Minerals Engineering 95 (2016) 138–145

Contents lists available at ScienceDirect

Minerals Engineering

journal homepage: www.elsevier.com/locate/mineng

# An estimation of the variability in automated quantitative mineralogy measurements through inter-laboratory testing

R. Lastra *, D. Paktunc

*Natural Resources Canada, CanmetMINING, 555 Booth St., Ottawa K1A 0G1, Ontario, Canada*

ARTICLE INFO

*Article history:*
Received 15 February 2016
Revised 2 May 2016
Accepted 26 June 2016
Available online 2 July 2016

ABSTRACT

Presently there are about 160 installations of automated mineralogy instruments such as QEM∗SCAN, MLA, TIMA, MINERALOGIC and INCAMineral. These instruments determine mineral quantities, and perform mineral liberation and mineral association analyses. Since the late 1990's the scientific community of applied mineralogy and automated mineralogy has expressed the need of determining the variability in the measurements of mineral quantities and mineral liberation analyses through inter laboratory testing of suitable reference materials to improve confidence in the quantitative mineralogy measurements. A sample representing the −28+65 mesh size fraction of a sulphide flotation rougher concentrate from the Clarabelle mill in Copper Cliff, Vale (Sudbury, Ontario) was prepared for this testing. This rougher concentrate contains chalcopyrite, pyrrhotite, pentlandite, pyrite, quartz and feldspars as the dominant minerals. A 10-gram sub-sample was sent to the participating laboratories of the round robin. The sub-samples were analyzed by the participating laboratories to determine the mineral quantities, the liberation of chalcopyrite and the mineral association analysis for chalcopyrite. The findings indicate that there is a good agreement in the mineral quantities. The liberation analysis results also indicate a good agreement with the exception of two participants. These results indicate that correct mineral quantities do not necessarily imply correct mineral liberation. The testing also revealed that there was no consensus on how the mineral associations are treated and reported. We recommend that the modal analysis by liberation classes should be the preferred approach in reporting the mineral associations because it is more relevant to understand the mineral concentration operations.



## 1. Introduction

Presently there are about 160 systems for automated image analysis. Since the late 1990's the scientific community of automated mineralogy has expressed the need of performing round robin testing that would help to check the variability that may arise from the use of different systems. Such a need was clearly expressed during a workshop held on September 6, 2008 in Brisbane, Australia in conjunction with the 9th International Congress for Applied Mineralogy (Paktunc, 2009). A sample for such a testing should fulfill several requirements. First of all, the sample size should be large enough to meet the current and future demands as a reference material. Secondly, it must be well homogenized and split by established techniques. Thirdly, it should have a mineralogical composition that is not overly complex. Fourthly, the dominant mineral of interest should be abundant but not completely liberated to allow obtaining a spread in liberation curves. Obtaining such a sample proved to be not simple.

In 1997, CanmetMINING entered in an agreement with McGill University to produce synthetic particles with determined liberation classes. McGill University produced binary particles of glass and leaded glass. The particle size was 425–600 μm and it was divided into 11 liberation classes with increments of 10 %, from totally free glass particles to totally free leaded glass particles. Details of this material have been described by Lin, 1997, and Lin and Finch, 2000. Several particle populations were made from these standards, prepared as polished sections and analyzed by CanmetMINING to obtain the measured liberation curves. These populations of synthetic particles with known liberation curves were used to assess different stereological correction procedures on the measured liberation curves (Lin et al., 1999) and to evaluate the sources and magnitudes of uncertainties arising from the measurement of quantitative mineralogical data on two-dimensional surfaces using synchrotron-based X-ray microtomography (Paktunc et al., 2001, 2004). This synthetic particulate material was time consuming to produce, expensive and only ~10 g within each liberation class were produced. Thus, this route is not practical for producing larger amounts of materials for testing and use as an automated mineralogy reference sample.

* Corresponding author.
*E-mail address:* rolando.lastra@canada.ca (R. Lastra).

http://dx.doi.org/10.1016/j.mineng.2016.06.025
0892-6875/

Iron ore mines require high grade ores to be commercially attractive. Thus, iron ores commonly have high content of hematite and magnetite. However, identification and separation of magnetite from hematite in most automated mineralogy systems (QEM∗SCAN and MLA) is not simple. Thus, a sample of an iron ore would not be appropriate for round robin testing.

The feeds to mineral processing plants concentrating sulphide minerals are commonly low grade. For example, it is common that the feed to a copper concentrator has less than 1% Cu. In this case, the amount of ore mineral would be too low and its liberation at moderate grinds is likely to be too high. In addition, the amount of the ore mineral in the lower liberation classes would be too low. Another characteristic is that ore bodies that are presently being exploited can reach high liberation at acceptable grinds to allow attractive return on investment. Therefore, the feed to a sulphide concentration plant cannot be used for round robin testing.

The ore bodies that have complex liberation problems are not being exploited such as the massive sulphide deposits in the Iberian Pyritic Belt (e.g. Espi et al., 2008). In 2010, we studied samples from the Duchas ore body on the Iberian Pyritic Belt, obtained from the Spanish Geologic and Mining Institute (IGME – Instituto Geológico y Minero de España). The samples were crushed to −1.7 mm, sieved into twelve size fractions from −1.7 + 1.2 mm to −38 μm and then prepared as polished sections. The study indicated that there is not enough material in all the liberation classes. Thus, the sample proved to be not suitable for the round robin.

In 2011, as part of a collaborative research project with the Polytechnic University of Madrid, we performed an automated quantitative mineralogy investigation of a polished section of the re-cleaner copper concentrate of the Kansanshi flotation plant (Zambia). This sample contained chalcopyrite, secondary copper sulphides such as digenite, chalcocite, bornite and covellite, copper oxide minerals such as malachite, delafossite, chrysocolla and abswurmbachite, pyrite, pyrrhotite, hematite, quartz, calcite, dolomite, albite, micas, and rutile. The concentrator aimed to recover copper minerals. Thus, if the copper minerals are considered as a group, then the mineral liberation of this ore does not present problems. However, this ore displays highly complex mineralogical textures among the copper minerals, thus the liberation analysis by particle composition would be different than the liberation analysis by particle surface (Lastra, 2002; Pérez Barnuevo, 2014). This sample is ideal to test methods for mineralogical texture analysis (Pérez-Barnuevo et al., 2012; Pérez Barnuevo, 2014), but it is overly complex as a mineralogical reference material for liberation and modal analyses.

## 2. Materials and methods

In 2012, we obtained ∼1000 kg of a dry sulphide flotation rougher concentrate from the Clarabelle mill in Copper Cliff in Sudbury, Ontario. This rougher concentrate contains chalcopyrite, pyrrhotite, pentlandite, pyrite, quartz and feldspars as the dominant minerals. The sample was dry-sieved on the 200 mesh (75 μm). The −200 mesh (−75 μm) size fraction was used to prepare a standard for chemical assays, named PTC-1b, as part of Canadian certified reference materials (CCRM). The +200 mesh size fraction was separated (∼200 kg) and stored in two large drums for about one year. From these drums a grab sample was taken and sieved into four size fractions +14, −14+30, −30+70, −70+100, −100+200 and −200 mesh (+1400, −1400+595, −595+212, −212+149, −149 +75 and −75 μm). A cursory examination under an optical microscope revealed that the chalcopyrite was almost completely liberated in the −70 mesh (−212 μm) size fraction. The chalcopyrite liberation was high but not complete in the −30+70 mesh (−595 +212 μm) size fraction. The +30 mesh (+595 μm) size fractions were too coarse and a polished section contained too few particles that could yield acceptable results for an automated mineralogy study. Thus, the −30+70 mesh size fraction was targeted, even though the liberation of chalcopyrite was not spread across all the liberation classes.

The whole amount in the +200 mesh (+75 μm) size fraction (∼200 kg) was dry-sieved using a pilot plant machine with a single and removable mesh deck. Several mesh decks were available for the pilot plant sieving equipment. The decks close to the target were the 28 and 65 mesh (589 and 208 μm) sieves. The −65 mesh size fraction was sieved out and then the +28 mesh was sieved out. Thus, about 20 kg of sample was obtained of the −28+65 mesh size fraction. A grab sample was taken for polished section preparation. It was observed that there were many agglomerated particles. Thus, the whole material in this size fraction was subjected to wet attrition using pilot plant equipment and diluting the sample with 1:1 water. The wet attrition was done for a period of 1 h. Then, the slurry was sieved to remove the −65 mesh fraction. This procedure yielded only ∼2 kg of the −28+65 mesh material, indicating that the procedure removed a lot of agglomerated fine particles.

The −28+65 mesh (−589+208 μm) size fraction (2.2 kg) was dried at room temperature and split using a rotary splitter into six sub-fractions. Each sub-fraction was further split into six fractions using a smaller rotary splitter. Then, each of the six subfractions was further split with a smaller rotary splitter with six partitions. Thus, a total of $6 \times 6 \times 6 = 216$ sub-samples of ∼10 g were obtained (Fig. 1).

Nine polished sections were prepared from randomly selected sub-samples. The polished sections were examined with an MLA instrument using the GXMAP mode. It was observed that some agglomerations remained in the samples. In addition, some very rounded particles were observed which appear to be recycled materials from a smelter dust. The agglomerations could be easily "broken" using the particle separation routine of the MLA. Also, many of the rounded smelter particles could be "broken" using the particle separation routine of the MLA. The MLA was instructed to remove any particles that were too small to be in this size fraction (i.e. <212 μm). In addition, it should be noted that the receipt of dry flotation concentrate samples is common for many automated mineralogy laboratories; however, there are sample preparation methods to de-agglomerate such particles.

Following the initial examination, it was decided that the sample was appropriate for the round robin testing. In total, 17 laboratories accepted our invitation to participate in the round robin testing. Each participating laboratory was provided with a subsample. The results are listed by sample numbers by which the participating laboratories can identify their results. Table 1 lists the sub-samples and the method used to prepare the polished section.

## 3. Results

### 3.1. Chemical assays

Two of the sub-samples were further split into three and assayed as three separate samples. The results of the assays are given in Table 2.

### 3.2. Mineral quantities

The round robin participants reported the quantities of >30 minerals present. Listing all those minerals in this paper is not practical and in fact, there are discrepancies in the reported minor and trace minerals. These discrepancies would be distracting; therefore, only the quantities of the major minerals reported by the participants are given (Table 3). Some participants analyzed