Schaider et al., 2007). However, due to its manual or laborious nature, the mining industry has found several problems with conventionally routine methods, particularly in geometallurgy or mineral processing.

Scanning Electron Microscopy (SEM) based Automated Petrography systems have been developed since the 1980s at Australia's Commonwealth Scientific and Industrial Research Organization - CSIRO (Hoal et al., 2009). Automated systems have numerous advantages, like their statically robustness, reliable mineral identifications, round-the-clock operations, high throughput, high degree of flexibility, multiple measurements modes, built-in quality-control programs, and data/information that can be useful in a diversity of industrial and research domains (Fennel et al., 2005). In this context, QEMSCAN has made huge advances in speed and accuracy in the last years, becoming a dominant mineralogical imaging system (e.g., Camm et al., 2004; Liu et al., 2005; Fennel et al., 2005; Goodall and Scales, 2007; Pascoe et al., 2007; Goodall, 2008; Rollinson et al., 2011; Santoro et al., 2014; Anderson et al., 2014). Although, it is gaining increasing recognition in other areas of research (e.g., Butcher et al., 2003; Pirrie et al., 2004, 2009; Al-Otoom et al., 2005; Sliwinski et al., 2009; Šegvić et al., 2014; Potter-McIntyre et al., 2014; Intellection (Pty.) Ltd, n.d., http://www.intellection.com.au/).

QEMSCAN creates phase assemblage maps of a specimen surface by scanning the electron beam along a predefined raster scan pattern and acquiring an energy-dispersive X-ray spectrum at each measurement point (Gottlieb et al., 2000). Low-count energy-dispersive X-ray spectra (EDX) are generated and provide information on the elemental composition at each measurement point. The elemental composition in combination with back-scattered electron (BSE) brightness and X-ray count rate information is converted into mineral phases (Gottlieb et al., 2000). So, QEMSCAN data includes bulk mineralogy and calculated chemical assays. Moreover, by mapping the sample surface, textural properties and contextual information, such as particle and mineral grain size and shape, mineral association porosity, and matrix density can be calculated, visualized, and reported numerically. Consequently, one of the main benefits is the acquisition of spatially resolved mineralogical data inferred from chemical spectra. This provides increased information on mineral phases (particularly with respect to trace minerals), fully quantitative and statistically valid data on ore mineral abundances, particle size and shape distributions, and quantitative data on mineral associations (Rollinson et al., 2011).

In industry, the technique is used primarily to gather information on the abundances and interrelations of ore and gangue minerals, the identification of gangue components that are detrimental to the mineral processing, the degree of liberation of ore components, and the identification of issues that affect recovery and losses during mineral processing (Rollinson et al., 2011).

QEMSCAN has been mainly applied to Au deportment studies (e.g., Butcher et al., 2000; Chryssoulis, 2001; Gu, 2003; Goodall, 2008) with only a few studies on Cu deportment (Fennel et al., 2005; Kalichini, 2015). Moreover, working with tailings is a difficult task, as representability and data integrity could be compromised by the small size and heterogeneity of the particles. According to Goodall (2008), such diagnostic tool gives insights into everything from what is being lost to why it is being lost and whether it will cause problems in the tailings, providing valuable information with geological, economic and environmental relevance. These issues justify the novelty and scientific value of the present study as QEMSCAN is applied to Cu enriched tailings to provide different types of useful information, namely the distribution of valuable elements and the grain size of key minerals as a diagnostic tool in identifying inefficient mineral processing, the liberation characteristics of key minerals to define root cause for losses, and the presence and nature of environmentally sensitive minerals, such as sulfides and composing PTE.

The aim of the present study is applying QEMSCAN analyses to the mineralogical characterization of tailings associated with a Cu(Au,Ag) skarn type deposit. Specific objectives comprise the determination of the modal mineralogy and the description of the mineralogical variation within this type of materials. Furthermore, this study explores the advantages and limitations of QEMSCAN applied to mine tailings and environmental issues such as the geochemical and mineralogical properties that control AMD generation and mobilization of pollutants.

2. Mineralogy and processing of skarn type deposits

Skarn type deposits are mineralogically very complex and variable, which is one of the major issues for the development of successful ore processing strategies. They are characterized by calc-silicate mineralogy, represented by the presence of garnet and pyroxene (Skarns and skarn deposits, n.d., http://www.science.smith.edu/geosciences/skarn/). Skarn are among the most studied deposits from the standpoint of metallogenesis (e.g., Meinert et al., 2000). Most economic skarn deposits form via metasomatism of country rocks surrounding intrusions, where the latter provide for hydrothermal fluids and are a source of the ore metals (e.g., Ciobanu and Cook, 2004; Pons et al., 2009). Many studies are focused on the geochemical properties of the intrusions and on the question as to whether specific magma sources are required to form economically significant accumulations of metallic ores of Cu, Pb, Zn, Au, Fe, Mo, W, and Sn (Meinert et al., 2005), as well as in other industrial minerals (e.g., wollastonite, graphite, asbestos, talc, fluorite).

In any mining extraction scenario, the response of the ore feed to different processing methods is a direct function of the mineralogical components and particle size distributions in the ore material (Rollinson et al., 2011). In order to fully understand the operating requirements for processing, such as the acid consumption, the likely recovery, the presence of detrimental substances or the energy consumption, it is important to develop an understanding of the mineralogical variability of the ore feed. The increasing scarcity of high grade deposits and the need of extracting all the metallic ore value the deposits deposit due to higher metal prices stimulate the interest to understand the cause for metal losses in processing operations. Therefore, it is very important to invest additional effort on performing elemental deportment characterization.

By using QEMSCAN, this work focuses interest on the mineralogical characterization of tailings associated with a Cu(Au,Ag) skarn type

deposit of Eocene-Oligocene age formed by metasomatic alteration of Cretaceous limestone. As for the mineralization, the site is delimited by leached, oxidized, mixed and primary sulfide (hypogene) zones. Bornite, chalcanthite, chalcocite and copper oxides are the main Cu-bearing minerals.

3. Methodological approach for QEMSCAN analyses

Samples preparation followed the standard procedures provided by FEI for QEMSCAN® (Gottlieb et al., (2000). Nine samples from tailings associated with a Cu(Au,Ag) skarn type deposit were analyzed. They were set up to measure particles in the range of −500 μm/+6 μm.

3.1. QEMSCAN operation modes

The samples were measured using FEI's QEMSCAN technology and iDiscover™ off-line image analysis software. The software package iDiscover consists of four software modules: Datastore Explorer (data management module), iMeasure (measurement module, SEM and EDS control), iExplorer (data processing and classification tools, mineral database management, reports), and SIP (species identification protocol) editor.

Mineralogical analysis was carried out through particle identification by backscattered electron (BSE) brightness and X-ray analysis for quantitative mineral identification. Automated mineral analyses were performed using the System QEMSCAN 650 FEG of the FEI's Center of Excellence for Natural Resources in Brisbane, Australia. Particle mapping modes, including Particle Mineral Analysis (PMA) and Trace Mineral Search (TMS) provide information on spatial relationships of minerals, namely liberation and association data and offer a visual representation of mineral textures (North American Nickel Inc, 2013). PMA is a two-dimensional mapping analysis, which is very useful to determine liberation and characteristics of a set of particles. TMS is an analytical protocol described by Butcher et al. (2000), which reports trace minerals that can be located by optimized search for bright phases. This is also known as Bright Phase Searching, which allows identification of trace phases whereas only undertaking X-ray mapping on a manageable number of particles (Goodall, 2008). The following stepping intervals were used in the measurement set up: PMA - 3 μm (pixel spacing), and TMS - 1.2 μm (pixel spacing). The finer stepping interval was used in the TMS measurement to ensure that fine intergrowths would not be "stepped over".

QEMSCAN's PMA measurement mode was set up to quantify all particles, within each field of view that were in the size range of −500 μm/+6 μm until a maximum particle count of 15,000 was obtained. Differences in measurement times between samples do vary (S1). The main reason is the differences in particle sizes (larger particles will have a much higher number of X-ray acquisition points measured). Another contributing factor is the variation in the number of particles outside of the specified size range, as these still need to be scanned but don't require X-ray data acquisition time (the total measurement time includes both BSE scanning and X-ray acquisition time). A full review of the QEMSCAN system for process mineralogy applications has been provided by Gottlieb et al. (2000).

QEMSCAN's TMS (Trace Mineral Search) measurement mode is an optimized search for bright phases. It was set to measure all particles containing phases with a backscattered electron (BSE) brightness greater than that of pyrite. It is used to increase the particle statistics of the trace element mineral phases. The samples measurement was set up to be terminated once it had been measuring for a total of 8 h. Differences in the number of particles measured do vary between samples (S2). One reason is the difference in the particle size distributions of the bright phases.

Table 1
General deposit information as background data for QEMSCAN analysis.

Source material	Mining method	Geological setting	Mineralization	Sulfides	Skarn/gangue mineralogy
Tailings	Open pit	Skarn deposit associated with a contact metasomatic process generated by intrusion of porphyry-style monzonite plutons	Cu(Au, Ag)	Chalcopyrite Chalcocite Bornite Pyrite Pyrrhotite Molybdenite Galena Sphalerite	Garnet Diopside Magnetite Quartz Plagioclase Ortoclase Calcite

3.2. Geological background

The linkages between geological and metallurgical information is very important for understanding of the significance of mineralogical parameters and their impact on metallurgical performance. The geological background is very useful to define the most appropriate analysis methodology and workflow to follow. It also helps to understand and improve mineralogical parameters for textural characterization of the ore and host rocks. Table 1 is a summary of the geological information available that describes this particular deposit.

3.3. Mineral analyses

The method is capable of identifying most rock-forming minerals in milliseconds by collecting their characteristic X-ray spectra, which are then compared to entries in a database containing the species identification profiles in order to assign the spectrum to a particular phase. Minerals are measured and then grouped into a more reportable list (S3).

4. Results and discussion

4.1. Mineralogy

Sulfides occur as chalcopyrite, bornite, chalcocite/digenite, covellite, pyrite, pyrrhotite, molybdenite, galena, and sphalerite. Additional Cu-bearing minerals of interest were detected. These included (but are not limited to) enargite and tennantite. Cu is present as sulfides, arsenosides, oxide-hydroxides and silicate minerals. Of the three elements of interest (As, Te, and Sb) the following were detected in some of the samples: As-bearing phases (enargite and tennantite); Te-bearing phases (hessite and tellurobismuthite); two different mineral phases were detected both contained Sb and W. One phase is most likely to be tungstibite.

Gangue minerals consist mainly of quartz, plagioclase, orthoclase, garnet (predominantly andradite), and calcite. One of the samples (Tailing-4) has lower contents of garnet and calcite comparison to the other samples. Other predominant phases include siderite (Ca-bearing), chlorite and other clay minerals, micas, and both Fe and Ti oxide minerals. Unclassified phases are mainly intergrowth phases. Except where noted, these phases do not include any minerals nor elements of interest.

QEMSCAN was used to provide quantitative modal mineralogy. The modal analysis (in wt%) are presented in Table 2. Most samples are similar in mineral composition and abundance and are dominated by garnet (16.4–40.2%), plagioclase (11.8–20.3%), quartz (9.3–15.2%), orthoclase (5.7–12.7%), and calcite (4.4–17.2%). Sample Tailing-4 differs from the others mainly because it contains minor amounts of garnet (2.9%) and trace amounts of calcite (0.4%), and abundant quartz

Table 2

Quantitative modal mineralogy (wt%) of the analyzed samples.

SAMPLE	Tailing-1	Tailing-2	Tailing-3	Tailing-4	Tailing-5	Tailing-6	Tailing-7	Tailing-8	Tailing-9
Tellurium minerals	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Chalcopyrite	1.1	0.3	0.2	0.4	0.4	0.3	0.5	0.2	0.7
Bornite	0.1	0.0	0.1	0.2	0.0	0.1	0.1	0.1	0.1
Chalcocite/digenite	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0
Covellite	0.0	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0
Other copper minerals	0.3	0.4	0.7	0.5	0.6	0.1	0.1	0.2	0.1
Iron sulfides	0.5	0.3	0.3	0.2	0.3	0.4	0.2	0.1	0.2
Other sulfides	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.1
Quartz	14.6	14.0	15.0	23.2	13.5	15.2	12.7	12.2	9.3
Orthoclase	11.7	11.5	10.2	22.2	12.7	9.6	10.8	8.5	5.7
Plagioclase	18.0	19.1	15.3	28.5	16.1	20.3	19.2	18.5	11.8
Mica	1.4	2.6	2.8	4.5	2.4	2.1	2.0	2.2	2.4
Clay	2.2	2.1	2.2	6.0	2.1	0.8	1.2	1.3	1.0
Chlorite	1.2	1.6	1.5	1.9	1.8	1.2	2.1	1.6	1.4
Amphibole/hornblende	0.7	0.9	0.6	0.1	1.2	0.9	0.9	1.1	0.7
Apatite	0.2	0.2	0.2	0.0	0.2	0.3	0.2	0.2	0.2
Garnet	16.4	21.8	31.5	2.9	28.0	25.2	22.8	19.6	40.2
Pyroxene	2.4	2.2	2.2	0.3	3.3	3.0	2.2	2.8	3.3
Epidote	0.4	0.9	0.3	0.2	0.7	0.6	0.6	0.6	0.9
Fe-Ti oxides	6.8	4.6	7.1	3.4	7.2	3.5	3.9	4.7	5.0
Calcite	13.0	10.5	5.8	0.4	4.4	10.0	10.5	17.2	10.8
Dolomite-ankerite	0.3	0.2	0.1	0.0	0.2	0.2	0.2	0.2	0.1
Siderite	3.1	1.2	0.7	0.5	0.7	1.7	2.3	2.3	0.6
Others	0.2	0.2	0.3	0.6	0.6	0.2	0.2	0.3	0.2
Unclassified	5.9	5.4	2.9	4.2	3.8	4.4	7.2	5.9	5.4

(23.2%), plagioclase (28.5%), and orthoclase (22.2%).

In general, the samples have minor to trace amounts of mica (1.4–4.5%), chlorite (1.2–2.1%), other clay minerals (0.8–6.0%), pyroxene (0.3–3.3%), and siderite (0.5–3.1%). They have minor amounts of Fe−Ti oxides (3.4–7.2%). Trace amounts of chalcopyrite (0.2–1.1%) are also present. Unclassified minerals are predominantly boundary phases (i.e., mixed X-ray emission spectra) that could be further refined. Some unidentified minerals are also present that could warrant further attention, but these occur only in trace amounts and do not include any elements of interest.

4.2. Estimated average grain sizes

Size calculations are based on stereological-corrected estimates of the diameter of a sphere of equivalent volume to the particles or grains, assuming that the measurements are random cross-sections through randomly oriented particles/grains. As such, it is only really valid when averaged across a large population of cross-sections. Since particles are rarely spherical, the actual particles will be both larger (in some axis) and smaller (in some other axis) than the estimated size (Table 3).

4.3. Elemental deportment

Elemental deportment is becoming a critical requirement for ore characterization projects in order to provide understanding of minerals that do not contribute to grade, as well as penalty elements that can cause environmental concerns with tailings storage (e.g., As (FEI, n.d., http://www.fei.com/natural-resources/ore-characterization/)).

Element deportment data were obtained using the PMA measurement mode and the results used to identify opportunities for additional recovery. Cu occurs in sulfide, arsenide, oxide-hydroxide and silicate minerals. Cu sulfides include chalcopyrite, bornite, chalcocite/digenite and covellite. However, chalcopyrite is the main contributor, hosting 24.14–80.85 wt%, with samples Tailing-1 and Tailing 3 displaying the highest (80.85 wt%) and lowest (24.14 wt%) values, respectively (Table 4). Bornite hosts 9.52–33.33 wt% of Cu, except in sample Tailing-5. Covellite hosts 17.07 wt% in sample Tailing-4, with minor amounts (2.13–9.52 w t%) in samples Tailing-1 to Tailing-3 and Tailing-6. Chalcocite/Digenite hosts 19.05 wt% of Cu in sample Tailing-

8, with minor amounts (2.44–6.67 wt%) in samples Tailing-4 and Tailing-7. Other Cu species are in the range of 2.13 to 20.83 wt%, with the highest values in sample Tailing-5. The presence of Cu-silicate phase(s) is more common in samples Tailing-1 to Tailing-5 and Tailing-8 (4.26–20.69 wt%) and less so in the samples Tailing-6, Tailing-7 and Tailing-9. Cu-hydroxide minerals occur mainly in Tailing-3 (13.79 wt%). Other samples contained minor and trace amounts only.

QEMSCAN can be used in the Cu deportment characterization for process troubleshooting and optimization. Understanding the fundamental Cu populations and mineral associations in feed material can be applied to highlight areas of inefficiency within the process as a forensic tool to decrease elemental losses, improve overall recoveries and enhance the viability of the project. The evaluation of the mineralogical association is critical, and could be used as a guideline to plan a mineral processing route, avoiding time and money losses (Santoro et al., 2014).

Modern mining production faces a wide variety of operational challenges, with increasingly mineralogical and texturally complex mineral deposits. Therefore, mining sustainability requires more attention to penalty elements. Valuable Cu resources can host significant quantities of As, Te or Sb−W, which require strict environmental regulations for management.

There is little available information on the changes in speciation and deportment of As, Te or Sb−W in tailings. As was detected in three of the nine samples (Tailing-4, Tailing-7 and Tailing-9) into two mineral phases: enargite and tennantite. Some of the enargite may contain traces of Zn. The presence of As in tailings may be also associated with Fe-bearing particles, probably ferric oxide and ferric oxyhydroxide (Moreno et al., 2007). For Te there is also two identified phases: hessite and tellurobismuthite. However, it was only detected in sample Tailing-7. Sb is present in a couple of mineral phases that have yet to be positively identified and may warrant further investigation. Due to the limited number of As, Te, and Sb particles found the elemental deportment data do not allow an accurate representation of the actual proportions and grades and are only indicative (S4).

Only four particles containing enargite (Cu_3AsS_4) and tennantite (($Cu,Fe)_{12}As_4S_{13}$) (Fig. 1) which are the most common As-bearing minerals associated with Cu-sulfide ore bodies, (Piret, 1999), were measured across all samples. The grains are only a few pixels in size and are hard to see in the images provided; they are circled in red for easier

Table 3
Estimated average grain size (μm) of the analyzed samples.

Sample	Tailing-1	Tailing-2	Tailing-3	Tailing-4	Tailing-5	Tailing-6	Tailing-7	Tailing-8	Tailing-9
Calculated ESD particle size (μm)	47	86	122	125	119	102	137	74	133
Tellurium minerals	0	4	4	0	0	4	4	4	5
Chalcopyrite	43	31	39	42	46	21	37	25	37
Bornite	9	10	35	20	14	14	17	22	32
Chalcocite/digenite	0	6	11	7	7	9	9	15	6
Covellite	10	12	26	15	8	9	7	7	7
Other copper minerals	8	8	9	6	9	6	6	6	6
Iron hulfides	28	31	113	87	46	40	31	20	55
Other hulfides	7	36	0	8	23	11	14	7	35
Quartz	23	28	22	25	27	26	23	24	29
Orthoclase	24	26	19	26	31	24	27	26	32
Plagioclase	30	32	23	29	32	30	30	32	33
Mica	10	13	14	16	14	13	11	12	18
Clay	10	10	10	12	10	8	8	9	9
Chlorite	10	12	12	9	12	13	17	13	14
Amphibole/hornblende	9	10	12	11	13	8	8	8	8
Apatite	13	16	25	19	21	25	15	20	17
Garnet	29	44	61	43	58	48	46	38	48
Pyroxene	19	17	24	11	21	14	16	17	22
Epidote	12	27	15	36	25	22	17	16	14
Fe-Ti oxides	21	26	29	16	27	25	26	24	35
Calcite	34	42	76	127	37	30	34	40	38
Dolomite-ankerite	17	17	81	10	15	12	20	14	15
Siderite	19	18	10	11	10	21	24	21	13
Others	8	9	12	30	17	10	7	11	10
Unclassified	8	8	8	8	8	7	8	8	8

location. They are grouped within other Cu phases, but there are also intergrowths of Cu phases in this same mineral group.

The oxidation behavior of enargite has been studied by several authors (e.g., Filippou et al., 2007; Lattanzi et al., 2008; Di Benedetto et al., 2011; Plackowski, 2014), taking into account its implications for ore processing. Numerous studies confirm the general rule that the As (III) species are more soluble in the environment than the As(V) species (Magalhães, 2002; Bhattacharya et al., 2001). So, based on the solubility, As(III) species such as tennantite would be more problematic than As(V) species, such as enargite. Penalties incurred by miners for arsenic in concentrates have increased significantly because the removal and disposal of As is difficult and costly for smelters (Bruckard et al., 2010). The precipitation of scorodite has been widely accepted as currently the most suitable method for stabilizing arsenic in the metallurgical treatment of copper concentrates (Ferron and Wang, 2003).

Te-phases are considered to be toxic to humans (Taylor, 1996). Te and other toxic metal(loid)s, such as Cu, are enriched at the tailings surface, potentially as efflorescent salts or sorbed species, with a predominance of Te(VI), which is less soluble and toxic than Te(IV), likely responsible for the low percentages of bioaccessible Te in tailings (Knight et al., 2015). Fig. 2 reveals that only three particles containing Te-bearing minerals (hessite or tellurobismuthite) were measured across all samples. All particles were found within the single sample: Tailing-7. The grains are approximately 6–8 μm. They are black in the

images provided and circled for easier location. The middle particle contains tellurobismuthite and the other two particles contain hessite.

Sb has properties similar to As with some of its compounds toxic to humans (Marmolejo-Rodríguez et al., 2013). In turn, traditionally, W has been assumed to be relatively insoluble in water and nontoxic, however, some species of W can move readily though soil and groundwater under certain environmental condition, and, therefore, it should be considered as a contaminant of concern (e.g., Koutsospyros et al., 2006; American Chemical Society, 2009; U.S. Environmental Protection Agency, 2014). However, the potential environmental effects of W are essentially unknown (Strigul et al., 2005). Fig. 3 reveals that two particles were detected in the Tailing-9 sample. As with the all of particles detected, a large area of unclassified pixels (light grey) surrounds the pixels that were detected containing Sb (darker grey).

QEMSCAN X-ray pixel information (Fig. 3) is presented (to the left) for two different phases detected within the Sb−W bearing particle. The brighter BSE phase X-ray spectrum is at the top of the page and the duller BSE phase X-ray spectrum is presented below the BSE image. The duller phase potentially contains either S or Mo in the mineral chemistry, along with Sb and W. The brighter phase contains Sb and W and potentially oxygen. A higher resolution EDAX analysis of these phases could be used to confirm the mineralogy.

Fig. 4a displays particles that were measured during the TMS measurement mode and includes small grains containing Sb, whereas

Table 4
Cu deportment data (wt%).

| Sample	Tailing-1	Tailing-2	Tailing-3	Tailing-4	Tailing-5	Tailing-6	Tailing-7	Tailing-8	Tailing-9
Chalcopyrite	80.85	52.38	24.14	36.59	58.33	50.00	63.33	38.10	75.00
Bornite	10.64	9.52	27.59	26.83	0.00	33.33	26.67	33.33	21.88
Chalcocite/digenite	0.00	0.00	0.00	2.44	0.00	0.00	6.67	19.05	0.00
Covellite	2.13	9.52	3.45	17.07	0.00	5.56	0.00	0.00	0.00
Enargite	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Tennantite	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cu-silicate	4.26	19.05	20.69	7.32	16.67	0.00	0.00	4.76	0.00
Cu-O-OH	0.00	0.00	13.79	0.00	4.17	5.56	0.00	0.00	0.00
Other copper minerals	2.13	9.52	10.34	9.76	20.83	5.56	3.33	4.76	3.13
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
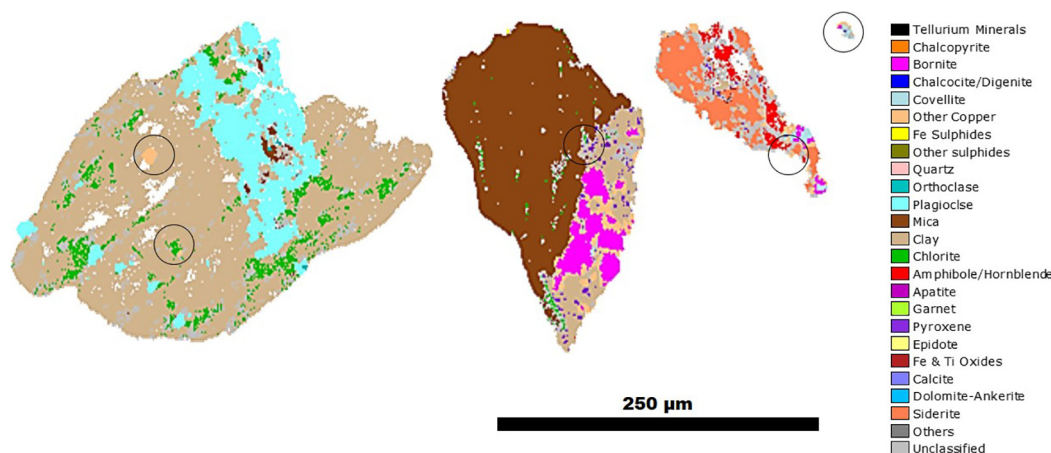
Fig. 1. Mineral map of particles containing As-bearing minerals.

Fig. 4b shows a BSE image highlighting the Sb-bearing. Fig. 5 shows particles that were detected during the PMA measurement mode and includes small grains of a mineral phase containing Sb–W. This phase is captured as "others" but largely also includes the unclassified area of the particle as well. The BSE image is displayed below.

4.4. Characterization of acid generating and neutralizing minerals

Acid mine drainage (AMD) is a serious environmental problem, which has been extensively studied globally (e.g., Gieré et al., 2003; Salmon and Malmström, 2004; Salzsauer et al., 2005; Kock and Schippers, 2006; Smuda et al., 2007; Korehi et al., 2014; Anawar, 2015; Erguler and Erguler, 2015). The major cause of AMD is the accelerated oxidation of sulfide minerals, especially pyrite (FeS_2), resulting from the exposure of these minerals to both oxygen and water, as a consequence of the mining and processing of metal ores. The study of the oxidative dissolution of sulfides, with emphasis on the behavior of pyrite, has been the subject of numerous research studies (Evangelou and Zhang, 1995; Kirby et al., 1999; Luther, 1987; Pratt et al., 1994; Rimstidt and Vaughan, 2003).

AMD typically has low pH and high concentrations of sulfate, iron and other metals (Blowes et al., 2003; Nordstrom, 2011). The production of AMD from tailings is strongly dependent on distribution of acid

and alkalinity generating minerals.

The ratio of these acid generating and neutralizing minerals as well as their locked or liberation characteristics will determine if a sample will be a net acid generator, thus assisting with environmental planning, closure planning, and rehabilitation projects (Acid generating minerals, n.d., http://www.sgs.com). According to the U.S. Environmental Protection Agency (1994), two main components should be kept in mind when evaluating information on acid generation potential: (i) components affecting the total capacity to generate acid, which include amount of acid generating minerals (sulfides), amount of acid neutralizing minerals, amount and type of potential contaminants, and (ii) components affecting the rate of acid generation, which include type of sulfide minerals (e.g., crystal form, defects, trace elements) (Evangelou and Zhang, 1995), type of carbonates (and other neutralizing minerals), particle size and surface area available for reaction, available water and oxygen, and bacteria. A review of the main controlling factors on acid generation potential can be found in Kwong and Lawrence (1994).

A list of the mineral groups that compose the tailing wastes is given in Table 5. Acid generating minerals correspond to chalcopyrite, bornite, chalcocite/digenite, covellite, pyrite, phyrrhotite and molybdenite, and other sulfides. Samples Tailing-6 (0.9 wt%) and Tailing-1 (1.9 wt%) have the lowest and highest contents of acid generating
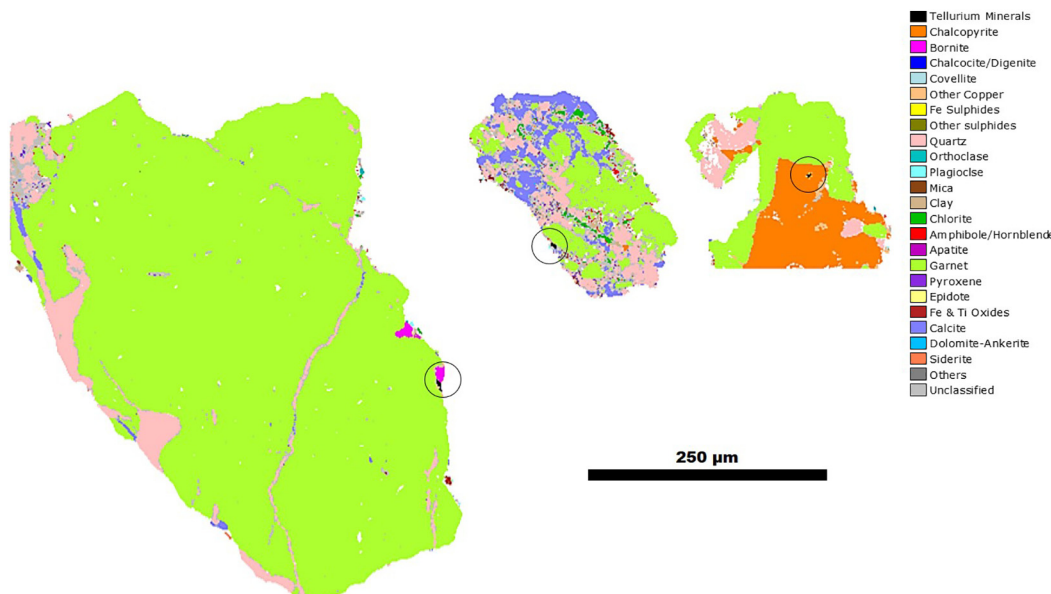

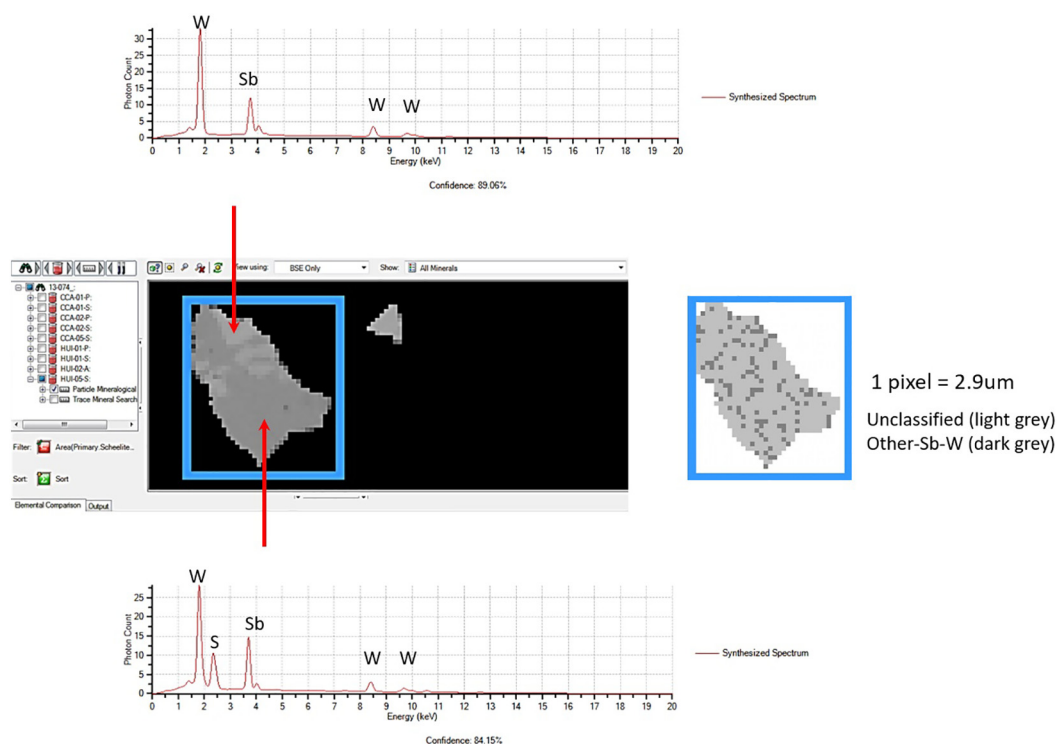**Fig. 2.** Mineral map of particles containing Te-bearing minerals.

Fig. 3. QEMSCAN backscatter image of particles containing Sb-W-bearing minerals detected in the Tailing-9 sample.

minerals, respectively. The group named "Acid neutralizing minerals" comprises only the highly soluble carbonates, which are the most effective contributors for neutralization: calcite and dolomite. Samples Tailing-4 (1.3 wt%) and Tailing-8 (20.3 wt%) have the lowest and highest contents of these carbonates, respectively. With less neutralizing capacity, there are also other groups, like exoskarn and endoskarn minerals. Exoskarn minerals are garnet, pyroxene, apatite, muscovite and chlorite; samples Tailing-4 (10.1 wt%) and Tailing-9 (50.0 wt%) have the lowest and highest contents of exoskarn minerals, respectively. Endoskarn include olivine, epidote, amphibole, and talc; samples Tailing-4 (0.4 wt%) and Tailing-5 (2.6 wt%) have the lowest and highest contents of endoskarn minerals, respectively. Fe−Ti oxides include mainly magnetite, hematite, ilmenite and titanite, with samples Tailing-4 (3.5 wt%) and Tailing-5 (7.5 wt%) showing the lowest and highest contents of Fe−Ti oxides, respectively. Other no-ore are quartz, feldspars, clay minerals (e.g., kaolinite, smectite, illite, pyrophyllite and montmorillonite), and minor mineral phases, with samples Tailing-9 (29.8 wt%) and Tailing-4 (83.3 wt%) showing the lowest and highest contents of these minerals, respectively.

Based on this data it is expected high capacity for neutralizing the acidity generated by the lower amounts of sulfides. In fact, such as calcite and dolomite are highly soluble, thus strongly contributing to alkalinity generation (Kwong, 1993). Though the classification presented in Table 5, it should be reinforced the potential role of calc-silicate minerals from the exoscarn and endoscarn for producing alkalinity. The same happens with some minerals of the group "Gangue" (such as plagioclase and clay minerals). This last group can contribute to acid consumption by surface reactions, namely cationic exchange. However, it should be noted the opposite behavior of Fe-host minerals (e.g., siderite) since the hydrolysis of iron may liberate protons, canceling out neutralizing capacity.

Prediction of AMD potential is a difficult task (Parbhakar-Fox and Lottermoser, 2015), mainly due to heterogeneity of the materials and the high number of controlling factors. Therefore, QEMSCAN data should be use to complement the classic static and kinetic tests usually performed in mining industry (Jambor, 1994, 2003; Jambor et al.,

2002, 2006, 2007) in order to improve the accuracy of AMD prediction.

In the long term, the goal of the tailings management is to prevent mobilization and release of PTE into the environment (e.g., Sahuquillo et al., 2003; Sutherland, 2010). Therefore, it is necessary to promote this type of automated mineral analysis as geochemical diagnosis, which allows determining both the concentration levels of various elements and the main generators of AMD. On the other hand, this diagnosis provides a very useful background to propose alternative solutions (e.g., treatment of AMD or add value to mining wastes) for the disposal of mining tailings, which presently constitute an environmental liability.

The present study shows that the problems associated with the mining tailings included the liberation of metals (Fe, Cu, Zn, and Pb) and metalloids (As, Te and Sb−W), and occasionally the generation of AMD. However, although the metalloids were detected in very low concentration in the tailings, they could be mobilized from them and enter the environment.

Future work should evaluate the geochemical mobility of elements present in mining tailings (e.g., Tessier et al., 1979; Filgueiras et al., 2002) to determine their potential as environmental contaminants, their bioavailability and thus their toxicity.

5. Conclusions

This study showed the potential of QEMSCAN to analyze tailings regarding aspects with industrial, economic, and environmental relevance. Namely, it emphasizes the usefulness of QEMSCAN analyses to maximize recovery processing plants by determining the mineralogical controls that cause the loss of valuable minerals in the tailings. Therefore, this tool is essential in determining which mineral phases are lost in the tailings, which is very useful also to avoid strategic minerals being lost.

The methodology was applied to the tailings of a Cu(Au,Ag) skarn deposit, providing spatially resolved quantitative modal mineralogy, textural information and detail on the deportment of elements of interest (Cu, As, Te and Sb−W). In addition, relevant new information has
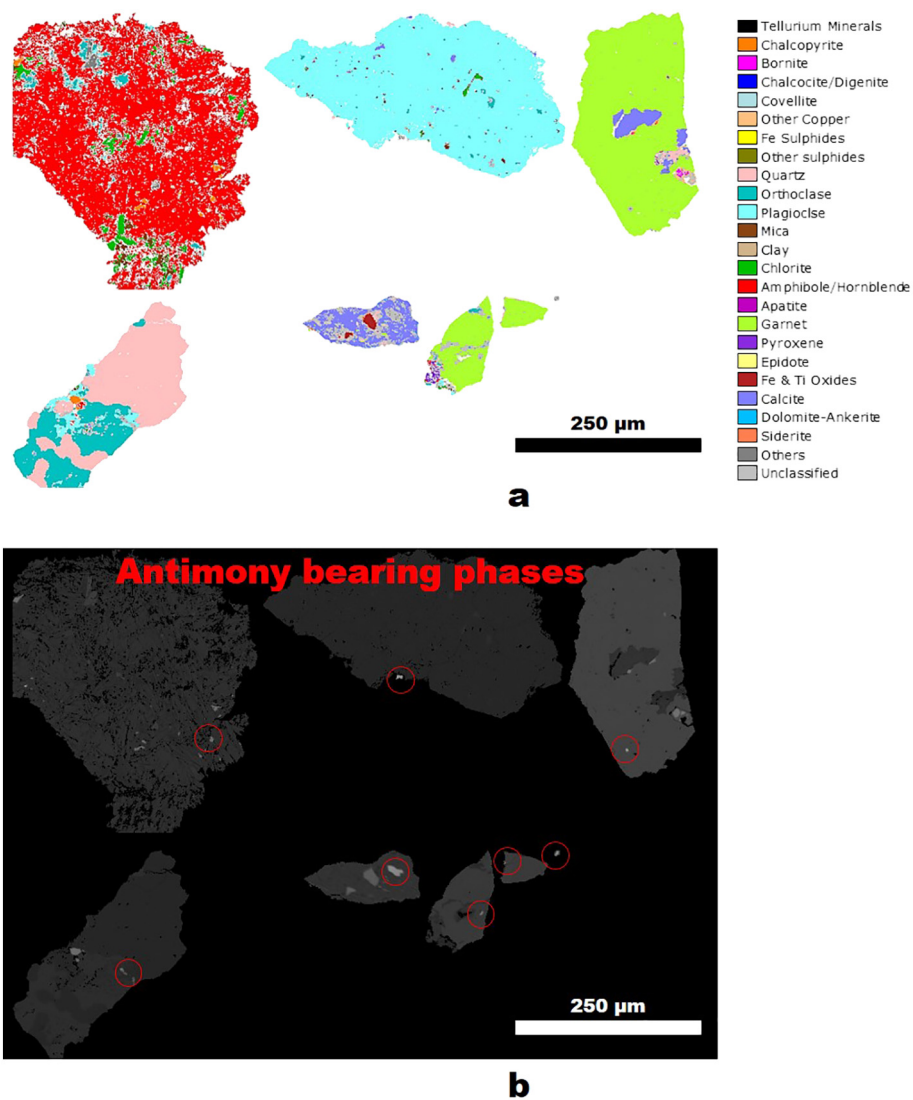
Fig. 4. (a) Mineral map of small grains containing Sb (TMS measurement mode). (b) BSE image of the Sb-bearing grains (TMS measurement mode).
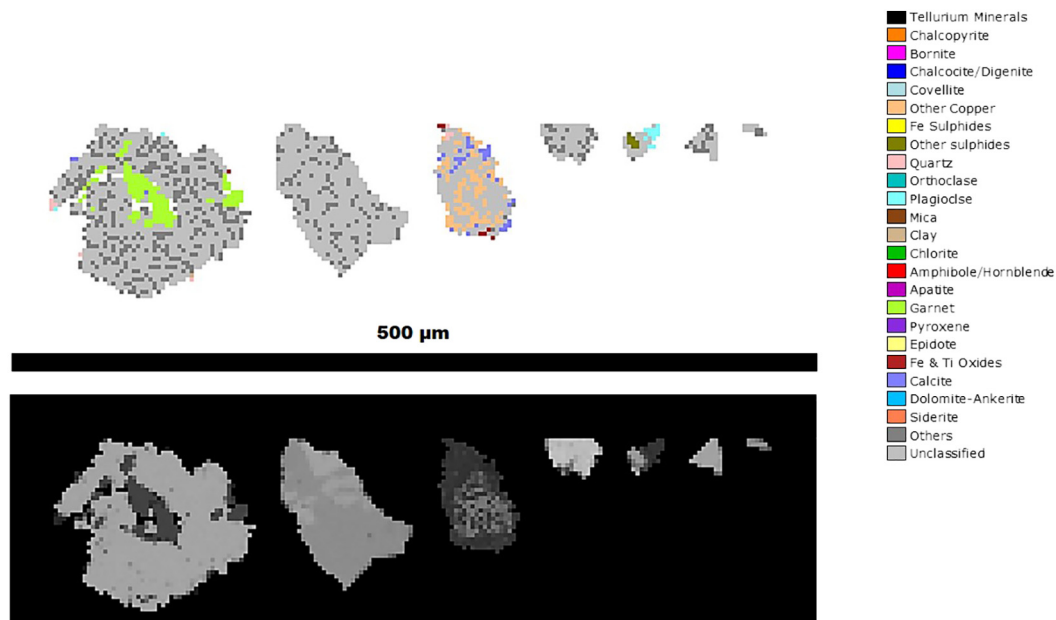

Fig. 5. Above and below, mineral map and BSE image, respectively, of small grains containing Sb−W (PMA measurement mode).

Table 5

Quantitative modal mineralogy (wt%) of the different types of mineral groups.

SAMPLE	Tailing-1	Tailing-2	Tailing-3	Tailing-4	Tailing-5	Tailing-6	Tailing-7	Tailing-8	Tailing-9
Mineral group									
Acid generating	1.9	1.1	1.4	1.4	1.3	0.9	1.1	0.7	1.1
Acid neutralizing	16.7	12.4	6.8	1.3	5.6	12.3	13.5	20.3	11.8
Exoskarn	22.9	30.1	39.0	10.1	37.1	33.7	31.6	28.4	50.0
Endoscarn	1.7	2.4	1.4	0.4	2.6	1.8	2.1	2.3	2.3
Fe-Ti oxides	6.9	4.8	7.3	3.5	7.5	3.6	4.1	5.1	5.1
Gangue	49.9	49.3	44.2	83.3	45.9	47.7	47.7	43.3	29.8
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

been collected on the distribution of minor and trace mineral phases. The elemental deportment reveals that As, Te, and Sb−W are present in several mineral phases, which were identified and quantified in the mining tailings. Also, it enabled a description of their mineral associations, grain size distributions and liberation characteristics. From this information, predictions on theoretically achievable grades and recoveries of tailings can be made in order to design an appropriate geometallurgical program.

As is distributed in enargite or tennantite, Te-bearing minerals were identified as hessite and tellurobismuthite. Sb-W-bearing phases (likely to be tungstibite) were identified as well, although they have yet to be clarified. In general, these elements of interest could be targeted as a potential by-product subject to further investigation.

The obtained results showed that the complex mineralogy of skarn type deposits can be quantified by QEMSCAN. Furthermore, this approach contributes to improve the geological lithotyping for skarn type deposits as QEMSCAN data can provided much greater detail on the elemental variability within and between the lithotypes.

Analysis by XRD remains important to validate the QEMSCAN data; however, quantitative XRD analysis alone is not able to provide the spatial information, the distribution of trace minerals and the distribution of low concentrations of metals within the minerals.

Similarly, this type of information is required for environmental characterization of ore deposits and mine wastes. In the present case it greatly assists in the characterization of the tailings, which are an important environmental liability and will support decisions on monitoring and remediation technical approaches.

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