ION4RAW

Research and Innovation Action (RIA)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 815748

Start date : 2019-06-01 Duration : 48 Months
http://ion4raw.eu

Technical note and methodology guide for sampling and sample preparation

Authors : Mrs. Pauline MOREAU (BRGM), Moreau Pauline, Gourcerol Blandine, Duhamel-Achin Isabelle, Delchini Sylvain, Négrel Philippe (BRGM), Werner Warsheid (Cumbrex Exploraciones), Chris Sangster (Scotgold Resources), Francisco Sánchez Ruiz (First Quantum Mineral LTD), Raúl Álvarez Cifuentes (Orvana Minerals Corp.)

ION4RAW - Contract Number: 815748

Project officer: Floriana la Marca

Document title	Technical note and methodology guide for sampling and sample preparation
Author(s)	Mrs. Pauline MOREAU, Moreau Pauline, Gourcerol Blandine, Duhamel-Achin Isabelle, Delchini Sylvain, Négrel Philippe (BRGM), Werner Warsheid (Cumbrex Exploraciones), Chris Sangster (Scotgold Resources), Francisco Sánchez Ruiz (First Quantum Mineral LTD), Raúl Álvarez Cifuentes (Orvana Minerals Corp.)
Number of pages	43
Document type	Deliverable
Work Package	WP2
Document number	D2.2
Issued by	BRGM
Date of completion	2020-11-23 10:30:07
Dissemination level	Public

Summary

D2.2 represents the second deliverable of the Work Package 2 where the sampling operations of the ores and pre-treatment steps before analysis are described. Five deposits were selected for the project. Each deposit/sample is described in a dedicated paragraph as follow: firstly a description of the deposit is given (geographic, geologic and reserves), then sampling operations for the ION4RAW project are described and finally a pre-treatment chart is given to detail the sequence of the physical preparation steps required before analytical procedure. Some difficulties encountered within this task are also explained in the 6th section.

Approval

Date	By
2020-11-23 11:16:14	Dr. Isabelle DUHAMEL ACHIN (BRGM)
2020-11-23 17:38:28	Mrs. Maria TRIPIANA (IDENER)

ION4RAW: Ionometallurgy of primary sources for an enhanced raw materials recovery

Project type:	Research and Innovation Action
Start date of project:	01/06/2019
Duration:	48 months

D 2.2: Technical note and methodology guide for sampling and sample preparation

WP n° and title	WP2 – By-product potential evaluation
Responsible Author(s)	Moreau Pauline
Contributor(s)	Gourcerol Blandine, Duhamel-Achin Isabelle, Delchini Sylvain, Négrel Philippe (BRGM), Werner Warsheid (Cumbrex Exploraciones), Chris Sangster (Scotgold Resources), Francisco Sánchez Ruiz (First Quantum Mineral LTD), Raúl Álvarez Cifuentes (Orvana Minerals Corp.)
Dissemination Level	PU

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

TABLE OF CONTENTS

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Annex

Tables

Figures

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

EXECUTIVE SUMMARY

D2.2 represents the second deliverable of the Work Package 2 where the sampling operations of the ores and pre-treatment steps before analysis are described. Five deposits were selected for the project. Each deposit/sample is described in a dedicated paragraph as follow: firstly a description of the deposit is given (geographic, geologic and reserves), then sampling operations for the ION4RAW project are described and finally a pre-treatment chart is given to detail the sequence of the physical preparation steps required before analytical procedure. Some difficulties encountered within this task are also explained in the 6th section.

KEYWORDS

Ore, deposit, sampling, physical pre-treatment

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

1 INTRODUCTION

The ION4RAW process has to be tested on different samples as ionometallurgy is mineral specific. A detailed understanding of the mineralogy of the ores is required to determine optimum processing routes for the product and by-product commodities. Thus, different ore samples from various deposit types were selected in the project. The aim of the WP2 is to fully characterize ore samples that will be used to produce concentrates. Whole-rock chemistry point chemical composition as well as detailed mineralogical characterisation will be realized using XRF, ICP-OES, ICP-MS, XRD SEM, EPMA, LA-ICPMS. This document presents the protocol for sampling and sample pre-treatment implemented in WP2 to be representative of the by-products that are naturally scattered in the ore rocks. Sampling and preparation steps are essential to guarantee a reliable and valid analytical result. In this deliverable, each sampling site is described in a dedicated paragraph, as well as the sampling procedures, and methodologies applied for preparing samples (splitting of samples, comminution by crushing or grinding, homogenization, polished thick/thin section preparation). Preservation of ores is a major consideration to conduce reliable and representative analytical measurements. As an example, an effort was done to reduce the effects of humidity and oxidation of mineral surfaces during sampling, shipment and preparation steps.

The whole-rock analyses and the respective mineralogy of ores with identification of by-product mineral species, elemental and mineral deportment that will be conducted to provide by-product potential evaluation, are not described in this report. They will be detailed in the D2.3 deliverable of ION4RAW project.

Five deposits were selected for this project. Table 1 presents the name of the deposits, location, owner of the project/mine and the paragraph in the report to refer to get information about sampling and preparation procedures.

Deposit	Country	Owner	Partner or EEAB*?	Paragraph of the report to refer
Cononish Gold mine	Scotland	Scotgold Resources	Partner	2
Cobre las Cruces	Spain	First Quantum Mineral LTD	EEAB	3
El Porvenir	Peru	Nexa Resources Peru	Partner : Cumbrex Exploraciones S.A.C.	4
Cerro Lindo	Peru	Nexa Resources Peru	Partner : Cumbrex Exploraciones S.A.C.	4
El Valle Boinas	Spain	Orvana Minerals Corp.	EEAB	5

Table 1 : Sampling site description: name, location, owner and link with the project (* EEAB corresponds to External Expert Advisory Board)

The total amount of sample required for the whole project was sampled at once to ensure that all partners could work with the same ore all along the project. Each partner gave the required amount of concentrate and raw ore for their operations. As a result, around 6 tons of primary sample were needed for each deposit. For Cobre las Cruces and El Valle Boinas, fewer amount were required because these samples will not be used in WP6 (e.g. around 300 kg of primary sample).

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

1.1 Sampling protocol applied for bulk samples

After primary sampling, all samples were sent to Wardell Armstrong (WAI) in Truro (UK), where the ore concentrates were prepared and then delivered to the different partners for the process development. From the primary sample, a representative amount of uncrushed ore (between 25 and 65 kg) was sub-sampled (secondary sampling) and sent to the BRGM for mineral characterization.

Soon after reception at WAI, for each bulk sample, a detailed Particle Size Distribution (PSD) was carried out on a sub-sample of uncrushed as-received ore (see for example (Mac Donald & Conybeare, 2019) for Cononish gold mine sample) before subsampling for characterization within WP2. These data (reproduced for each sample) permitted to evaluate the particle size heterogeneity / homogeneity and thus to determine the amount of ore required for analytical purpose.

As exposed by Gy (2002), a sampling operation is correct if all the fragments constituting the sample batch have the same probability of being taken. One of the main risks when looking at the trace elements in ores is the nugget effect that could be induced for some elements such as gold. In order to ensure the production of a sample for analysis and mineralogical characterization as representative as possible of the initial sample, careful attention has to be taken to sample mass reduction from the secondary sampling. Practically, several sampling stages, alternating with homogenization and size reduction stages were performed at BRGM.

Mass reduction can be achieved in many ways (grab sampling, splitting or incremental sampling). Mass reduction by splitting is an appropriate method to divide stationary samples of several tens of kilograms and with fragments of size less than 100 mm. In our case, this method was favoured given that there was between 25 kg and 65 kg of sample to treat, depending of the ore with top particle sizes of at least 40 mm. Two variants of splitting were used: coning and quartering method for samples with particles size greater than 1 cm and riffle splitting method when particles size was smaller than 1 cm. These two methods have the advantage to be quick and efficient even if the coning and quartering induce a higher variability of representativeness than riffle splitting (Gerlach et al., 2002).

The following paragraphs present in detail the sampling and the preparation procedure applied on the five bulk ore samples. These different steps are synthetized in Figure 1.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

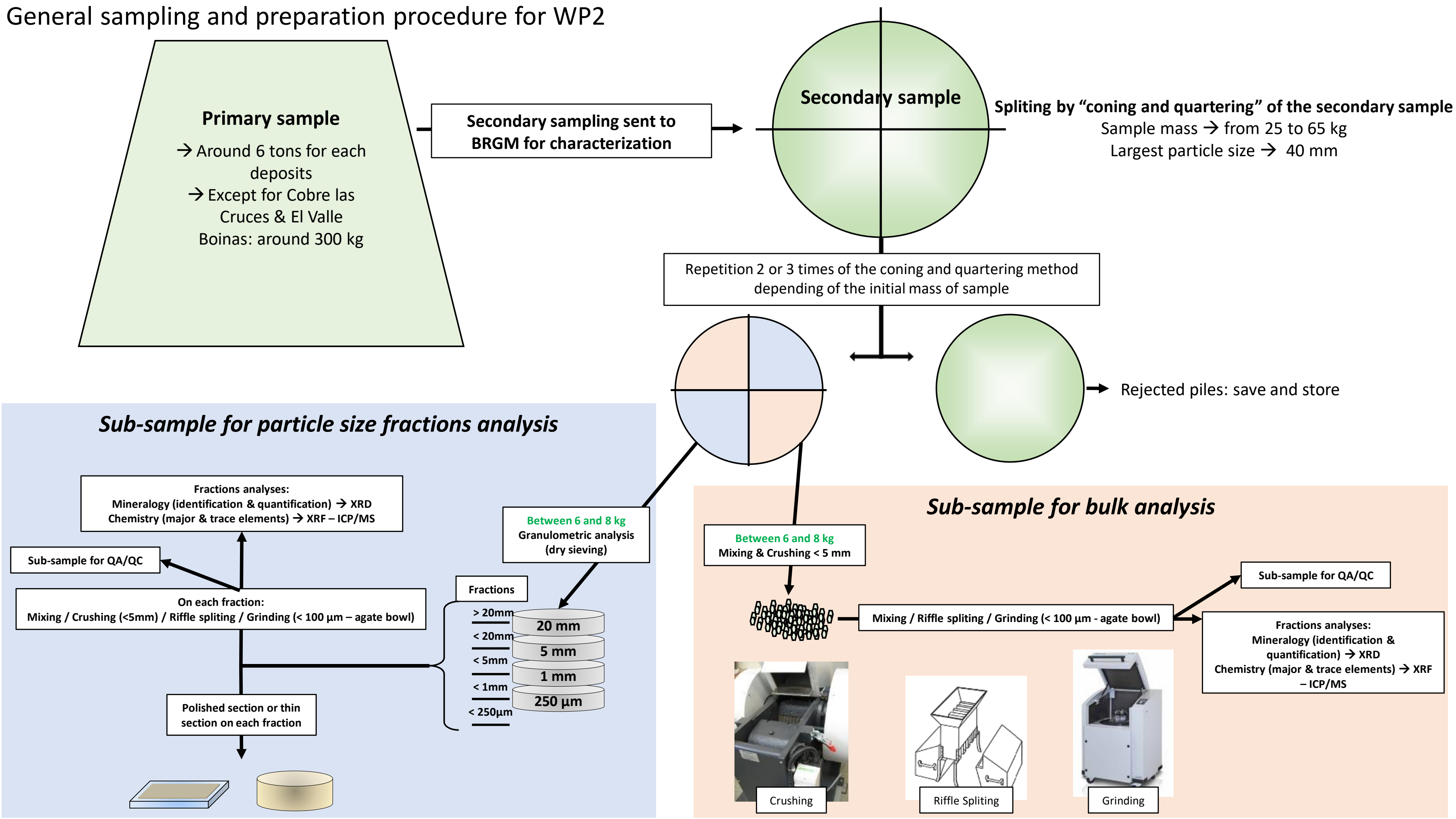


Figure 1: Overall sampling and sample preparation procedure.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

1.1.1 Secondary sample splitting by "coning and quartering"

Coning and quartering principle is detailed in Figure 2 and Figure 3. It involves mixing and pouring the material in order to homogenize it until a uniform conical pile is obtained. The pile must be made so that the natural segregation in the cone is radially symmetrical. The cone is then spread from the centre to form a flattened disk of material. This disk is then divided into quarters using perpendicular boards. Randomly, one pair of opposite quarters is removed, and the other pair is used as the sample.

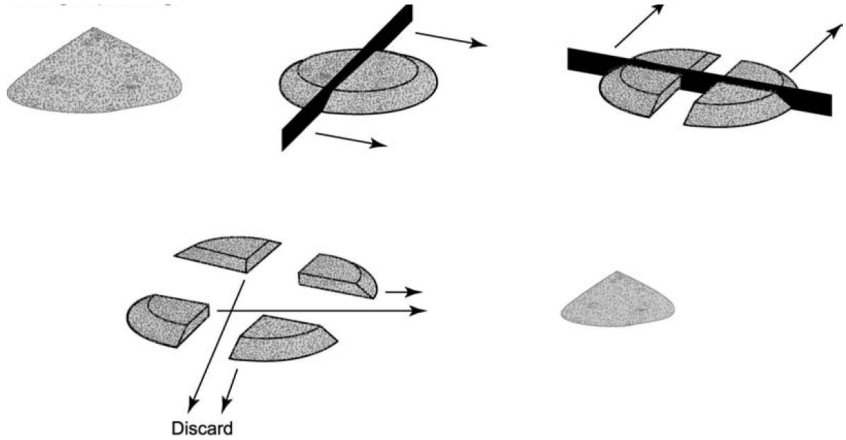


Figure 2: Coning and quartering method principle, from (Gerlach et al., 2002)

Figure 3: Coning and quartering method: illustration on a sample

If the sample is too large, then it can be coned and quartered again until the desired sample size is obtained. In our case, coning and quartering was repeated successively two or three times depending of the initial mass of sample, until two sub-samples with masses between 6 kg and 8 kg were obtained. The rejected piles were saved and stored. According to the Theory Of Sampling (Gy, 1982; Pitard, 1993), this procedure could generate grouping and segregation error and preparation error. In order to minimize these errors, a thorough mixing was performed and careful sample preparation procedures were applied. The first sub-sample obtained was dedicated to the bulk analysis (see 1.1.2), while the second sub-sample underwent a particle size separation by dry sieving in order to analyse the different particle size fractions (see 1.1.3).

1.1.2 Sub-sample preparation for bulk analysis

A succession of sample mass and size reduction were done on the 6-8 kg sub-sample to obtain the sample for analysis. To minimize the grouping and segregation error and the sample preparation error related to these preparation steps, careful attention was paid to the sub-sample mixing, handling and losses. Moreover, the processing appliances were carefully cleaned before all preparations to avoid potential contaminations. The different preparation steps performed are as follow:

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

- After mixing, the 6-8 kg sub-sample was crushed with a jaw-crusher (Minemet) to reduce the particle size to less than 5 mm. This step could be prone to preparation error (Desroches et al., 2019). It was minimized by implementing good laboratory practices.
- The crushed sub-sample was mixed again and then divided using a riffle splitter (Figure 4). Riffle splitter consists of a series of chutes that run in alternating directions: when material is poured into the top of the splitter, it flows through the chutes and is randomly divided into two equal-sized fractions. In order to work properly, these splitters must be fed using a special pan that has exactly the same width as the top of the chutes, otherwise the amount of material entering the two end chutes is different. This procedure was repeated until 30 -50 g samples were obtained. The discarded material was saved and stored.
- Finally, the particle size of the 30-50 g final sample was reduced to less than 100 µm using a ring mill equipped with an agate bowl. The sample was final material, which would be analysed.

Figure 4: Riffle sample divider

1.1.3 Sub-sample preparation for particle size analysis

In order to provide information about particle size distributions and to determine if there is valuable differences related to the particle size distribution, the second 6-8 kg sub-sample was dried sieved with five granulometric fractions (<250µm, 250 µm to 1 mm, 1 to 5 mm, 5 to 20 mm, > 20mm, see Figure 1). Each fraction was then divided as follow: (1) the first half was prepared to obtain a powdered sample of 30-50 g for chemical analyses implementing the procedure explained in 1.1.2; while (2) the second half was used to prepare polished thick or thin sections.

2 Cononish gold mine

2.1 Sampling location and description

The Cononish gold mine was acquired by the mining company Scotgold Resources in 2007. It is located is the Scottish Highlands, around 140 km west from Edinburgh and 116 km north from Glasgow as presented in Figure 5.

The deposit consists of an orogenic gold deposit along with quartz veins formed through magmatic fluid circulation within the Dalradian metamorphic rocks coeval with intrusion of granites between 435 and 395 Ma (Spence-Jones et al., 2018). The main commodities are Au, Ag, Cu, Zn, Pb and Te (Te/Au≈2.4). The mineral resources are given in Table 2. An aerial view of the mine is given in Figure 6.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Figure 5: Cononish gold mine location

Figure 6 : Aerial view of the Cononish gold site and the stockpile (in a red circle)

	kt	Ag grade g/t	Au grade g/t
MEASURED + INDICATED	541	59.9	14.3

Table 2: Summary of Mineral Resources for Cononish gold mine[1]

[1] https://www.scotgoldresources.com/projects/cononish-project/#cononish-gold-and-silver-mine

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

2.2 Sampling operation for ION4RAW process development

Sampling for the Ion4Raw project was done by Scotgold Resources in July 2019 from the ore stockpile mined from the underground mine between 1989 and 1991. The stockpile initially contained 7000t but the majority (c.6000t) was processed through a trial processing plant during 2017 and 2018. The ION4RAW sample was taken as a composite sample from the material remaining and had been crushed to around 50mm during the trial. About 5.3 tons of primary sample were taken from the stockpile with a small digger, packed in 1t bulk a bag and palletized for transportation. The pictures presented on Figure 7 represent the stockpile with a 9 cm scale.

Figure 7: Pictures of the stockpile of the Cononish gold mine, from where the sample for the ION4RAW project was sampled (scale: double arrow = 9cm)

The 5.3 tons sample was received by WAI in August 2019. For characterization at BRGM, an total of 65 kg of uncrushed as-received ore and 500 g of sulphide concentrate were requested (secondary samples) and sent to the BRGM on December 2019.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

2.3 Pre-treatment of uncrushed as-received ore before characterisation

The procedure applied for quartering and subsampling a 65 kg representative uncrushed as-received ore sample for the BRGM characterisation is described elsewhere (Mac Donald & Conybeare, 2019).

The 65 kg of ore was pre-treated as presented in Figure 8, before analytical steps.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

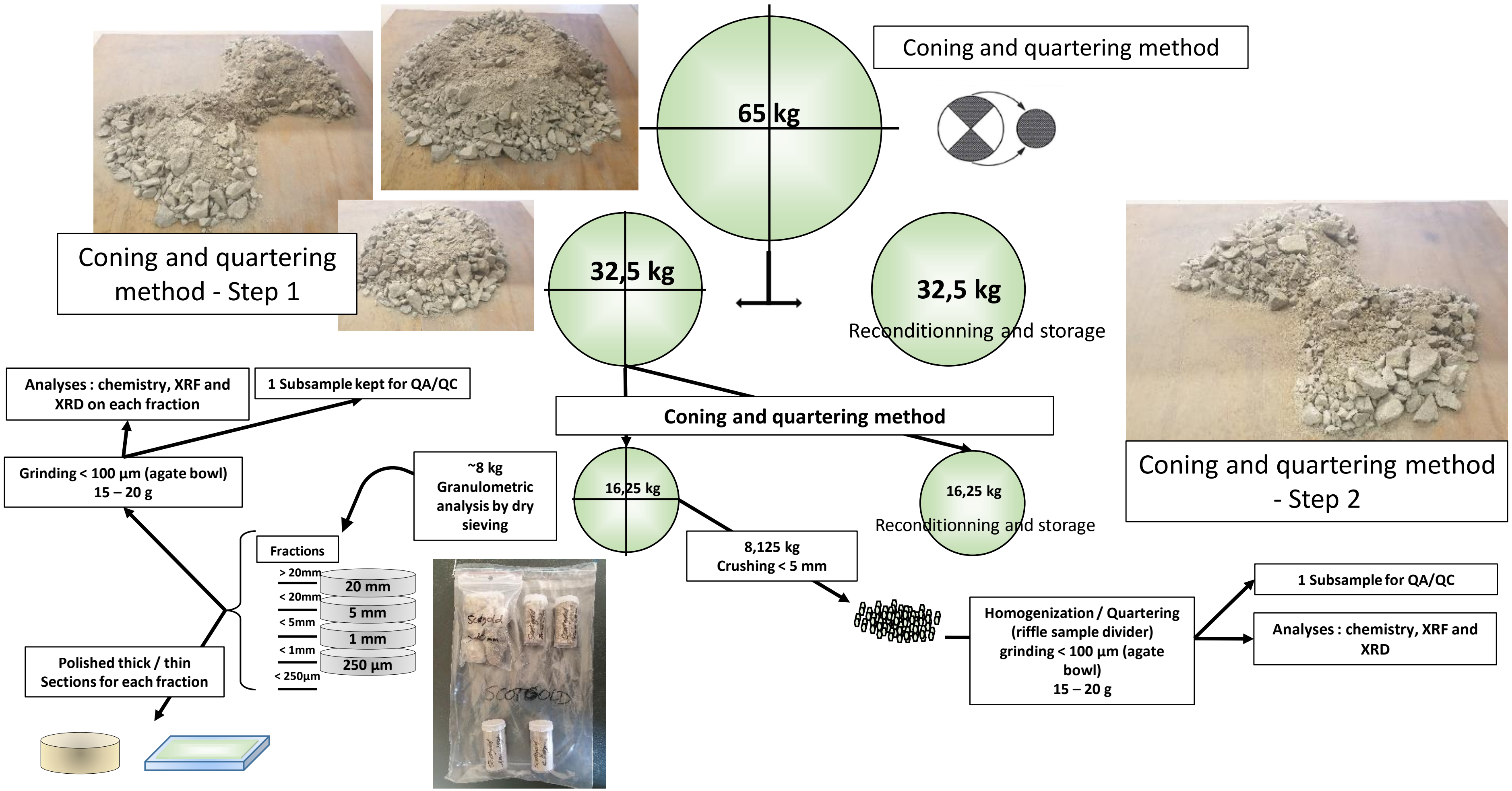


Figure 8: Pre-treatment chart before analysis steps for Cononish gold mine uncrushed as-received ore

A total of 10 polished thick sections and 4 polished thin sections were produced.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

The detailed PSD, carried out on a sub-sample of uncrushed as-received ore at WAI (Mac Donald & Conybeare, 2019), is presented in Figure 9 together with the size division performed at BRGM. The results are consistent for both studies.

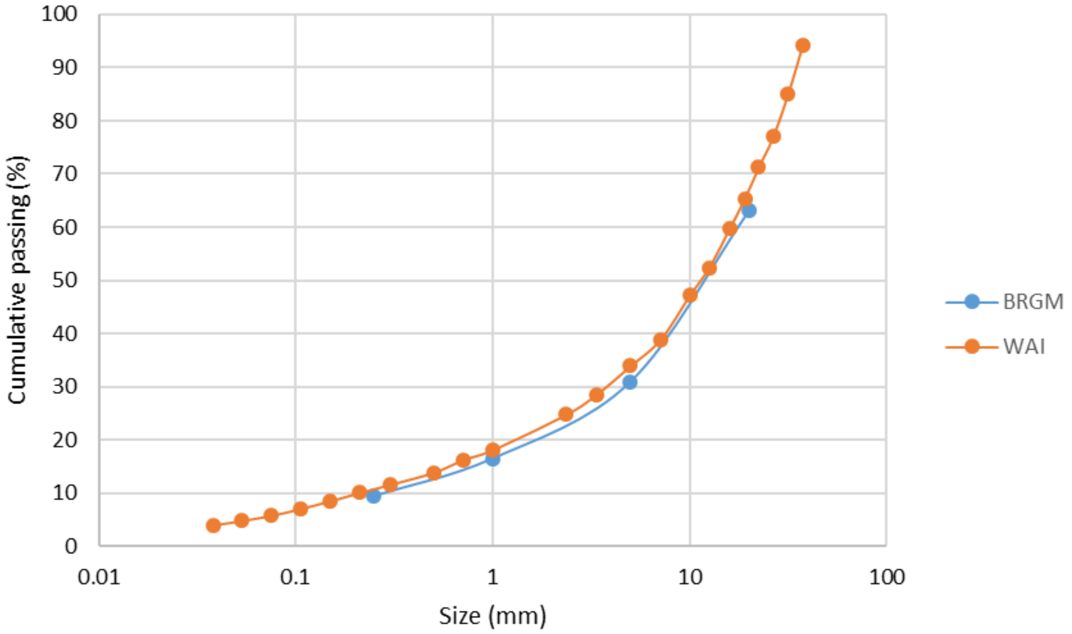


Figure 9: Cumulative particle size distribution for Cononish gold mine samples (uncrushed as-received ore) (see (Mac Donald & Conybeare, 2019) for "WAI" results)

3 Cobre las Cruces mine

3.1 Sampling location and description

Cobre las Cruces (CLC) is a very high grade (5-6%) open pit copper mine with a plant located 24 km north from Sevilla (Spain). It is owned by First Quantum Mineral LTD. Figure 10 shows a map with the location of the mine. Figure 11 shows an aerial view of the open pit and the plant. The deposit, which have been discovered in 1994 is located within the Iberian Pyrite Belt and classified as a Volcanogenic Massive Sulphide (VHMS) deposit type. The copper production started in 2009 with a production of 72 kt Cu per year. According to First Quantum Mineral LTD. information, main copper tenors are secondary copper sulphides with a very low presence of chalcopyrite. The geological description of the mine is given in Figure 12. Mineral resource statement for Cobre las Cruces stockpiles is given in Table 3.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Figure 10: Location of the CLC mine

Figure 11: Aerial view of the CLC mine

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

	Mt	Cu grade %	Pb grade %	Zn grade %
MEASURED + INDICATED	4.3	1.21	1.73	2.53

Table 3: Mineral resource statement for Cobre las Cruces stockpiles (31/12/2019) [2]

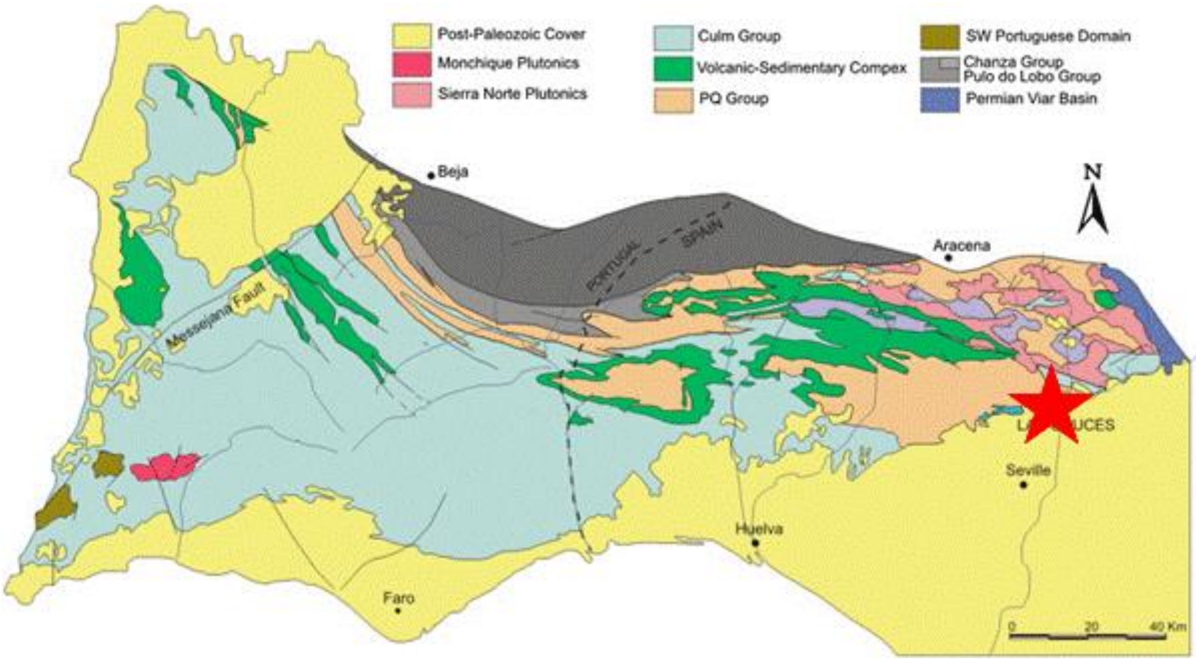


Figure 12: Geological map of Cobre Las Cruces mine (Internal CLC Company Presentation)

3.2 Sampling operation for ION4RAW process development

Sampling was performed by First Quantum Mineral LTD at the beginning of October 2019. Available sample for Ion4Raw was extracted from the open pit and stored outside few months before sampling. About 300 kg of primary sample were required for the project to prepare the sulphide concentrate, and 30 kg of "Run Off Mine" for ore characterisation within WP2. The 300 kg sample was collected after the primary Jaw crusher (see Figure 13). A dimension of 100 cm was defined as the maximum size, because it is the opening size of the equipment. However, particles of this size are unexpected and the experience from First Quantum Mineral LTD is that high quantity of fine material would come from the mine. Then, it was considered that P80 of less than 500 mm will be the feed of the crusher. The 30kg "Run of Mine" sample was collected before the primary jaw crusher. The sample was packed in a bulka bag, as presented in Figure 15.

[2] https://www.first-quantum.com/English/our-operations/operating-mines/cobre-las-cruces/reserves-and-resources/default.aspx

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

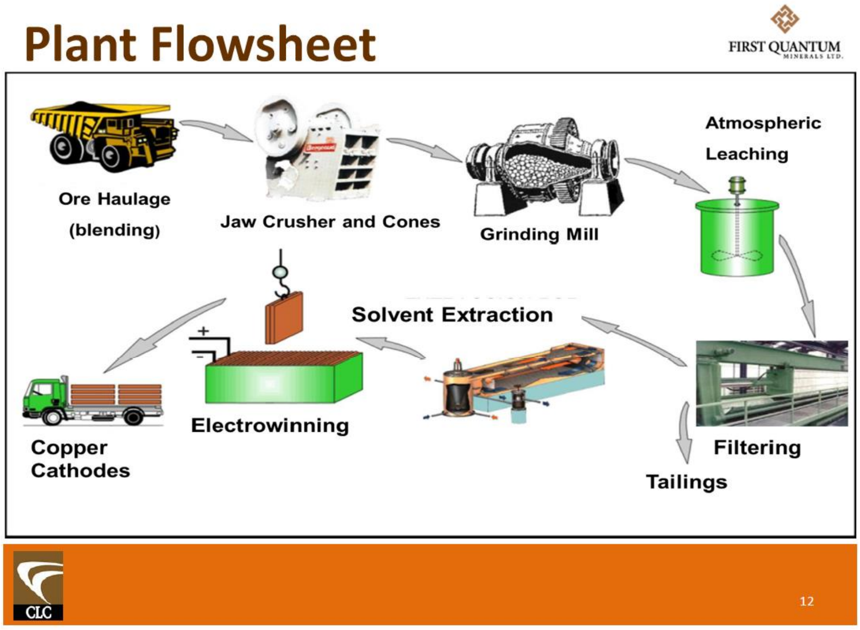


Figure 13: General plant flowsheet used by First Quantum Mineral LTD at the CLC mine site (Internal CLC Company Presentation).

The 300 kg of primary sample sent for the project represents a composite of several sampling areas in order to maintain the average grade of the deposit. The locations of sampling areas in the open pit are given in Figure 14. The different colours are the code that miners use to identify various grades.

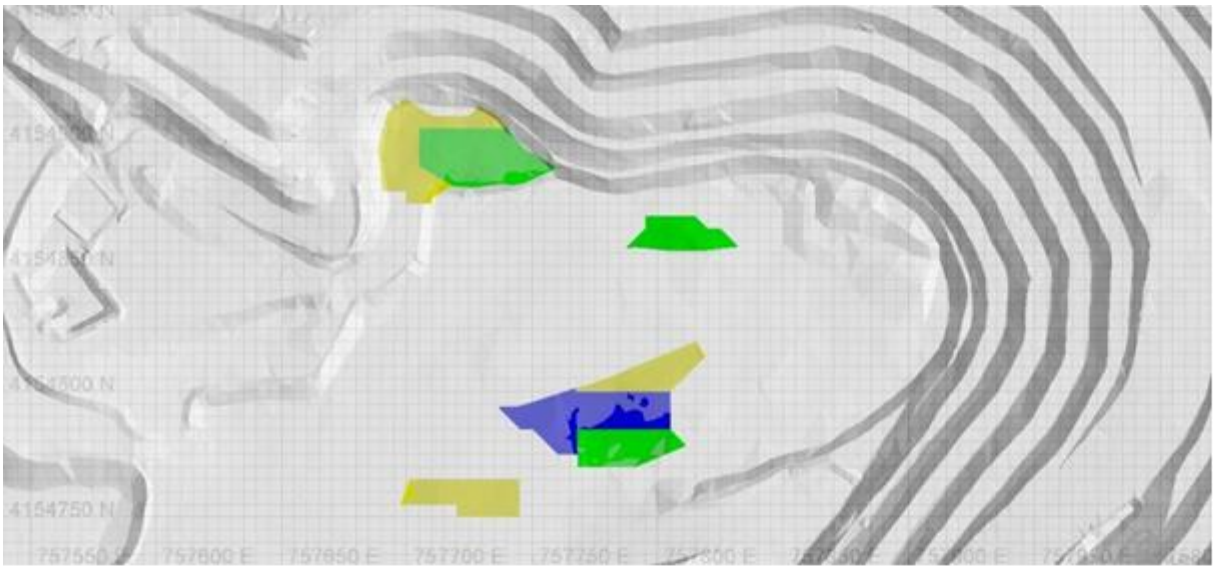


Figure 14: CLC sampling - Composite of sampling area (Internal CLC 3D Production model (Vulcan))

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Figure 15: Pictures of sample from CLC ready for shipment from CLC to WAI

Both samples (the 300 kg “bulk” sample for process and 30 kg “run of mine” sample) were received by WAI in Truro (UK) at the end of October 2019. The 30 kg “run of mine” sample was not used by WAI and was sent to the BRGM without treatment. A visit of the BRGM on WAI site at the end of October allowed visual examination of the samples. The 30 kg “run of mine” sample contained some blocs of few centimetres but no larger size bloc. The 300kg “bulk” sample contained a lot of fine particles and some larger blocs. One of these blocs was selected and it was sent to the BRGM together with the 30 kg “run of mine” sample on December 2019.

3.2.1 Pre-treatment before characterisation

3.2.2 Powder of Run of mine sample

The 30 kg “run of mine” sample was sent from WAI to the BRGM unmodified. A picture of the sample as received is given in Figure 16. The pre-treatment chart applied at the BRGM before the analysis steps is depicted in Figure 17.

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Figure 16: Picture of CLC run of mine sample received at BRGM

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

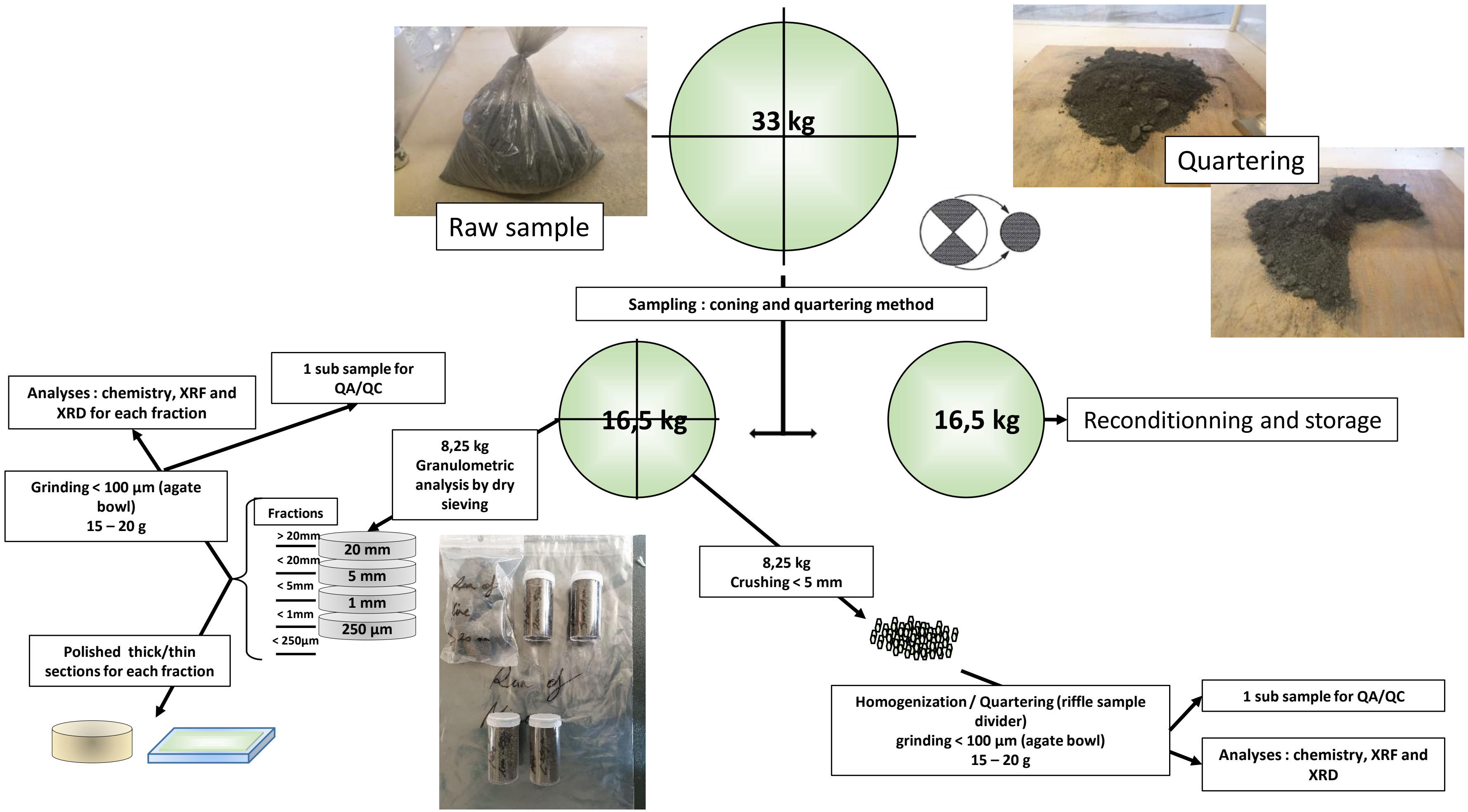


Figure 17: Pre-treatment chart before analysis steps for CLC "Run of Mine" powder sample

A total of 10 thin polished sections and 4 polished thick sections were produced.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Within the pre-treatment procedure, a size division was carried out on a sub-sample of approx. 8 kg (see results in Figure 18). These data are consistent with the data given by First Quantum LTD as 90% of the sample is < 20cm.

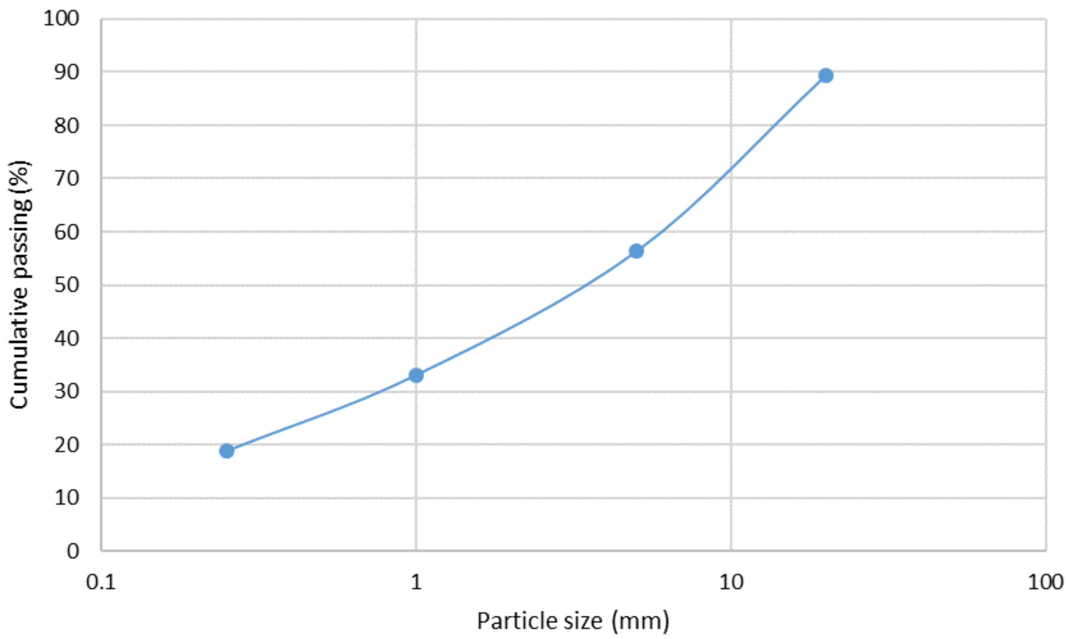


Figure 18: Cumulative particle size distribution for CLC Run of Mine powder sample

3.2.3 Bloc from bulk sample

One block from bulk sample was also available. The pre-treatment chart for this sample is given in Figure 19.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

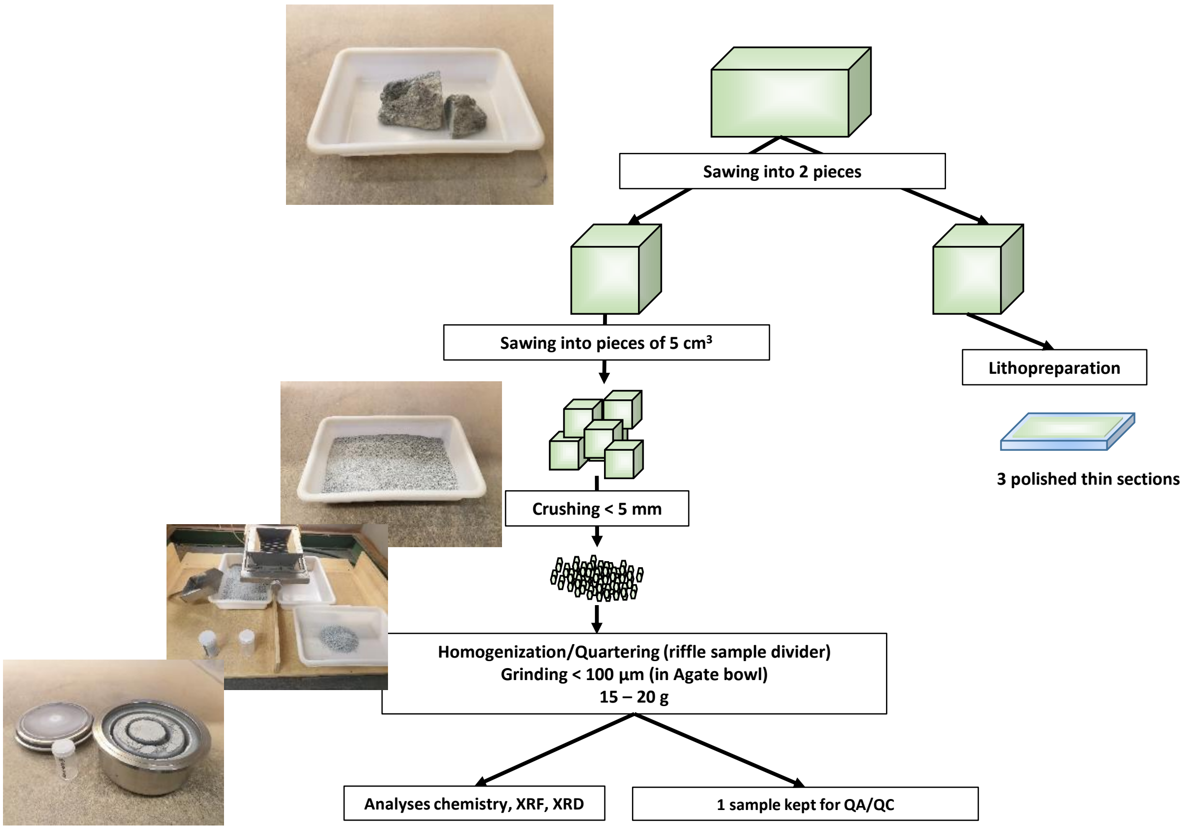


Figure 19: Pre-treatment chart before analysis steps for CLC "Run of Mine" block sample

Three polished thin sections were prepared from this sample.

4 El Porvenir and Cerro Lindo mines

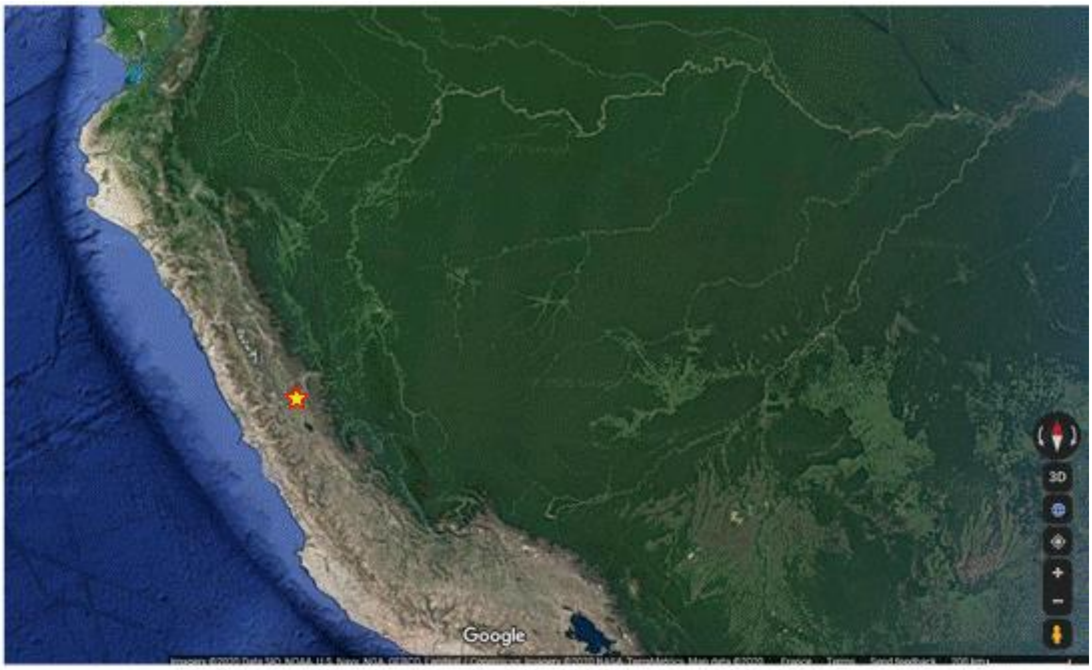
In this deliverable, samples from El Porvenir and Cerro Lindo mines are described in the same paragraph because they underwent the same sampling and preparation protocols. They are both located in Peru, central Andes.

4.1 Sampling locations and descriptions

4.1.1 El Porvenir

El Porvenir mine is an underground mine owned by Nexa Resources Peru. It is located in about 285 km northeast from Lima (Peru), in the Pasco province. El Porvenir is part of the Western Cordillera of the Andes mountain range in central Peru, hosted by Eocene-Miocene Polymetallic, and Miocene Au-Ag Epithermal Belts (Saez et al., 2017). Mineralization occurs at the contact between the upper Triassic limestone (i.e exoskarn) and the granodioritic-dacitic intrusive rocks (i.e. endoskarn). In El Porvenir, operations began in 1949 as a small scale and artisanal mine. The map depicted in Figure 20 (a) shows the location of the mine and an aerial view of the mine is available on Figure 20 (b). Since El Porvenir mine is very close to Atacocha mine, as shown in Figure 21, the mines are currently undergoing an integration process, through which they will form the Pasco mining complex. El Porvenir reserves are given in Table 4, a plan/description of the mine is given in Figure 22 and a picture of the mine is given in Figure 23, showing also the mine levels where sampling was performed for ION4RAW project, form of the upper and intermediate zones.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).



(a)

(b)

Figure 20: Location of El Porvenir mine (a) and aerial view of the mine (b)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

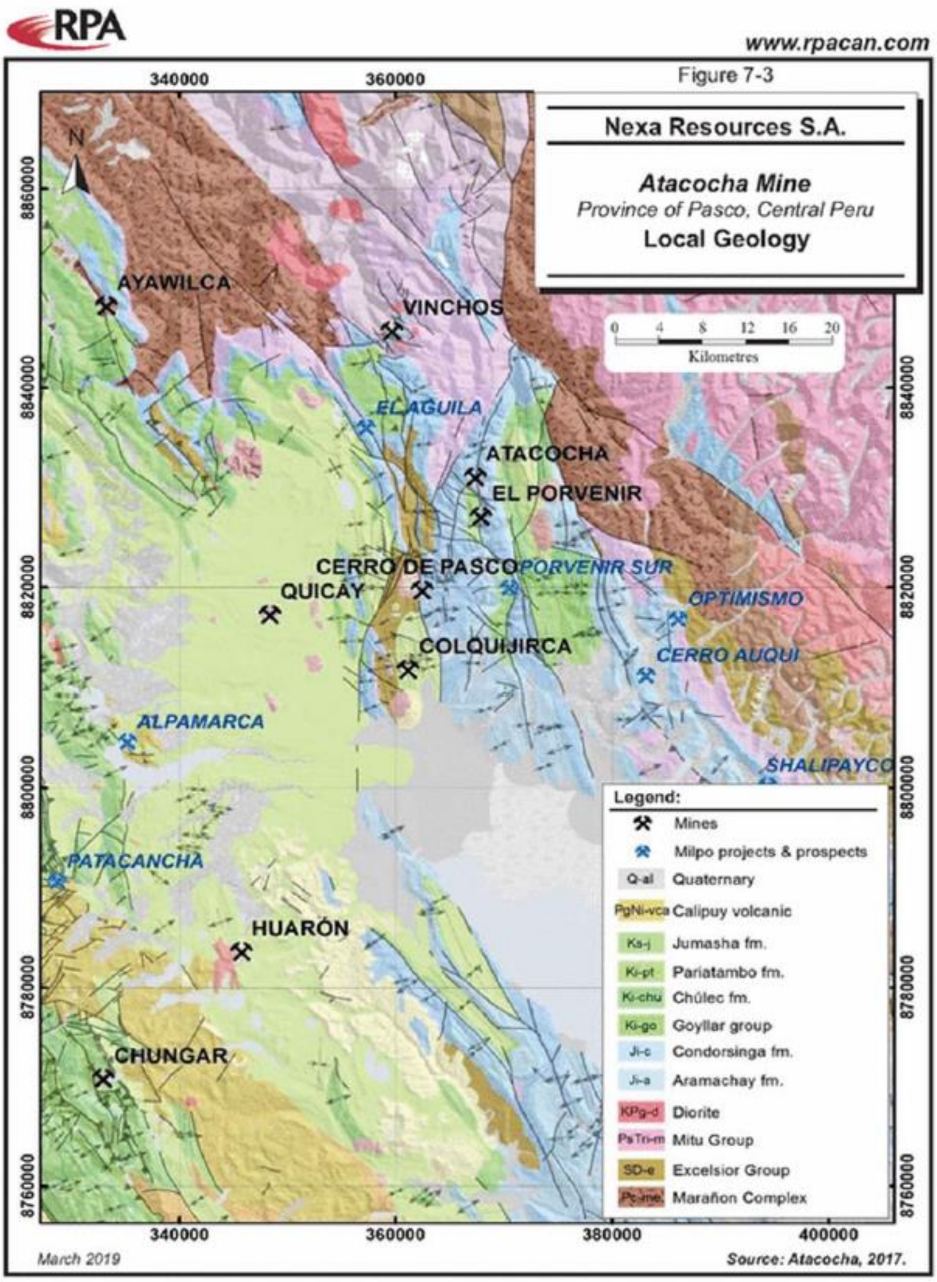


Figure 21: Regional geology of El Porvenir area (Ladd et al., 2019)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

	Mt	Zn Grade %	Cu Grade %	Pb Grade %	Ag Grade g/t
PROVEN	6.99	3.51	0.21	0.82	52.3
PROBABLE	9.22	3.84	0.24	0.70	42.7
PROVEN+PROBABLE	16.21	3.7	0.23	0.75	46.9

Table 4 : El Porvenir mineral reserves statement – data from Dec 2019

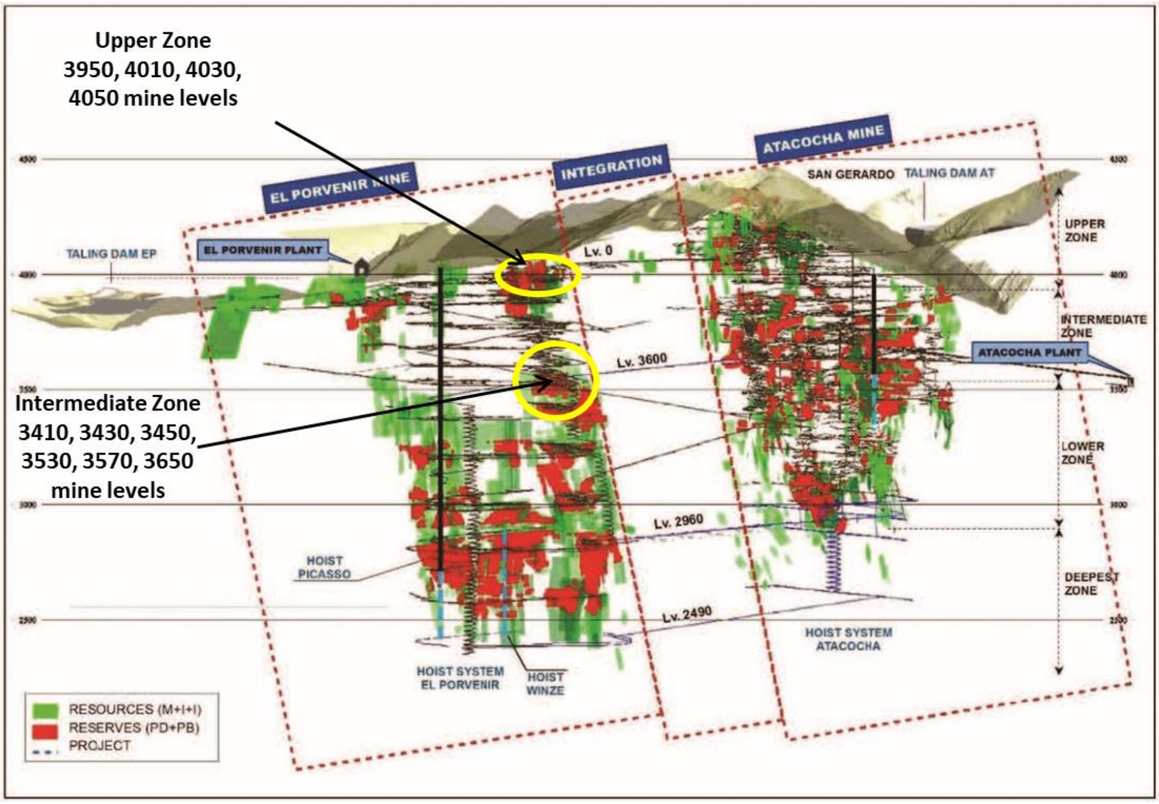


Figure 22: Description of El Porvenir mine and integration into Atacocha mine provided by NEXA[3]

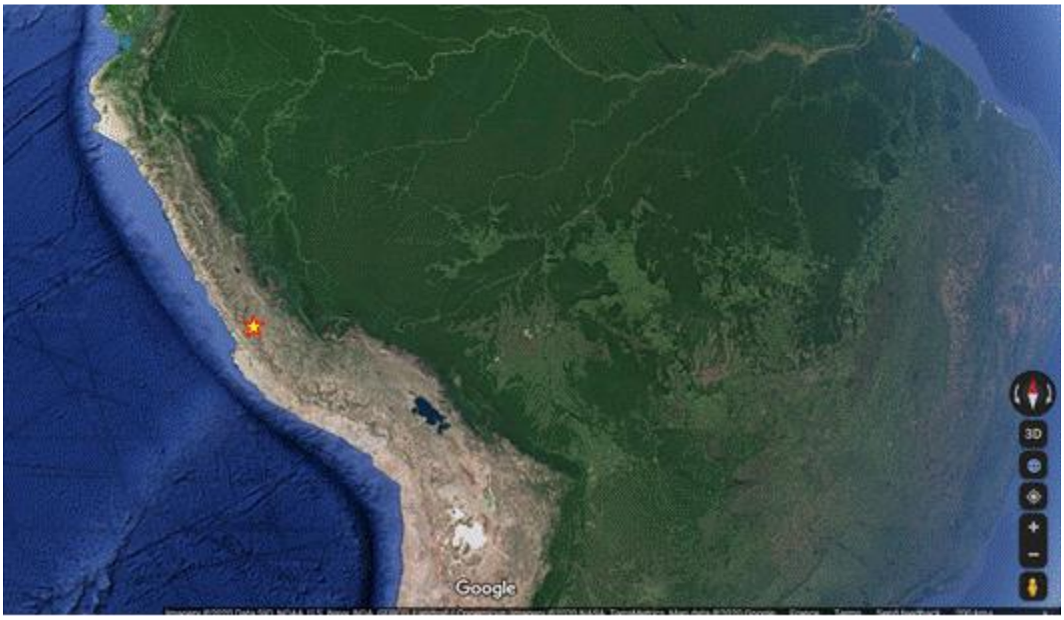
[3] Nexa Resources prospectus, October 2017

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Figure 23: Picture of El Porvenir mine (from CUM)

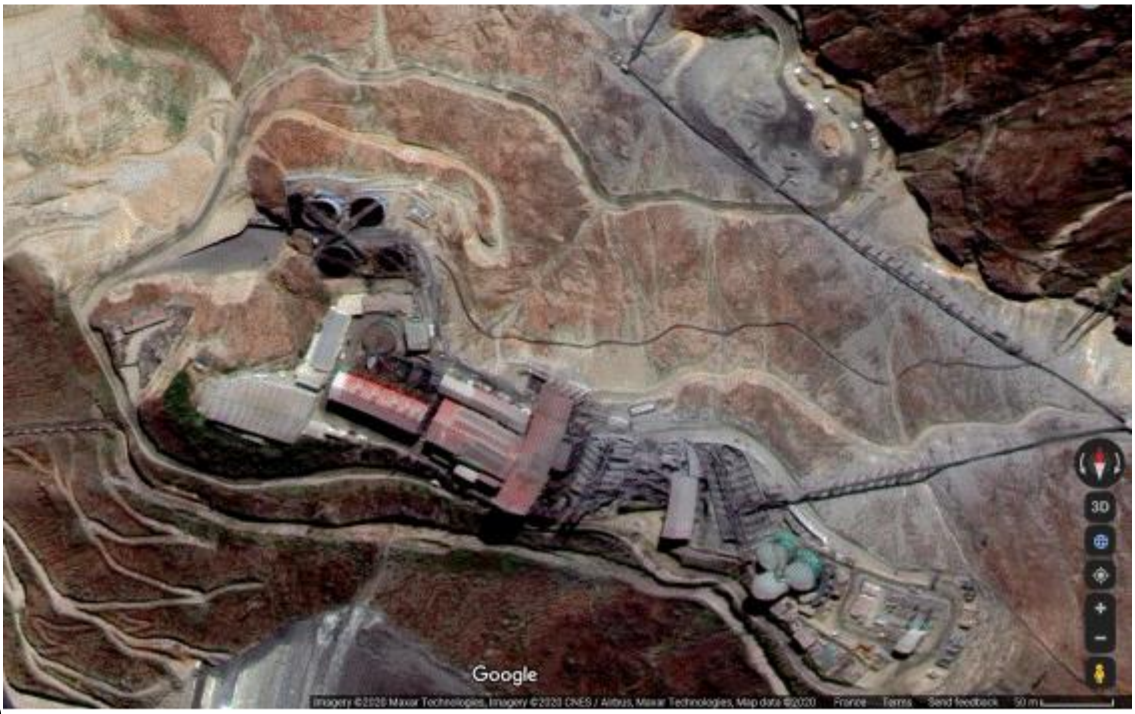
4.1.2 Cerro Lindo

Cerro Lindo mine is an underground mine located 243 km southeast from Lima (Peru). It is owned by Nexa Resources Peru. The Cerro Lindo Volcanogenic Massive Sulphide (VMS) deposit is hosted by the Cretaceous Casma Group volcano-sedimentary rocks near rhyolitic domes. It is among the world's 15 largest zinc mines[4]. Operations began in 2007. The map depicted in Figure 24(a) shows the location of the mine and an aerial view of the mine is available on Figure 24(b). The mineral reserves statement for Cerro Lindo mine is given in Table 5. A description of the mine is given in Figure 25 where the Zn and Cu rich areas are indicated with arrows and described in a table below with the precise names of the mining fronts that were sampled and the average grades for the ores.

(a) 

[4] https://www.nexareport.com/2018/en/nexa-resources/

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).



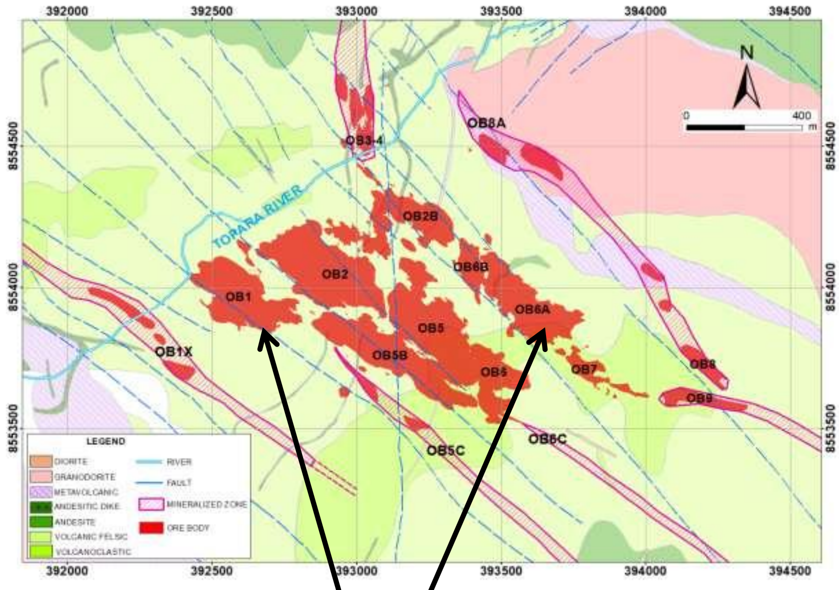
(b)

Figure 24: Location of Cerro Lindo mine in Peru (a) and aerial view of the mine (b)

	Mt	Zn Grade %	Cu Grade %	Pb Grade %	Ag Grade g/t
PROVEN	21.52	1.53	0.63	0.19	19.0
PROBABLE	26.84	1.05	0.67	0.14	18.7
PROVEN+PROBABLE	48.37	1.26	0.65	0.16	18.9

Table 5: Cerro Lindo mineral reserves statement– data from Dec 2019

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).



Cerro Lindo Ore samles for Ion4Raw							
Type	Zone	Orebody	Elev	Zn %	Pb %	Cu %	Ag oz/t
Zn Ore	Upper Zone	OB6A-T751A	1910	2,9	0,35	0,37	0,62
Cu Ore	Lower Zone	OB1-T021G	1600	1,31	0,02	1,92	0,98

Figure 25: Geological description of the Cerro Lindo mine provided by NEXA[5], location and description of the Zn and Cu rich areas

4.2 Sampling operation for ION4RAW process development

Sampling operations at the El Porvenir and the Cerro Lindo mines were performed by NEXA, in the presence of one person from Cumbrex Explorationes S.A.C. and one person from WAI at the beginning of October 2019. A picture of the sampling operations at El Porvenir mine is given in Figure 26. At El Porvenir, the sample was taken with a frontloader from the mine operations stockpile from the mixed ore of different mine fronts given in Figure 22. While at Cerro Lindo, the sampling was grab of one mining front for the Zn ore and another mining front of the Cu ore (see Figure 25 for ore descriptions and locations).

[5] Nexa Resources Prospectus, October 2017

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Figure 26: Picture of El Porvenir sampling operations

For ION4RAW project, 14 tons of ore were extracted form Cerro Lindo and form El Porvenir mines. Approximately half of each were shipped, i.e. 7,695 tons of ore from the El Porvenir mine, 3,094 tons from the Cerro Lindo in the copper rich area, 4,291 tons from the Cerro Lindo in the zinc rich area. These amounts of primary sample were packaged in big bags (see Figure 27) for shipment into containers. Transport was carried out by sea. They were sent to WAI facilities in UK.

Figure 27: Picture of big bag ready for shipment (sampling of El Porvenir and Cerro Lindo mines)

The following amounts of ores were required for analyses at BRGM (secondary sample):

- El Porvenir 25kg
- Cerro Lindo Cu 25kg
- Cerro Lindo Zn 40 kg

They were received at BRGM on june, 22th.

4.3 On site sampling for primary ore characterisation

Sampling of primary ore was performed by Cumbrex Exploraciones S.A.C while sampling operations by NEXA were in progress. The block samples were selected from the bulks of the mine sample aiming to be representative and with geological criteria for the mineral characterization. The following samples were sent to BRGM by plane: 4 blocks from the El Porvenir mine, 2 blocks from the Cerro Lindo for the

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

Cu rich area and 2 blocks from the Cerro Lindo from the Zn rich area. Samples were received at the BRGM on December 19th 2019.

4.3.1 Pre-treatment before characterisation

4.3.2 Blocks

Each block has undergone the treatment described in Figure 28. A total of 26 polished thin sections and 1 polished thick section were prepared.

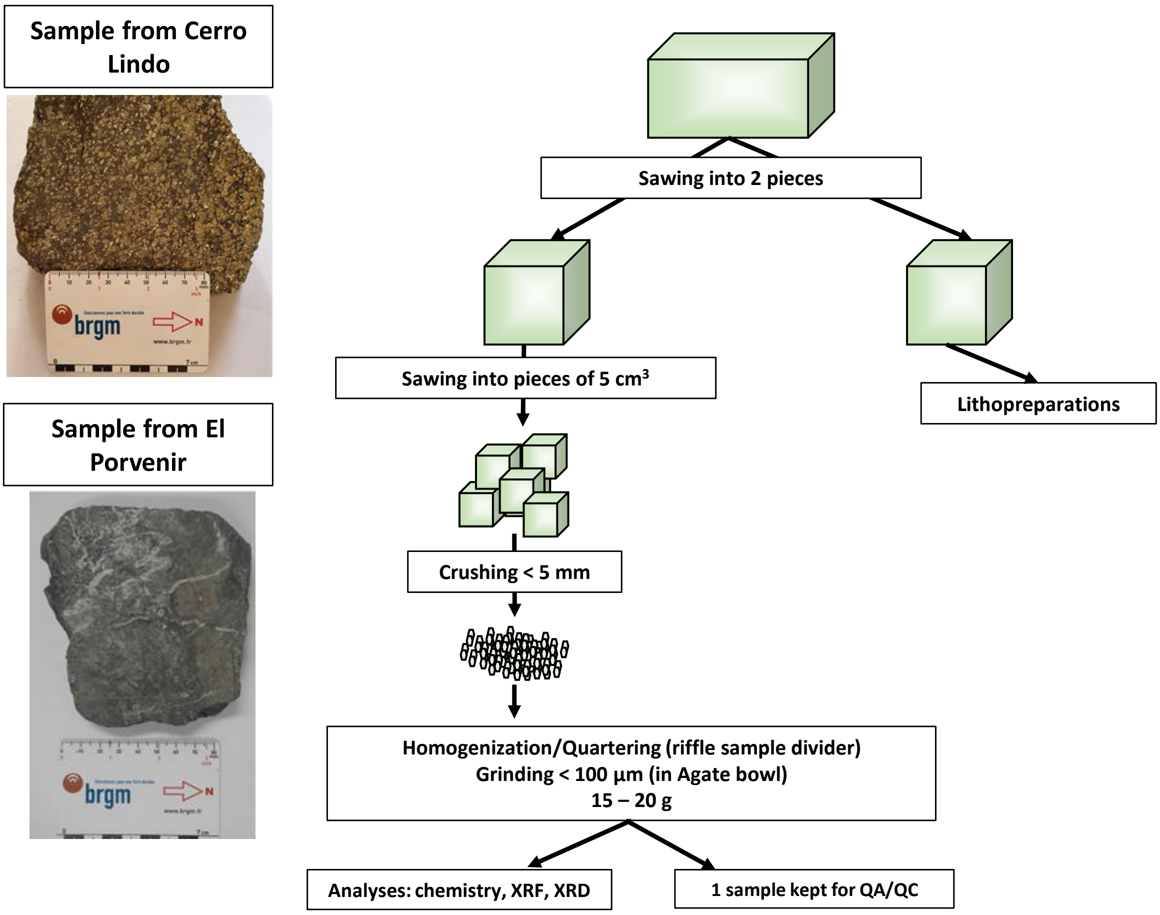


Figure 28: Pre-treatment chart before analysis steps for 8 blocks from Cerro Lindo and El Porvenir mines

4.3.3 Uncrushed as-received ore

The following charts present pre-treatment undergone by for samples from the Cerro Lindo (from Cu rich area), Cerro Lindo (from Zn rich area) and the El Porvenir, in Figure 29 to Figure 31. For each sample, a total amount of 10 polished thick sections and 4 polished thin sections were produced.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

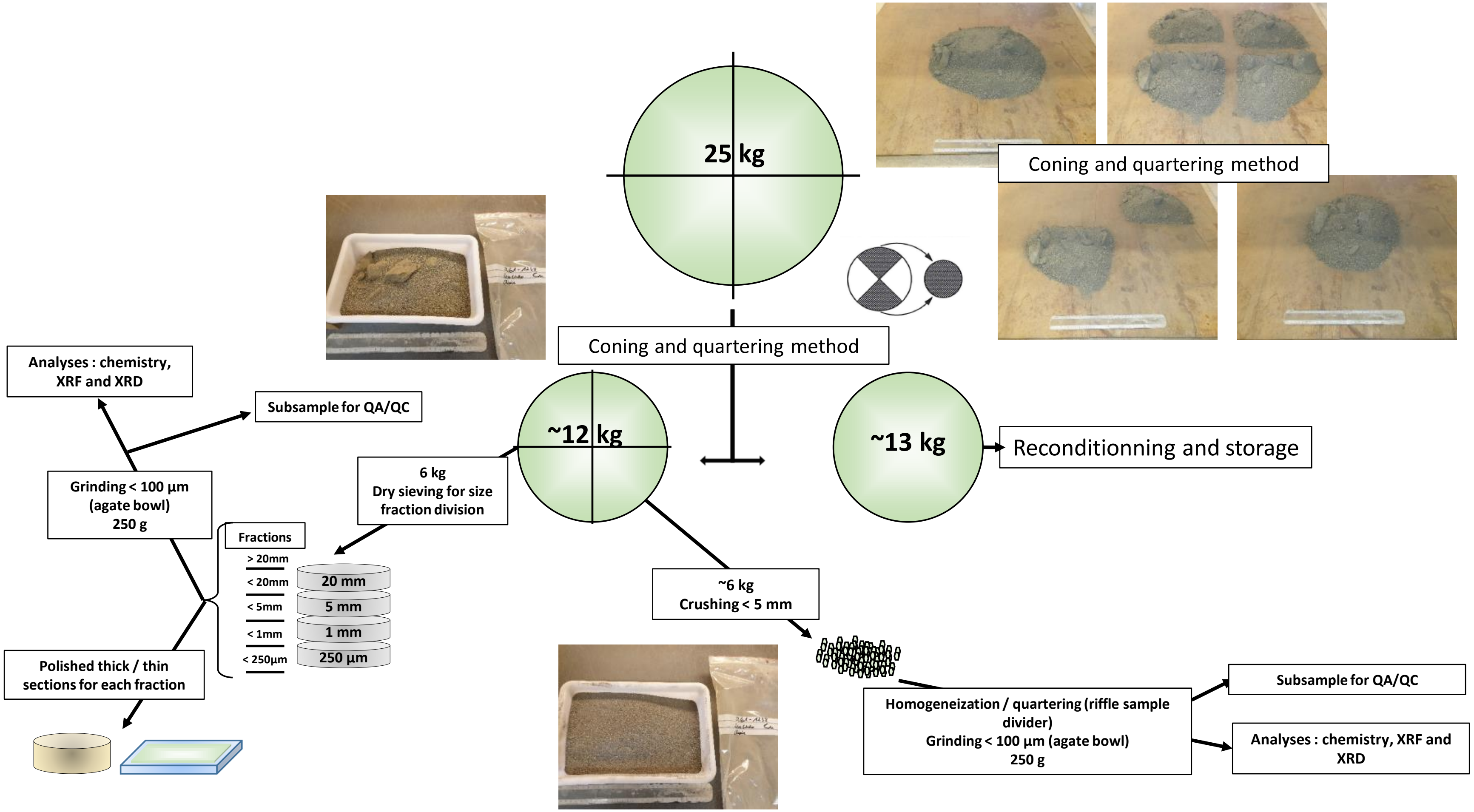


Figure 29: Pre-treatment chart before analysis steps for the Cerro Lindo sample from the Cu rich area

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

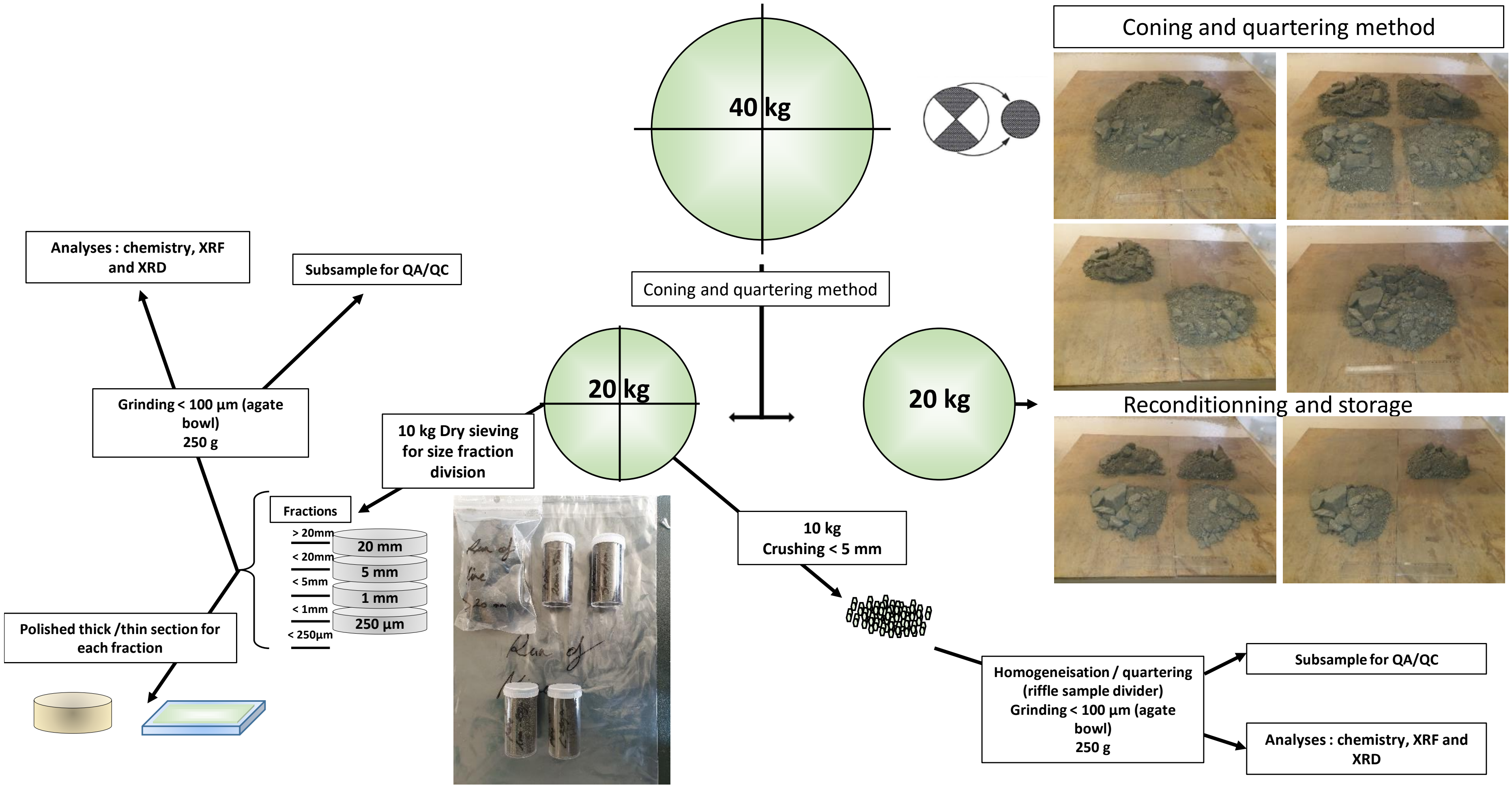


Figure 30: Pre-treatment chart before analysis steps for the Cerro Lindo sample from the Zn rich area

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

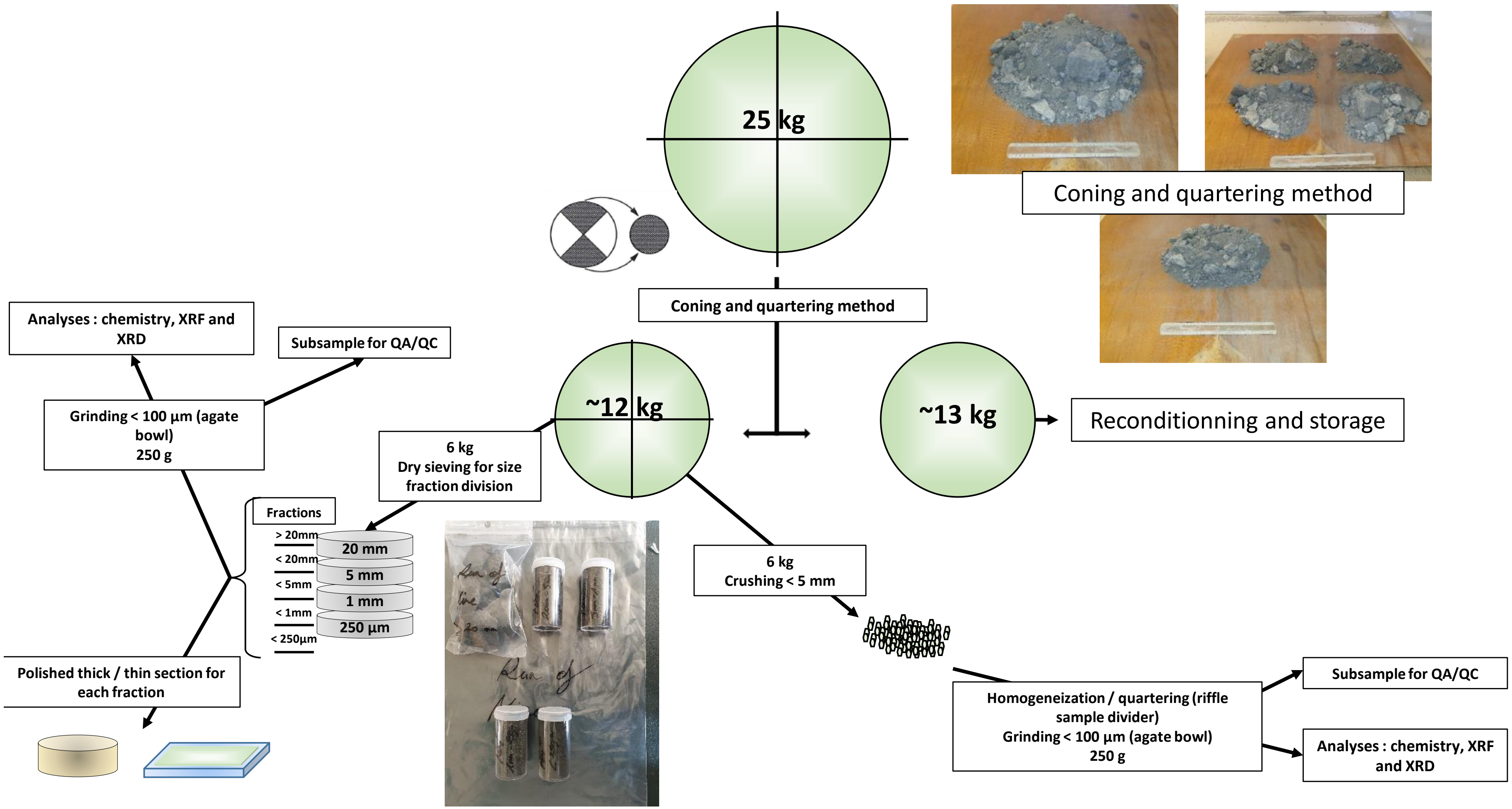


Figure 31: Pre-treatment chart before analysis steps for the El Porvenir sample

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

The detailed PSD carried out on a sub-sample of uncrushed as-received ore at WAI are presented in Figure 32 to Figure 34, together with the size division performed at BRGM. A difference could be observed for sample from the Cerro Lindo from the Cu rich area (Figure 32) as there is a larger population of grains around 1mm in the WAI sample than in the BRGM's fraction.

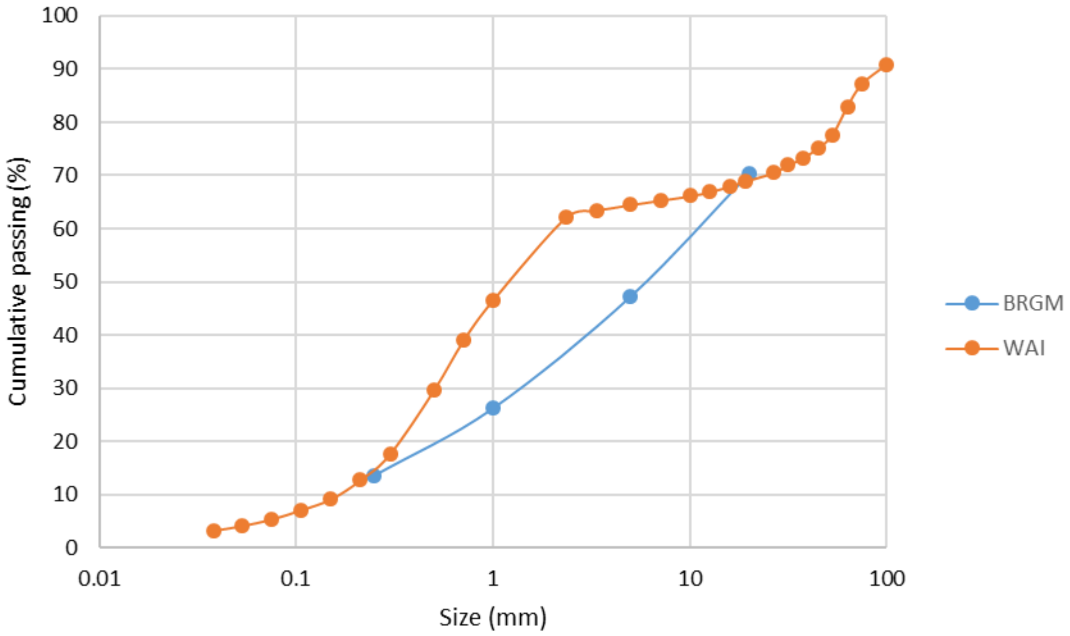


Figure 32: Cumulative particle size distribution for Cerro Lindo sample from Cu rich area

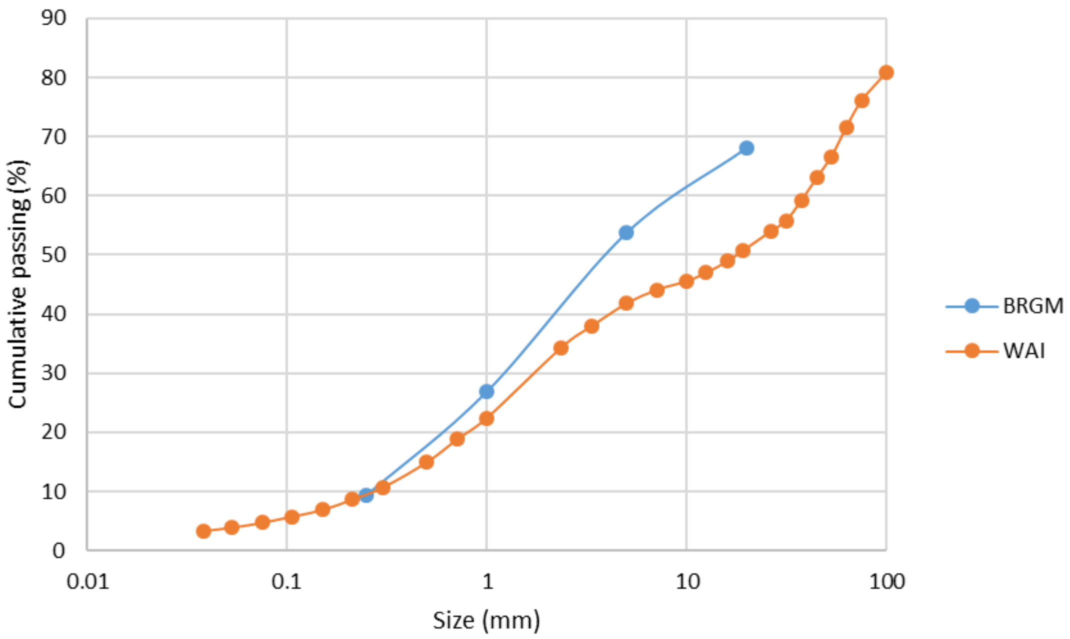


Figure 33: Cumulative particle size distribution for the Cerro Lindo sample from Zn rich area

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

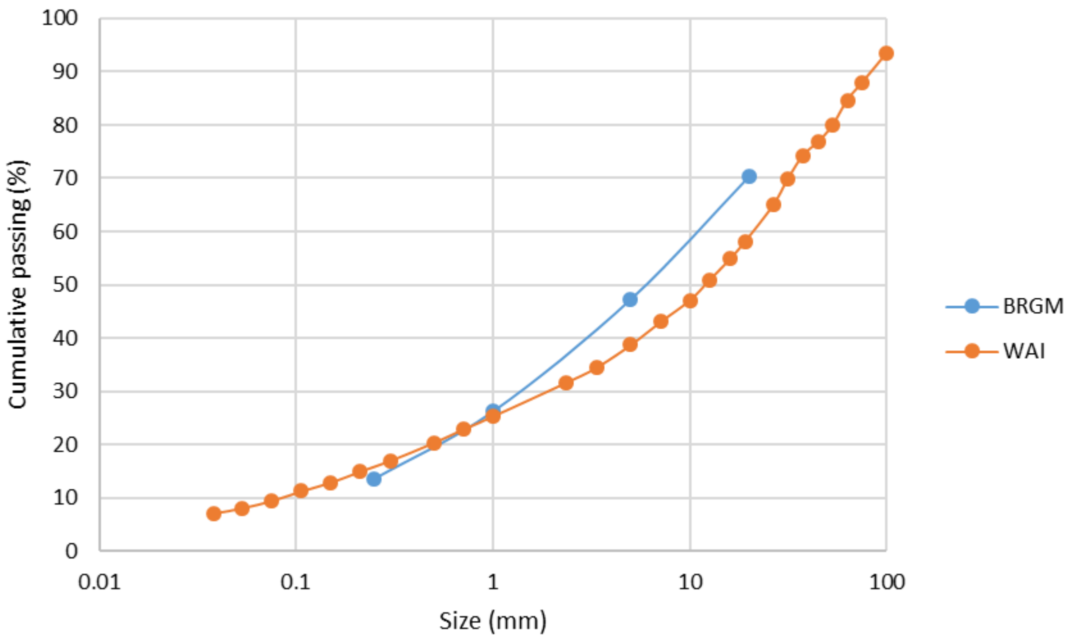


Figure 34: Cumulative particle size distribution for the El Porvenir sample

5 El Valle-Boinas sample

5.1 Sampling location and description

El Valle-Boinas mine is located 60 km southwest from Aviles in Spain. It is owned by Orvana Minerals Corp. The map (Figure 35 (a)) shows the location of the mine and an aerial view of the mine is available in Figure 35 (b). It consists of a gold-copper-silver mineralization. Figure 36 and Figure 37 show respectively the locations of the calcic-rich skarn mineralization and the younger epithermal vein mineralization. The mineral assemblages consist of chalcopyrite, bornite, (arseno)pyrite, magnetite, and pyrhhotite in the skarns, and native gold, electrum, native copper, chalcopyrite, and chalcocite in the epithermal mineralization[6]. Summary of Mineral Resources Inclusive of Mineral Reserves for El Valle & Carles is given in Table 6.

[6] https://www.orvana.com/English/operations/el-valle-boins-carls/geology/default.aspx

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

(a)

(b)

Figure 35: Location of El Valle Boinas mine in Spain (a) and aerial view of the plant (b)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

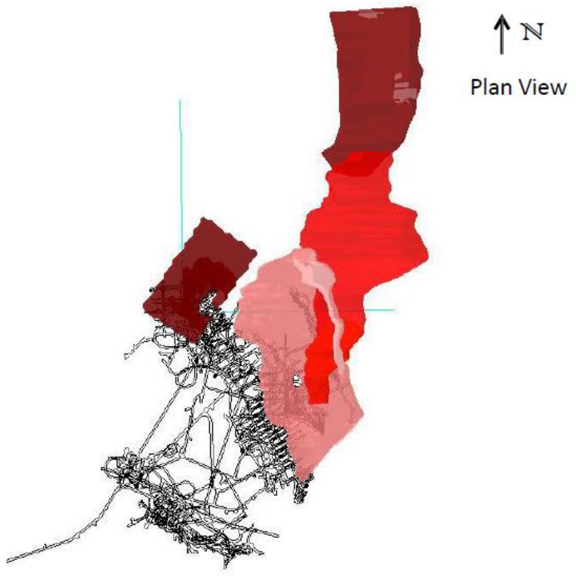


Figure 36: Location of the epithermal mineralization in El Valle Boinas mine (Orvana Minerals Corp. internal report)

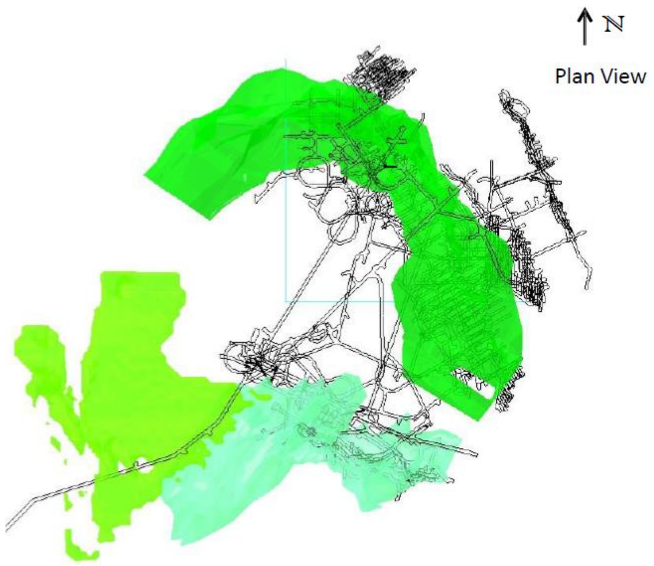


Figure 37: Location of the skarn mineralization in El Valle Boinas mine (Orvana Minerals Corp. internal report)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

	kt	Cu (%)	Ag (g/t)	Au (g/t)
BOINAS OXIDES	2,500.4	0.52	9.11	5.60
BOINAS SKARN	2,679.6	0.82	18.22	2.98
CARLES	1,179.2	0.49	9.39	3.89

Table 6: Summary of Mineral Resources Inclusive of Mineral Reserves for El Valle & Carles - September 30, 2019[7]

5.2 Sampling operation for ION4RAW process development

Sampling operations at the El Valle Boinas deposit were performed by Orvana Minerals Corp. at the end of December 2019. For ION4RAW project, 2 tons (primary sample) of ore were extracted from the mine and sent to WAI facilities in February 2020. Around 25kg of ore were required for characterisation at BRGM (secondary sample).

5.2.1 Pre-treatment before characterisation

The pre-treatment chart applied at the BRGM before the analysis steps is presented in Figure 38. A total of 10 polished thick sections and 4 polished thin sections were produced.

[7]https://s2.q4cdn.com/372236871/files/doc_financials/2019/Orvana-AIF-2019-Formatted-December-27-FINAL.pdf

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

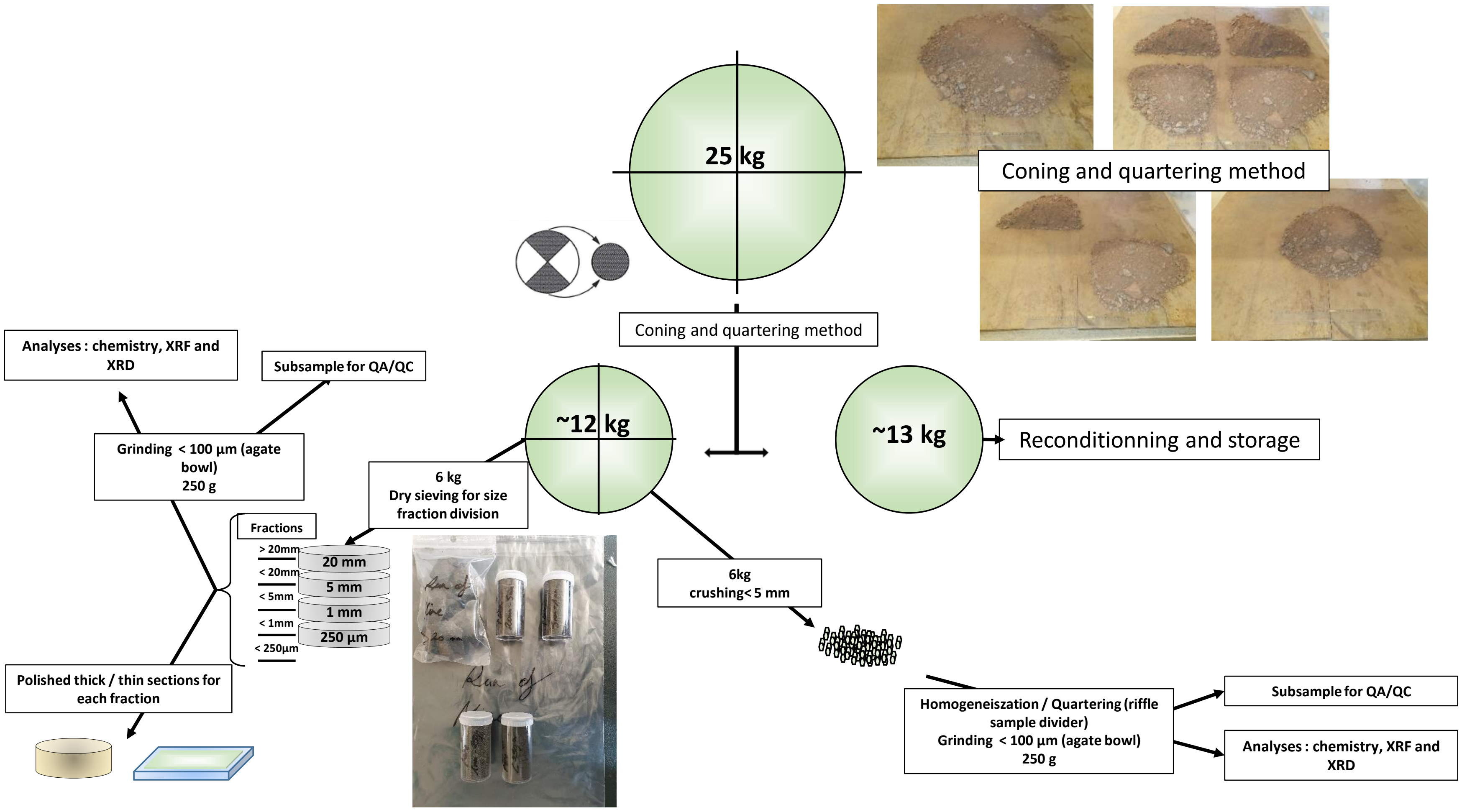


Figure 38: Pre-treatment chart before analysis steps for the El Vallee Boinas sample

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

The detailed PSD carried out on a sub-sample of uncrushed as-received ore at WAI is presented in Figure 39, together with the size division performed at the BRGM. relatively good consistency can be pointed out.

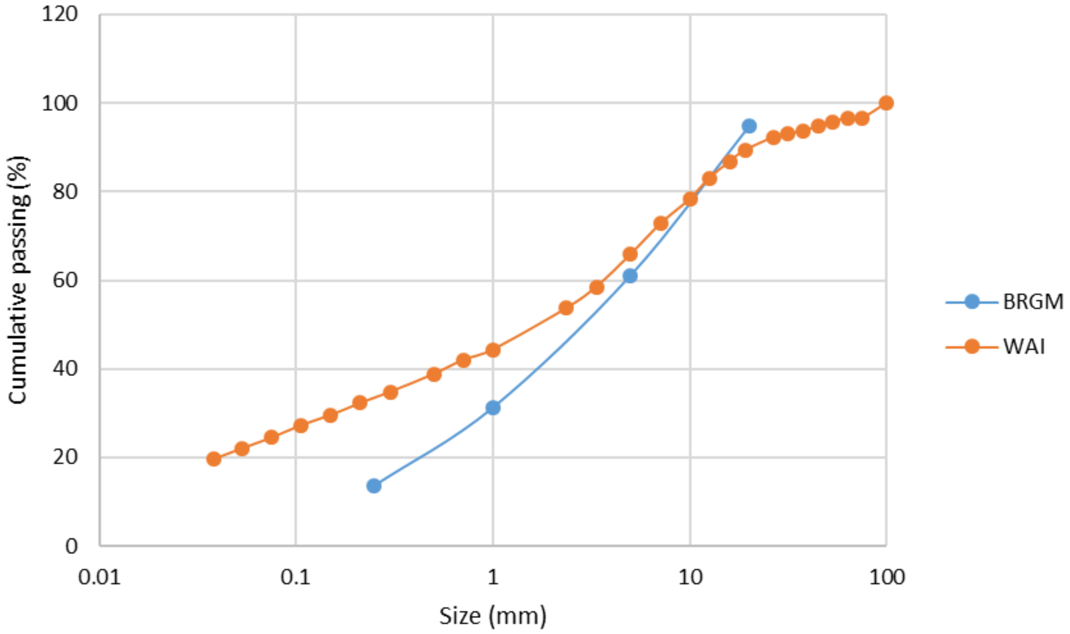


Figure 39: Cumulative particle size distribution for El Vallee Boinas sample

6 Difficulties encountered and solutions proposed

The main issues for sampling operations is the representativeness of samplings, at different scales:

- obtaining a representative sample from a site (e.g., order of magnitude of the amount : ton). Due to the spatial variability of the ore assemblage, representativeness of the site is almost impossible to ensure. There could be biases in the material sampled. For example, the project could not control what was sampled (host rock, run of mine…) because on site sampling was performed by the mining companies. Moreover the delay between sampling and shipping may vary significantly from the different mines. For example for the Cononish gold mine, sampling from the underground mine was performed 20 years ago and the ore was stored outside which may induce potential alteration and oxidation whereas samples from Peru were relatively fresh as they were sampled especially for the project.

- obtaining a representative sample of the stockpile received at WAI (order of magnitude of the amount: few tens of kilograms). These samplings were performed by WAI after homogenization/quartering. This step must ensure that the sample received for characterization is the same as the one used for preparing the concentrate that will be used by partners to develop the process. All good sub-sampling practices were applied to ensure it.

- obtaining a representative sample for analysis (order of magnitude of the amount: few grams). A sub-sampling and sample preparation procedure for bulk samples was established at the BRGM laboratory. It was as identical as possible for all samples to ensure that they all undergo the same protocol. All preparation steps were performed implementing good laboratory practices in order to minimize preparation errors and to get a representative sub-sample for analysis.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

CONCLUSION

Sampling and sample characterization are crucial steps when developing a treatment process on ore sample. To this end in the ION4RAW project, a sampling and sample physical preparation procedure was established at BRGM laboratory. It was applied for all bulk samples to ensure that all samples underwent the same treatment steps. This procedure included several steps among sample mass reduction, comminution (crushing and grinding) and homogenization. All preparation steps were performed implementing the good laboratory practices in order to minimize preparation errors and to get a representative sub-sample for analysis.

Sampling of 6 bulk ore samples were performed from July to December 2019. Two samples were already out of the mine (Cononish gold mine and Cobre las Cruces open pit) and they were stored outside in stockpiles. The four other samples were specifically sampled for the ION4RAW project. One from the El Vallee Boinas (Spain) and three samples from Peru: El Porvenir and two samples from the Cerro Lindo as there is two distinct areas (i.e., a Cu- and a Zn-rich areas). For each deposit, the required mass sample could vary from 300kg to 7 tons.

The complete characterization of the ores consisting of mineralogical observations and whole-rock chemistry will be detailed in the D2.3 deliverable of the ION4RAW project.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).

BIBLIOGRAPHY

Desroches, D., Bedard, L. P., Lemieux, S., & Esbensen, K. H. (2019). Representative sampling and use of HHXRF to characterize lot and sample quality of quartzite at a pyrometallurgical ferrosilicon plant. *Minerals Engineering*, *141*, Article 105852. https://doi.org/10.1016/j.mineng.2019.105852

Gerlach, R. W., Dobb, D. E., Raab, G. A., & Nocerino, J. M. (2002). Gy sampling theory in environmental studies. 1. Assessing soil splitting protocols. *Journal of Chemometrics*, *16*(7), 321-328. https://doi.org/10.1002/cem.705

Gy, P. (2002). *Sampling of particulate materials theory and practice (Vol 6)*. Elsevier.

Gy, P. M. (1982). *Sampling of particulates - Theory and Practice - second Edition*. Elsevier.

[Record #331 is using a reference type undefined in this output style.]

Mac Donald, N., & Conybeare, D. (2019). *Preparation of bulk sulphide concentrate for DES testing*.

Pitard, F. F. (1993). *Pierre Gy's sampling theory and sampling practice: heterogeneity, sampling correctness, and statistical process control*. CRC Press.

Saez, F., Gilbertson, J., Mondragon, A., Samaniego, A., Sepulveda, D., & Winkelmann, N. (2017). *Independant Technical Report pursuant to National Intrument 43-101 of the Canadian Securities Administrators for El Porvenir Mine*.

Spence-Jones, C. P., Jenkin, G. R. T., Boyce, A. J., Hill, N. J., & Sangster, C. J. S. (2018). Tellurium, magmatic fluids and orogenic gold: An early magmatic fluid pulse at Cononish gold deposit, Scotland. *Ore Geology Reviews*, *102*, 894-905. https://doi.org/10.1016/j.oregeorev.2018.05.014

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 815748. The content of this report does not reflect the official opinion of the European Union. Responsibility for the information and views expressed lies entirely with the author(s).