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Characterization of waste — Sampling of waste from extractive industries

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National foreword

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Charakterisierung von Abfällen - Probenahme von Abfällen der mineralgewinnenden Industrie

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Contents

Foreword

This document (CEN/TR 16365:2012) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/395), which assigned the development of standards on the characterization of waste from extractive industries.

This Technical Report is intended to supplement the existing series of five Technical Reports dealing with sampling techniques and procedures for waste, and provides specific information for sampling of waste from the extractive industry. It follows the principles laid down in EN 14899, *Characterization of waste — Sampling of waste materials — Framework for the preparation and application of a Sampling Plan*. Further information on the relationship between the production of a sampling plan and the overall testing programme objectives can be found in CEN/TR 15310-5.

— CEN/TR 15310-1, *Characterization of waste — Sampling of waste materials — Part 1: Guidance on selection and application of criteria for sampling under various conditions*;

— CEN/TR 15310-2, *Characterization of waste — Sampling of waste materials — Part 2: Guidance on sampling techniques*;

— CEN/TR 15310-3, *Characterization of waste — Sampling of waste materials — Part 3: Guidance on procedures for sub-sampling in the field*;

— CEN/TR 15310-4, *Characterization of waste — Sampling of waste materials — Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery*;

— CEN/TR 15310-5, *Characterization of waste — Sampling of waste materials — Part 5: Guidance on the process of defining the sampling plan*.

This Technical Report focuses mainly on sampling for geochemical rather than geotechnical requirements. Sampling for geotechnical requirements is only addressed to a limited extent and references are made to existing documentation. The Technical Report elaborates on a range of potential approaches and tools of specific relevance to the sampling and testing of wastes from the extractive industry. This approach enables the project manager to tailor his sampling plan to a specific testing scenario and continues the ‘shop shelf’ approach to sampling plan development for waste testing outlined in CEN/TR 15310-1 to -5. This approach allows flexibility in the selection of the sampling approach, sampling point, method of sampling and equipment used. It provides the necessary background information pertaining to the factors that influence the choice of these detailed components of the sampling exercise, and information on the necessary statistical choices that can then be applied to determine the most appropriate testing programme for any given sampling scenario.

This Technical Report also makes references to the overall guidance document for characterization of waste from extractive industries (CEN/TR 16376) which gives guidance and recommendations on the application of methods for the characterization of waste from extractive industries.

Introduction

The guidance outlined in this Technical Report is focused on the key elements to be considered in the development of a sampling plan for extractive waste. This report should be used in conjunction with EN 14899 and its supporting technical reports and is intended to supplement the information contained in these documents with specific and essential information relevant to the sampling of waste from the extractive industry. Where appropriate this report also makes reference to the overall guidance document for characterization of waste from extractive industries (CEN/TR 16376) which gives guidance and recommendations on the application of methods for the characterization of waste from extractive industries[1].

1) Specific features of extractive waste

The extractive industry includes, metal mines, rock quarries, salt mines, coal mines, sand and gravel, limestone and onshore oil and gas operations. When mineralogical material is extracted it is exposed to changes in physico-chemical conditions, which may result in chemical and physical instability of previously stable geological material.

The life cycle of extractive industries starts with the early phase of exploration through operation to closure and after care. In the context of sampling three phases have been defined in this document:

— exploration (including design and permitting);

— operation (extraction and processing, including transport and deposition of waste); and

— closure (including existing waste deposits).

From a sampling perspective different sampling scenarios may be more relevant than the operational phases. For example sampling from diamond drill cores may take place both during exploration and operation, sampling at existing waste rock dumps and tailings facilities may take place both during operation and at closed sites. Both operational phases and sampling scenarios are used as parallel concepts in this document.

One significant feature that makes characterization of extractive waste different from waste characterization in general is the fact that sampling and characterization ideally take place before the waste is produced, i.e. based on drill cores (or drill mud) from exploration drilling. Characterization during exploration is critical since subsequent waste management plans are developed on the basis of this information. However, the availability of material for sampling and characterization at the exploration stage is commonly limited which means that follow-up checks to ensure that the initial data and interpretation are correct will often be needed during operation. If pilot scale tests, extraction and/or processing, are carried out this will have the added benefit of producing a larger number of potential samples for sampling and testing as well as giving the opportunity to sample process waste, i.e. tailings. While the majority of waste is commonly produced during the operation phase of a mine, waste characterization needs to be considered for all phases of the mine life.

The operational phase of a mine or quarry encompasses all the activities from mineral deposit development to detailed planning for closure. There are two main waste streams from the production process that need to be characterised, i.e. waste material generated as part of the extraction that will not go through mineral processing and the waste produced during processing. The waste produced prior to mineral processing will primarily be waste rock separated at the excavation front. In a hard rock mine, sampling may be done before blasting from drill cores, or after blasting. After mineral processing the waste will primarily be tailings (i.e. tail end of the process), and samples may be collected from pipelines, discharge trenches or conveyer belts. Extractive waste may contain chemicals added as part of the production process. Normally, if not recovered for construction purposes, all extractive waste is deposited on site.

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1) As defined in Directive 2006/21/EC.

This guidance is also applicable for sampling from closed sites in case sampling and testing of waste is required. Sampling at closed sites, including abandoned historic mine-sites, may in some cases require specific approaches e.g. due to accessibility and limited background information.

NOTE Given the great variety of waste types, sampling situations and objectives, this Technical Report cannot provide definitive instructions that cover all scenarios. Instead, it discusses the basic considerations to be followed, and provides guidance on selection of sampling approaches that might be relevant to the three principle phases of a mine: 1) Exploration, 2) Operation and 3) Closure. Sampling of existing waste deposits at mines that are still in operation would be very similar to Scenario 3) Closure.

2) Document structure

The structure of this sampling guideline is based on the concepts and procedural steps outlined in Figure 2 of EN 14899:2005 and subsequent subclauses, with some additions to address specific features of the extractive industry.

Clause 2 key elements of a sampling plan, is the core of this guidance document. This clause is divided into ten sub-sections that describe the steps of developing sampling plans, from defining the involved parties to describing the sampling techniques. It lists possible objectives for the different stages of the extractive waste characterization, background information that may be available, explains generic levels of testing and describes sampling approaches and techniques.

1 Scope

This Technical Report gives additional and specific information on sampling for testing of waste from the extractive industry to support the development of appropriate sampling plans. This supplementary guidance to EN 14899 is required because waste from the extractive industry differs considerably from the waste types and sampling scenarios covered in the existing technical reports (CEN/TR 15310-1 to -5) that support the Framework Standard. This guidance document should be used in conjunction with EN 14899 and its supporting technical reports CEN/TR 15310-1 to -5.

The approach to sampling described in this document is primarily focused on the requirements to undertake mineralogical and geochemical testing of the waste. Whilst much of the background information provided is also relevant to geotechnical investigations there may be important additional requirements or differences in approach for determining relevant physical parameters. For example, many geotechnical parameters are determined using field tests, which are not discussed in this document. References to alternative source documentation are provided.

The guidance provided in this document applies only to above-ground exposure to radio-nuclides present in the undisturbed earth crust and **not** to the production, processing, handling use, holding, storage, transport, or disposal of radioactive substances that are or have been processed for their radioactive, fissile or fertile properties.

This Technical Report provides some discussion of current best practice, but is not exhaustive. To clarify the text, the document provides a number of worked examples in the Annexes.

2 Key elements of a sampling plan

2.1 General

The sampling plan identifies the appropriate and practical activities required to achieve the set objectives of the characterization testing programme. The purpose of a sampling exercise shall be clearly understood by the sampler. The development of a sampling plan helps to ensure that the objectives of any waste testing programme are consistently met and is crucial for cost effective and appropriate sampling. The sampling plan provides traceability which can be used to validate the data produced. This is especially important where new datasets will be generated over time.

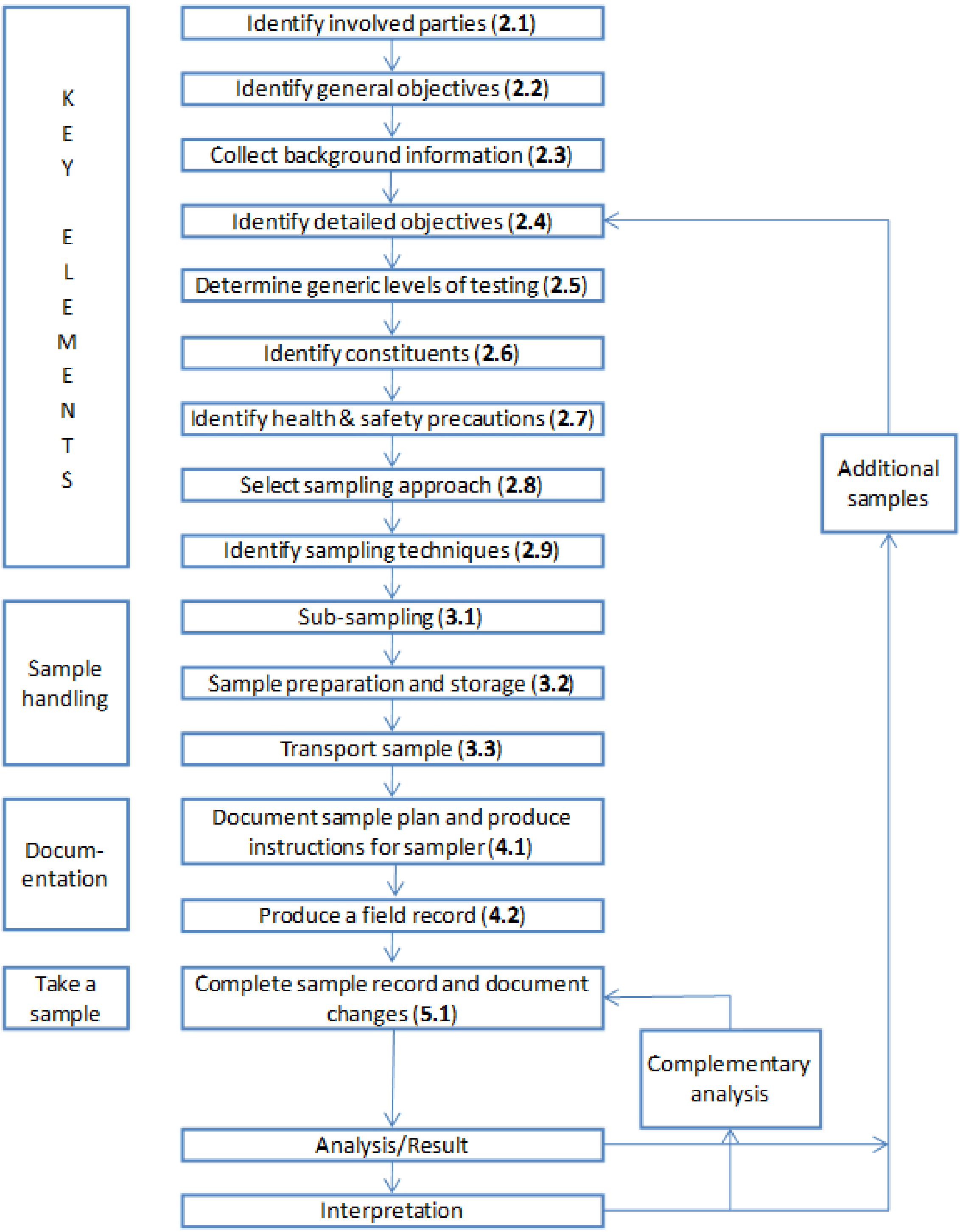
The framework standard EN 14899 identifies a process flow chart that defines the essential elements of a sampling plan and how those elements are linked. The basic steps identified in this flow chart have been followed in this supplementary guidance, with some minor changes (see Figure 1) to account for specific circumstances of the extractive industry. The flow chart indicates a step by step process to sampling plan development, although in reality they may be considered out of order. Some elements of the sampling plan may be prepared in parallel and iterations may be necessary. Additional information is provided in the following sections that are specific to the extractive industry.

It is important to recognise that characterization may often be an iterative process. The sampling plan may initially be developed for screening purposes and may then form the basis of a characterization study. The characterization study may require testing of samples previously collected but not subjected to testing or form the basis for collection of more samples required for more comprehensive testing all specified in the sampling (Figure 1).

Characterization of waste from a quarry may be relatively simple in comparison to wastes from metal extraction. However, generating a sampling plan using the outline approach advocated in this guidance document is still recommended. Development of a comprehensive sampling plan will facilitate discussions with stakeholders, and it may help the operator to identify issues that may require consideration.

2.2 Identify involved parties (EN 14899:2005, 4.2.1)

It is recommended that sampling plans for a given site and phase of operation are, where possible, discussed with involved parties prior to any sampling taking place. It is important to identify and include parties with an interest at an early phase in the testing programme. This may include e.g. people from different parts of the organisation of the operator, consultants, regulators and local stakeholders. This approach may avoid or minimise confusion at a later date and facilitate acceptance from involved parties. Well-communicated sampling plans may provide confidence that the results generated from the sampling and testing programme will be valid.



The numbers in the chart refer to the clauses within this document.

Figure 1 — Flowchart of sampling plan modified from Figure 2, EN 14899:2005

2.3 Identify general objectives (EN 14899:2005, 4.2.2)

The overarching objectives of a testing programme define the type and quality of information required from sampling and analysis. During the various phases in the life of a mine there may be more than one objective and therefore a need for more than one sampling plan. Whilst the overall objective can be identified prior to any background data collection and on-site investigation the technical objectives can usually only be set once this information has been gathered.

Example overarching objectives may be to contribute data to:

— design a waste facility;

— development of a waste management plan;

— design/evaluation of closure options;

— design/evaluation of management options for abandoned sites.

The overall guidance document (CEN/TR 16376) includes a discussion on when and why characterization may be needed and the context within which characterization data may need to be applied. However, it does not cover information on how to apply these characterization results, e.g. for dam design or closure planning. For guidance on how to use characterization results correctly for predictive modelling or design purposes references are made to other sources of information.

2.4 Collect background information and undertake field inspection (EN 14899:2005, 4.2.5)

2.4.1 General

It is essential to obtain pertinent background information to develop good sampling plans for future, existing, closed and abandoned facilities. The background information allows the development of clear and precise instructions for sampling. Background information can be divided into three categories: existing information including site background information; field inspection information; and analogous geology. These three categories are discussed in the following clauses.

2.4.2 Existing information

Obtaining background information on a proposed extractive operation and collating this information to feed into the development of a testing program and sampling plan is essential when defining appropriate detailed objectives for sampling and testing. Careful inclusion of valid prior information can greatly reduce the cost per unit of error and dramatically reduce the size of the samples required. Relevant existing information may include data from previous exploration work as well as investigations carried out for other land uses or local data on background concentrations of key constituents in soils surface waters.

The use of existing information would typically be complemented by a field visit. It is important to emphasise that although existing data may be very useful, its relevance should be evaluated prior to using the data in any decision making process. If data is available from previous sampling programmes, the evaluation of suitability would include looking at the:

1) type of sampling undertaken;

2) consistency of test data and analytical procedures used;

3) objectives of previous sampling and testing.

If a site moves from exploration to operation a new sampling plan will need to be developed and the waste characterization data used for design and permitting will then become the background information for the operation stage.

The overall guidance document (CEN/TR 16376:2012, Clause 5) gives further recommendations on site background information that may be useful.

2.4.3 Field inspection

A field inspection provides valuable information on local conditions, e.g. any access restrictions that may impact upon the planned sampling approach e.g. appropriate selection of sampling points. The information that may be gathered by a field inspection will be very different for an exploration site compared to an on-going operation or a closed waste facility. Examples of information that should be collected include:

a) *Exploration*

   1) topography, accessibility;

   2) geology;

   3) mineralogy/iron phases in drilling material, overburden and exposed bedrock;

   4) relevant environmental factors.

b) *Operation*

   1) accessibility;

   2) geology;

   3) mineralogy/iron phases;

   4) additives;

   5) grain size variation;

   6) erosion features and slumping;

   7) excavation and transport methods;

   8) material type (tailings, waste rocks);

   9) potential hazards;

   10) relevant environmental factors.

c) *Closure and closed sites*

   1) accessibility;

   2) geology;

   3) mineralogy/iron mineral phases;

   4) seepage/seepage colour and possible field measurements of seepage;

   5) water/air erosion features at disposal facilities;

   6) grain size variation;

   7) slumping;

8) potential hazards;

9) relevant environmental factors.

Signs of water and air erosion, especially on tailings, can indicate, to some degree, the extent to which the waste material has been transported from its original position. The following information can be used to identify the area to be sampled.

— erosion features;

— aeolian transport of tailings from original deposit;

— stability of existing tailings and waste rocks.

Mapping the deposit mineralogy and the presence of iron phases (for sulfide containing mineralisation) is an essential part of evaluating the risks associated with current and future waste from the site. The mineralogy rather than the bulk chemistry provides a first indication of potential drainage quality, acid generating minerals, and neutralizing minerals. If the rocks have been exposed to air and water variations (where groundwater is being lowered due to pumping) over short or long periods, secondary minerals may give an indication of how the waste will behave when it is subsequently exposed to air and water.

The type of processing and use of additives, and type of waste being generated will give an indication of the type of analyses that may be necessary and define health and safety precautions during sampling and handling of the samples. If first evaluation indicates that there is a potential for acid/neutral rock drainage (A/NRD) or leaching from tailings, it may be necessary to obtain samples from a pilot processing plant.

The presence of seepage, colour and field measurements of the seepage, aid in defining, for example, the level of testing and type of analysis. These observations and field measurements give an indication of the oxidation reactions and leaching taking place within the waste material. For example:

— Drainage pH and total dissolved solids may indicate if there is already A/NRD or alkaline drainage from existing waste dumps or from the mineralization of *in situ* outcrops.

— Colour of drainage water or waste rock may provide an indication of A/NRD or alkaline drainage and the rock's neutralizing potential and reactivity. The colours yellow, orange, red, brown are commonly linked with different iron phases precipitated as a result of acid drainage.

2.4.4 Analogous sites

During evaluation of background information it may be possible to identify where the mineral deposit has analogous sites with information. Information from the analogous sites may be used to identify key issues and help focus the sampling plan on relevant aspects. In some cases this information from the analogous sites may replace the need to undertake some or all of the planned sampling.

2.5 Determine specific objectives and corresponding level of testing (EN 14899, 4.2.3)

2.5.1 Introduction

The technical objectives of a testing programme and sampling plan, which may be numerous, affect the location, number and volume or weight of samples taken, minimum testing requirements and precautions needed to preserve sample integrity during transport to the laboratory. The same samples can often be used for several tests. By identifying all the data requirements prior to collecting any sample and by storing samples in such a way that their integrity is maintained, they can be used for multiple tests and also re-used at a later date.

2.5.2 Determine the level of testing

Examples of potential sampling objectives linked to the phases of operation and related sampling scenarios at an extraction site or mine are provided in Figure 2 and Table 1. There are a couple of worked out examples of sampling plans to illustrate the variety of sampling plans for different type of operations in the annexes from the complex metal mining during permitting phase to simple quarry operations during production with confirmation testing. Moreover, the overall guidance document gives examples of typical questions to be answered and guidance on methods available for determination of specific properties.

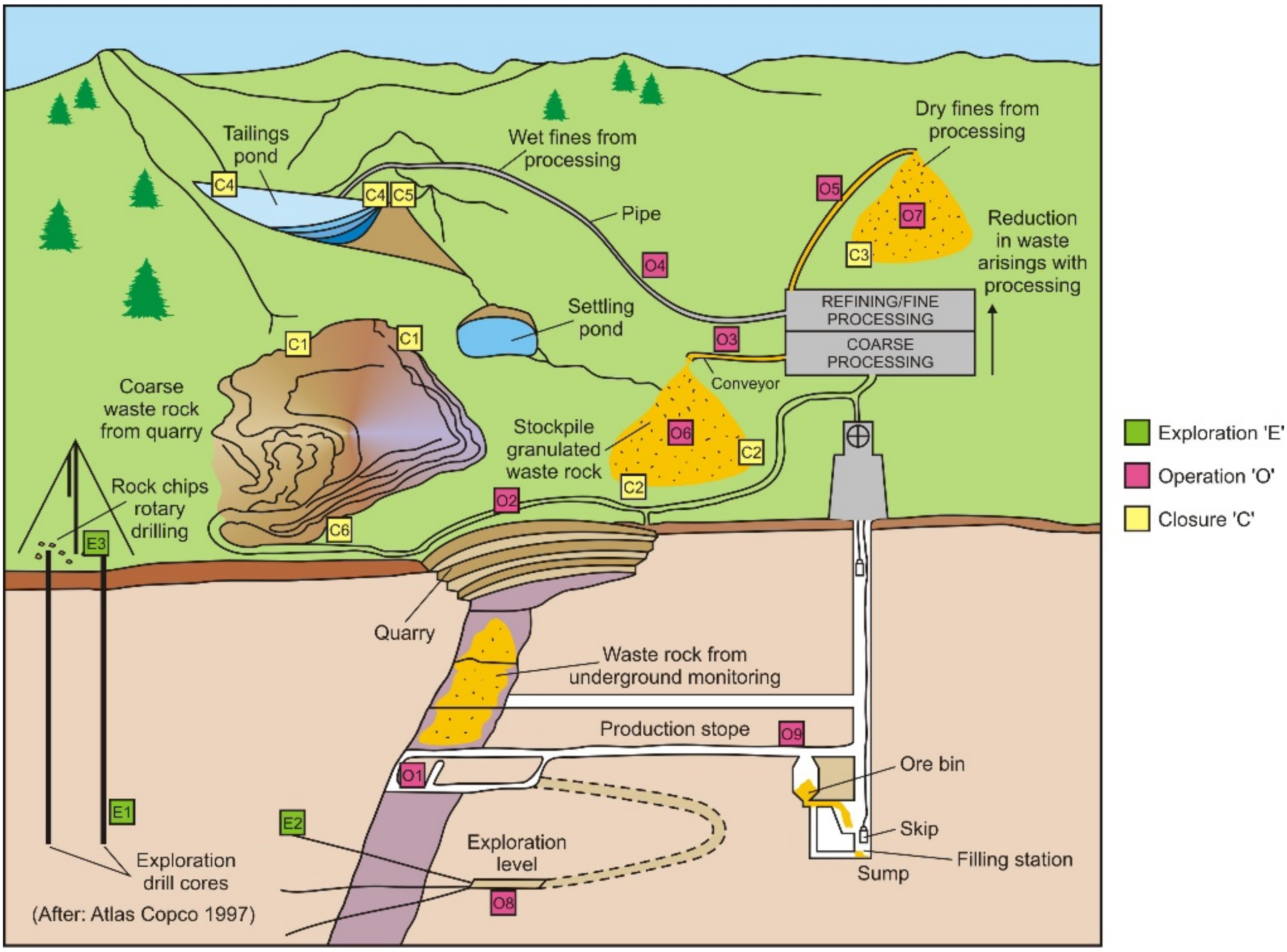


Figure 2 — Schematic of phased operation of mineral extraction linked to potential sampling objectives

In the context of waste characterization for the extractive industry at least three main objectives (Table 1) can be readily identified:

Screening characterization: this objective, sometimes termed basic characterization, applies typically for the waste rock characterization during the exploration stage where waste-rock samples are commonly taken from drill cores. Screening typically includes a set of quick and simple tests to get an overall picture of waste categories and variability. The screening is commonly performed early during the exploration phase, especially if sulfides are present. Screening level testing may be all that is performed or necessary to perform for mineral deposit that is not likely to generate drainage with poor quality water.

Table 1 — Example sampling objectives linked to the different phases of an extraction site or mine

Phase	Code	Scenario	Material type	Objective
Exploration (E)	E1/E2/E3	Anticipated extractive wastes from exploration or development by sampling excavation fronts or drill material.	rock cores, cuttings, sludges, in-situ rock	Screening or Comprehensive characterization
Operation (O)	O1	Blasted rock from excavation front during operation.	in-situ rock	Comprehensive characterization or Confirmation
	O2/O3/O4/O5	Excavated or processed waste in pipelines or on conveyer belts.	dry coarse rock, wet coarse rock, wet fines; dry fines, tailings slurry (clay to coarse sand)	Comprehensive characterization or Confirmation
	O6/O7	Extractive waste in existing dumps.	coarse rock, dry fines	Comprehensive characterization or Confirmation
	O8	Exploration or development drill material.	dry coarse rock, wet coarse rock, wet/dry fines	Comprehensive characterization or Confirmation
	O9	Blast material from excavation front.	dry coarse rock, wet coarse rock, wet/dry fines	Comprehensive characterization or Confirmation
Closure (C)	C1/C2/C3/C6	Extractive waste in existing dumps.	coarse rock, dry fines,	Screening, Comprehensive characterization or Confirmation
	C4/C5	Previously deposited tailings.	saturated fines, unsaturated fines	Screening, Comprehensive characterization or Confirmation

Comprehensive characterization: for a new operation, this may be performed later in the exploration phase. Comprehensive characterization typically includes more costly and time consuming test work like leach tests and/or kinetic testing together with a higher number of samples. Comprehensive characterization may not be needed when screening gives a clear result with an appropriate level of confidence. For current operations there may be a need to perform comprehensive characterization if this was not performed before or if the composition of the waste has changed significantly.

Confirmation testing usually consists of a few samples collected and tested for key parameters identified through the comprehensive characterization in order to determinate whether waste produced conforms with expected results, e.g. fits with the design criteria/requirements set in the permit or specified in the waste management plans. The testing programme will commonly be built upon the results from screening or the comprehensive characterization. Due to the long life-time of a typical mine, significant amounts of data are likely to result from on-going confirmation testing, which should be used for continual updating of waste management and closure plans.

The objective shall also be linked to the ‘population’ that requires evaluation. In the evaluation of the variability the ideal aim is to have access to the whole population that requires evaluation, whilst during confirmation sampling access is typically reduced to identify sub-populations within the waste (e.g. a waste stream generated under a certain condition in a fixed time regime). For example confirmation samples taken from drill cores or pipelines during the operation of the mine give access to an easily defined sub-population. When sampling from a dump or a dam, commonly only the perimeters are sampled and thus the samples are commonly not representative of the whole “deposited tailings” population (this would require that every particle of waste has an equal chance of being sampled).

In the majority of cases the general objective (2.3 and the more specific objectives) of a testing programme are too general to be useful as an unambiguous instruction to the sampler. It is, therefore, necessary to translate this objective into a number of practical technical goals, which provide a more detailed specification for the sampling activity, which can then be further developed and linked to specific sampling and analytical requirements.

Commonly a phased approach will be needed to meet each technical goal; each may require its own specific sampling plan.

An example where a general objective is translated into a technical goal or instruction might be:

The objective of the sampling and testing programme is to evaluate the general acid base accounting (ABA) characteristics of a mineral deposit, with known carbonates and variable sulfide, for different geological units. The associated technical goals might be in two parts; the first would be to sample the geological units and test for total sulfur, ABA and mineralogy (screening). The second technical goal could relate to a targeted second round of sample collection and analysis using the results of the phase I testing (comprehensive sampling and testing).The specific objectives may range from simple field confirmation, like observing if the colour is constant or that a specific mineral is present or not present, to very detailed objectives as part of a comprehensive characterization scheme.

2.5.3 Determine the required number and size of samples

The number of samples required for source characterization of each material type depends on the following:

a) the amount of disturbance (i.e., the volume/mass of material extracted or the amount exposed on pit/mine walls or production tonnage as determined by the block model);

b) the compositional variability within a material type; and

c) the statistical degree of confidence that is required for the assessment.

Initial estimates of sample numbers are typically based on professional judgment, and experience. The number of required samples typically grows during each of the early phases of mine development as the knowledge base and project needs grow. Ultimately, for sites characterized as having ARD potential, a full geostatistical model often provides the basis for control plans where material segregation is part of the mine plan. (GARD, 2009)

The size of the samples to be collected is in many cases dictated by the required analytical methods. However, it is recommended that a larger sample (3 kg to 4 kg) is collected to ensure representativity and ensure adequate sub-sample size for each analytical test. If the objective is comprehensive characterization of the waste material, several samples may be needed for each of the test methods and substantially larger samples may be needed. Although many of the laboratory test methods require only 100 g to 200 g of material, ensuring that the sample is representative requires due consideration of the particle size and variability at the scale of sampling.

Relatively undisturbed samples may also be useful when performing leach tests/column-kinetic tests on coarser tailings material. The sample can be collected in a Plexiglas/acrylic (or similar) cylinder. By using such a sample it is possible to approximate the original flow path of the material for both water and oxygen and, thereby, obtaining more reliable/applicable results than if using a grab sample (see 2.9.3.4).

The number of samples will be dependent on some or all of the following aspects:

— stage of the project;

— complexity of the geology;

— questions to be answered (objectives);

— type and complexity of the operation;

— environmental issues of concern;

— degrees of uncertainty required or accepted;

— variability in the material of parameters to be analyzed.

The number of samples that should be collected is difficult to evaluate before an initial dataset has been generated. The BC Guideline (1990) suggests for potential sulfidic waste, a minimum amount of samples as a function of mass of waste unit (10 000 ton > 3 samples; 100 000 > 8 samples; 1 000 000 > 25 samples, 10 000 000 > 80: 100 000 000 > 260 samples). This system was based on the experience of the authors of the document. Australian Guidance on sample numbers for mining environmental assessment (2007) does not specify sample numbers. It describes the need for increased numbers of samples as the exploration project develops and that an optimum number of samples should be collected as part of the permit application.

2.6 Identify constituents to be tested (EN 14899:2005, 4.2.4)

The sampling plan should identify the properties (e.g. colour, grain size, hydraulic conductivity) and constituents (e.g. elements and mineral concentrations) to be investigated. These will depend on the material type and the objectives of the testing programme which will differ between extractive sectors, geographical locations and phases of the operation. Examples of specific information that may be relevant are given below.

— mineralogical analysis (identification of minerals, texture);

— geotechnical methods (e.g. soil index properties such as grain size distribution; specific surface, specific weight; Atterberg limits; and permeability; as well as mechanical properties such as shear strength; and compressibility);

— geochemical analysis (content of sulfur species, metals, static tests, kinetic tests);

— leaching tests;

— biological tests (ecotoxicity test, microbes in the waste material, growth potential).

Furthermore, the texture and grain size of the material can dictate choice of tests and therefore of sampling techniques (see 2.9.3, 2.9.3.3, 2.10 and Clause 3),

2.7 Identify health and safety precautions (EN 14899:2005, 4.2.6)

The sampling plan should identify all necessary health and safety precautions that should be followed during the sampling programme. It is recommended that a hazard assessment is carried out before any sampling at a site to protect, and minimise any risks to, or created by, those involved in on-site activities. The type of health and safety equipment that may be needed on site to perform sampling according to the sampling plan should be specified. Any organisation involved in sampling would be expected to have a health and safety policy that sets out the requirements of safe working.

The health and safety issues are commonly very different for the exploration phase of mining relative to the operational phase, and again different from the issues that may arise in sampling of closed/abandoned waste deposits. However, some general issues can be identified for all three settings and especially to the production of emergency plans in the event of an accident taking place during sampling.

A mine should have a detailed health and safety plan, in which case the sampler should be aware of it, and in some cases, be trained by the operational personnel. Some general health and safety issues may include:

— very large equipment (e.g. 320 tonne trucks) where driver visibility is greatly reduced at close range (e.g. 30 m);

— dumping of waste material on areas to be sampled;

— unstable out-slopes;

— saturated tailings;

— loose material in the production phase;

— unstable material in pits, trenches and underground tunnels;

— poor air quality as a result of mineral reactions (e.g. H_2S, CO_2, CO or CH_4 gas) or from the equipment used; and

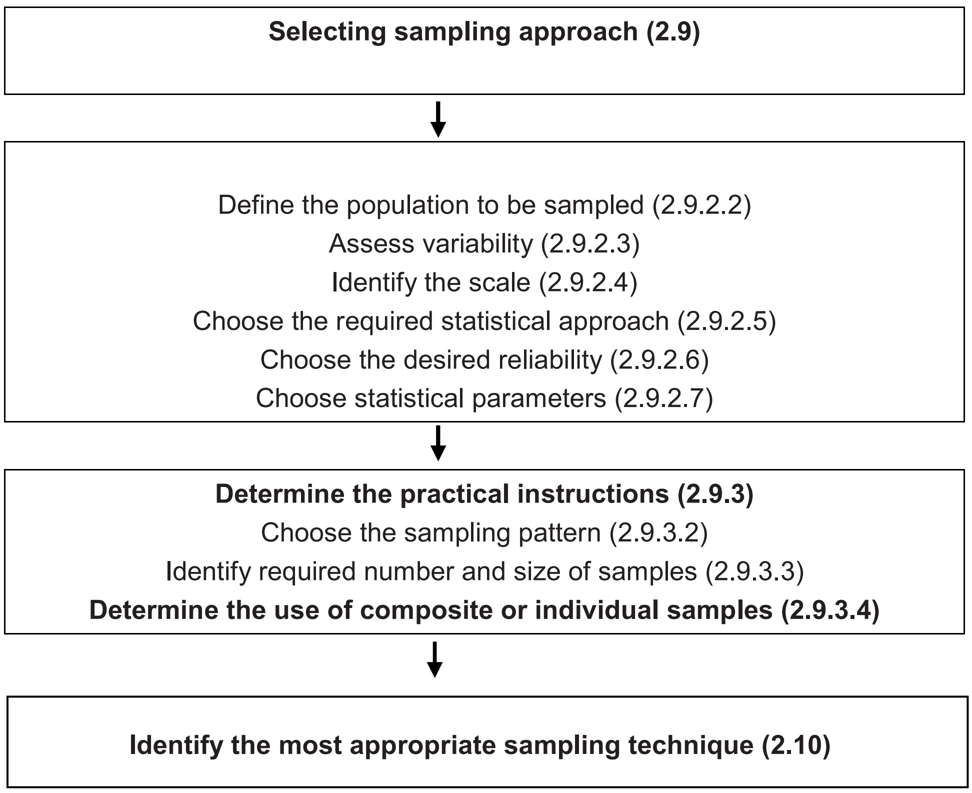
— dust problems.

Detailed guidance on safety during site investigations and sampling is given in ISO 10381-3 and in EN 14899:2005, 4.2.7.

2.8 Select sampling approach (EN 14899:2005, 4.2.7, and CEN/TR 15310-1:2006, Clause 4)

2.8.1 General

One of the key activities in the development of a sampling plan is the consideration and selection of what can collectively be regarded as the 'statistical criteria'. The importance of this step is identified in 4.2.7 of EN 14899:2005. Due consideration of these criteria ensures that appropriate boundaries are set for the sampling exercise, and in particular that the type and number of samples taken will ensure that the data collected is fit for purpose. Generally, the process identified in Figure 3 should be followed in the development of any sampling plan.



The numbers in the figure refer to the subclauses in this document.

Figure 3 — Flow chart showing the possible steps to take in developing sampling approach

The sampling approach commonly used in the exploration and operation phases for evaluation of mineral reserves can also be subsequently used for sampling waste. The guidance provided in the following clause is consistent with commonly accepted statistical principles outlined in EN 14899 and CEN/TR 15310-1:2006, Clause 4.

Determination of the sampling approach will identify how, where and how many samples should be collected and what they need to be tested for. This activity can be divided into the development of the technical goals and translating these into practical field instructions that can be followed in the field.

2.8.1.1 Population

Two terms of population are described in this clause: Overall population and sub-population.

The term overall population represents, in this document, the total volume/mass of waste about which information is required through sampling. The overall population may be the total extent of an ore deposit or volume of waste to be generated during production for the permit period or the lifetime of a mine. In practice, it may be difficult to sample from the overall waste population because the whole mineralization may not be well defined and the “future waste” can be assessed only through selected drill holes and/or surface samples. It is, therefore, customary to define the population for sampling as a convenient sub-set of that overall population which importantly is believed to be typical of the overall population, but which restricts sampling to a more convenient spatial extent or timeframe. Geological and/or process knowledge is the key to define an appropriate sub-population, as it relies on experienced judgement.

Defining one or more sub-populations may be necessary to:

— target known changes in mineralogy,

— take changes in the production process into account, and/or

— undertake a 'particle' division where access is restricted, e.g. operating tailing dams.

During exploration it is important that drill holes are installed throughout the geological formation that eventually will constitute the waste material. Geological logging, usually performed on the drill cutting/cores can be used to define sub-populations that may require separate handling and therefore separate sampling and testing programmes. Where mineralization is exposed on the surface the identification of sub-populations may be based on geological mapping and geological units. Where this approach is used sampling and testing should be undertaken to verify the sub-population scheme and identify the key parameters and constituents for waste characterization. Examples of geological characteristics that may be used for defining sub-populations are:

— rock types (granite; limestone; basalt, etc.);

— alteration zones (serecitisation; hydrothermal; supergene, etc.);

— mineralogy (pyrite – non-pyrite; mineral assemblages, etc.).

Table 2 gives examples of sampling locations relative to sampling population and sampling approach. Codes used in the table refer to Figure 2.

Table 2 — Examples of sampling approach for specific objectives and locations and codes listed in Table 1

Sampling location	Code	Sampling population	Materials description	Sampling approach
Exploration drilling	E1/E3	Total rock cores/chips to be generated during exploration phase	Rock cores/chips	Based on geological sequence, variability, depth and inclination of strata to ensure all formations/units are intercepted and representative core lengths/chip intervals are obtained.
Operation drilling	E2	Total rock cores/chips to be generated during operation phase	Rock cores/chips, drill mud	Based on geological sequence, variability, depth and inclination of strata to ensure all formations/units are intercepted and representative drilling material are obtained.
In situ ore/ development rock	O1/O8/O9	Total waste/potential waste to be generated during the extraction	In situ rock	Based on geological sequence and variability ensure representative samples are obtained from all relevant strata/geological units.
Conveyers	O2/ O3/ O5	Waste produced by processing areas during the life of the mine	Dry and wet, coarse rocks	Fixed single sampling point, allows samples to be collected over time and assess temporal variability.
Large dumps	O6/ O7/ C1,/ C2/ C3/ C6	Total amount of waste intended to be deposited in dumps	Corse rocks and dry fines	Location and number of samples to suit size of waste dump, safe access and the need for representative range of samples. NOTE This is very difficult to achieve once the dump established.
Tailings pipelines	O4	Waste produced by plant during lifetime of mine	Tailings slurry	Fixed single sampling point, allows samples to be collected over time and assess temporal variability.
Tailings dams	C5/ C6	Total amount of waste intended to be deposited in a tailings facility	Unsaturated and saturated fines	Location often limited by safe access. Requires a depth campaign to ensure all horizons are identified. In a complex deposit this may require continues sampling.

It is also important to define the sub-population explicitly over space and/or time; if this is not done, it is impossible to say whether a particular sampling exercise will result in representative samples. In defining the population for sampling it is important to consider the issue of ‘scale’.

2.8.1.2 Variability

A key element in designing a testing programme is to understand the main components of variability in the population being investigated. In general, variability is a characteristic of the waste that cannot be changed without intensive manipulation of the waste. The more that is understood about the spatial and temporal variability of the material under investigation, the greater the opportunity will be to use that knowledge in designing a sampling programme that will produce representative results.

For example, if a preliminary sampling exercise shows that the week-to-week variation in the waste rock from a production process is much greater than the variation within any single day, this indicates that, to characterise a month or even the annual production of waste from the plant it would be a waste of effort to take several samples in any one week. The most reliable result would be obtained by taking a sample from as many different weeks as possible. However, temporal variation is indicative of spatial variation since the processing method is commonly relatively constant over smaller timeframes (months). The temporal variation of the waste stream can, therefore, be used to evaluate the spatial variation in addition to processing variations. Spatial variation and processing method can also be used to indicate how often temporal sampling should be conducted for different waste streams.

The types and causes of any variability in the waste to be sampled should be understood and used to ensure sampling is appropriately targeted. The different types of variability that may need to be addressed include:

— If a waste is consistent and temporal variability low then it is reasonable to select a short sampling timeframe. Depending on the purpose of sampling, knowledge of a marked temporal cycle would give an option to (a) sample systematically over the cycle to smooth out that component of variation (composite sample of a drill core from e.g. tailings or exploration drilling), or (b) target the sampling to the worst point in the cycle (sulfide containing layers in a waste dump).

— If spatial variability is high and stockpiling and moving the waste leads to mixing, it may be more appropriate to sample the material as it is placed for final storage than direct from the production line (e.g. composite tailings drill core). This should be assessed together with the waste management plan in order to see if mixing should be avoided.

— The more that is known about the factors that affect the waste quality the more it will be possible to reduce the need for new sampling (e.g. defined hydrothermal alteration zones).

— Temporal variability of climatic changes (e.g. wet and dry; cold and warm, frost and thaw) may influence issues like secondary mineral stability, moisture content, and stability.

2.8.1.3 Scale

The chosen scale (or lot – a term often used parallel to the term “scale") defines the volume or mass of waste material that a sample represents and it is linked to the variability in composition of the material that will be studied. The scale of sampling should be agreed between involved parties prior to sampling and testing. The scale of sampling shall be consistent between different phases of testing, although some testing during characterization may be needed at more than one scale. Testing will generate a mean concentration value for the parameters of interest at the chosen scale and the variability between samples at a small scale may be more pronounced than the values generated from larger scales. This means that hot-spots of differing quality within the scale of sampling will be compensated for by larger amounts of ‘normal’ quality material within that same volume. The selected scale will be a trade-off between the level of variability in the material that is important to the testing objective and what is feasible in terms of the required accuracy, available resources and management of sample size.

Compositing, i.e. use of composite samples, may be a useful tool to identify the mean characteristics of a larger scale which may be a core interval or rock volume, such as an open-pit mine bench depth or waste zone. However, information on the smaller-scale characteristics may be lost due to the “smearing” of geochemical properties and analytical results. This “smearing” may lead to samples with anomalous qualities not being recognized, even though it may be those materials that govern the composition of a mine or process effluent.

The mine geologic model and block model may be used to select representative samples. If geochemical testing indicates that special handling of waste materials will be required, the block model may be populated with diagnostic A/NRD indicator parameters (e.g. total sulfur). In this case, a comprehensive set of discrete samples would be needed to build the geostatistical model (GARD 2009).

Depending on the objective of the testing programme, the scale of sampling may be equal to the size of individual particles of the waste (for particulate waste materials), the size of the sub-population or even the whole population of waste under investigation. At each stage for the test programme, the purpose of the test work should be considered to determine whether the results impact on management of materials on site or pose a risk to the environment. In some cases, the provision of contingency mitigation measures coupled with operational testing during mining will be more effective than additional pre-mining prediction test work, which could be inconclusive or of limited significance to the overall mine plan (Price, 2009). Test data may indicate that a change in the selected scale may be needed to provide additional information.

Scale can also be defined in terms of time. For example if the population is the total amount of waste produced in one year, the scale may be one year (the whole population) but also one month, week or day, depending in the objective of the testing programme. The scale is defined by the mass of the waste deposit divided by the amount of samples that will be collected.

a) Exploration phase:

For a proposed underground mine, the scale of sampling may be large due to a paucity of drill holes into the intended waste rock material. The scale will therefore, to a large extent be dictated by the scope of the exploration programme. Where there is substantial mineralogical variability within the mineral deposit a larger number of drill holes will be required to characterise the ore and will allow for a reduction in scale.

b) Production phase:

1) Drill cuttings from one blast hole at the production front could be taken to represent the material half way to the next drill hole. The variability within each blasting unit will indicate how many of the blasting holes should be sampled, which again identifies the scale of the sampling and how much waste material each sample will represent.

2) The scale can be a one-day production of tailings transported through a pipeline to the tailings dam. Depending on the variability of the tailings, a single sample or a composite compiled from several increments are collected from e.g. the discharge to the dam.

3) Similarly the selected scale for a granular waste material may be a one-day production of waste on a conveyor or a part of a stockpile produced during specific production conditions. For example. if the chosen scale represents a huge volume and the property of concern exceeds the specified quality criteria for the disposal of the waste, this may lead to costly additional management procedures at the disposal site. A balance needs to be struck between liability and an increased testing burden.

c) Closure phase and closed sites:

An appropriate scale of testing at a waste dump may be changed depending on the results from the first round of sampling.

2.8.1.4 Statistical approach

EN 14899 distinguishes two primary types of sampling; termed ‘probabilistic’ and ‘judgemental’ sampling.

Probabilistic sampling is traditionally defined as a statistically based approach whereby any part of the population has an equal chance of being sampled, and this allows limits of uncertainty to be calculated for any resulting data. This is in direct contrast to judgemental sampling, where samples are taken from a restricted sub-population (e.g. the edge of a tailings pond or side of a stockpile) may not necessarily be representative of the population and sample numbers and sample locations are usually considered to have no statistical basis. Combined approaches are also taken whereby probabilistic sampling is undertaken within certain sub-populations identified judgementally, or whereby judgemental sampling is used to complement probabilistic sampling.

Figure 4 shows examples of probabilistic and judgmental sampling with: probabilistic sampling; probabilistic within one judgmentally selected layer; systematic-judgmental; judgmentally selected area of the population; discrete zones selected judgmentally.

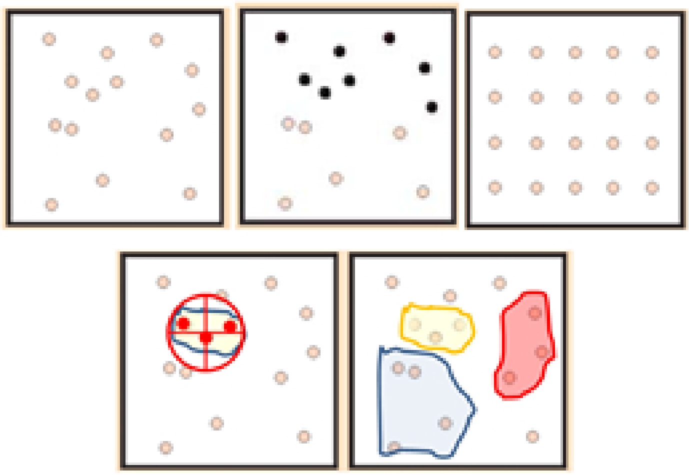


Figure 4 — Typology of sampling: probabilistic, stratified, systematic, judgmental, and discrete

Geostatistics (a branch of statistics that offers a method for analyzing spatially correlated data) is also applicable to characterisation of extractive waste. It has originally been developed to predict probability distributions of ore grades for mining operations and is a useful tool for waste rock characterization. Geostatistics is not tied to a population distribution model that assumes, for example, all samples of a population are normally distributed and independent from one another. Most of the earth science data (e.g. rock properties, contaminant concentrations) often do not satisfy these assumptions as they can be highly skewed and/or possess spatial correlation (i.e. data values from locations that are closer together tend to be more similar than data values from locations that are further apart). To most geologists, the fact that closely spaced samples tend to be similar is not surprising since similar physical and chemical depositional/transport processes have influenced such samples. Geostatistics therefore incorporates both the statistical distribution of the sample data *and* the spatial correlation among the sample data.

The application of geostatistical theory can also provide a means to assess the variability of sampling in time and space using techniques such as variogram, krigeage and confidence intervals to estimate the ore reserves in mining (as defined by Matheron, 1963). It can be used to define the sampling required for waste management and undertake sampling plan optimisation during exploration (Chilès and Delfiner, 1999). Additional guidance on appropriate sampling approaches for particulate materials has been detailed by Gy (1982 and 1998) and refined by Francois-Bongarcon (1999) and Sketchley (1999). These references provide an approach to assess, understand and cope with variability in a matrix to be sampled and ensure that the sample is representative of the target population and that representatively is maintained to the point of analysis in the laboratory. The principles outlined in these references underpin the guidance provided in EN 14899 and CEN/TR 15310-1. It may be appropriate to refer to these source documents to ensure the most appropriate choices are made in selecting the approach to sampling.

To be able to extrapolate any data from a limited number of samples to a much larger population is the ideal of any sampling exercise. It is clearly important that any sampling exercise follows the probabilistic route whenever possible (informed judgemental sampling). Even when judgemental sampling is the only option, a well-chosen sub-population and subsequent adherence to a statistical approach in terms of the sample number, size and sampling pattern will give additional information over an uninformed judgemental sampling (e.g. ‘spadeful of waste taken from not sure where’).

The use of judgemental or probabilistic sampling depends on the objective of the characterization program. For example, sample selection may target areas with visual sulfides to provide an indication of worst-case drainage quality. Similarly, judgemental sampling may be more effective in ensuring a sample set with a complete range of compositional characteristics than probabilistic sampling. Probabilistic sampling may be appropriate during operations in determining the appropriate location for waste disposal (e.g. waste segregation based on total sulfur content).

Use of judgemental sampling is illustrated in the following examples:

- samples are collected from exploration drill holes near the defined area of zone of mineralization when it is anticipated that waste rocks adjacent to this zone may present a greater risk to the environment e.g. increased sulfide content towards the mineralisation;

- samples are collected from the production front based on set mineralogical assemblage or rock phase colours (an example specifically for mineralogical and geochemical characterization);

- samples are collected based on colour differences in the drill cores of tailings; or specifically selecting only the finer material from the waste rock dump.

Variability and distribution parameters such as neutralisation potential (NP) and metal concentrations are typically more important than central tendency or average compositions. Descriptive statistics such as the 10^{th} and 90^{th} percentile and median are a useful way to describe the variability, in addition to plots showing the distribution of the data. Non-parametric statistics may be more useful than those that assume a normal distribution. Sensitivity analysis can be used to determine whether additional information is required. Spatial variability is important in determining when geochemically different materials are mined e.g. to evaluate segregation cam be beneficial.

2.8.1.5 Reliability

The objective of the programme will influence the degree of reliability that is regarded as acceptable, but the final selection of reliability criteria will nearly always need to be a compromise between cost and expectation. Given the important decisions that are likely to rest on the findings of a screening and comprehensive characterization exercise, it is suggested that the reliability should be as high as possible. Conversely, given the ‘check’ format envisaged for confirmation testing, the achievable reliability for any one assessment will in many cases be low. However, this could be offset to some extent where a large number of similar checks are available and checks are being made against a large characterization dataset.

The reliability is the consistency of a set of analyses done on a population of samples. It depends on the robustness of the analytical methods employed but mainly on sampling method used. The reliability is inversely related to random error. In sampling program or testing program reliability embraces three statistical concepts, ‘bias’, ‘precision’, and ‘confidence’:

- The bias of an estimator (statistic) is how far the average statistic lies from the parameter it is estimating. In other words, if we imagine we could repeat a survey over and over again, and use the same method for each acquired sample to create the same statistic, then we expect the different values for the statistic to be randomly distributed around the parameter we are attempting to estimate. Bias occurs if those estimates for the statistic are systematically lower or systematically higher than the parameter value.

- The precision measures of how close an estimator is expected to be to the true value of a parameter. Precision is usually expressed in terms of imprecision and related to the standard error of the estimator. Less precision is reflected by a larger standard error.

- The confidence level describes the uncertainty associated with a sampling method. Suppose we used the same sampling method to select different samples and to compute a different interval estimate for each sample. Some interval estimates would include the true population parameter and some would not. A 90 % confidence level means that we would expect 90 % of the interval estimates to include the population parameter; A 95 % confidence level means that 95 % of the intervals would include the parameter; and so on.

These different concepts can be illustrated by shoots on a target:

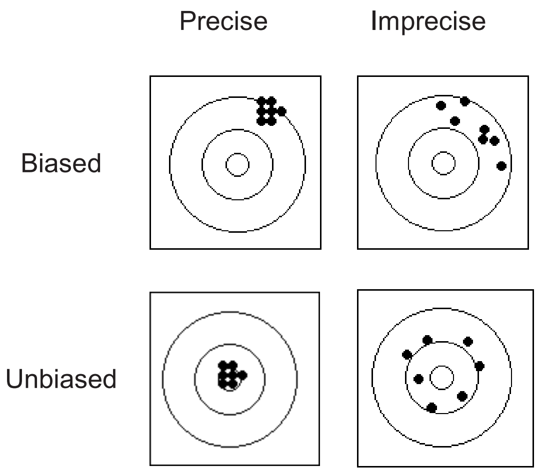


Figure 5 — Relationship between precision and biasness of analysis

Further information on the importance and consideration that should be given to the various elements of reliability is provided in CEN/TR 15310-1. Sampling and testing in the exploration phase commonly gives the poorest reliability due to the limited amount of material available. However this data provides the foundation for the design to be followed during operation and permit decisions (waste management plans, environmental impact evaluations, initial closure proposal, insurance premiums, etc.). If the results from the analysis indicate that the characteristics of the waste material have an acceptable margin of safety from relevant environmental acceptance limits, high precision may not be needed. Similarly, where risk criteria are substantially breached and special measures will be required to handle any of the waste it may not be necessary to undertake substantial sampling. Three examples are provided to illustrate this point:

— The pyrite concentrations in a limestone quarry range from non-detectable to a maximum of 0,5 wt. % and a mean value of 0,2 wt. %. No harmful trace metals are detected and the neutralisation potential is more than 10 times higher than the acid producing potential in the waste material. In this case high data precision is not necessary because the confidence that the material will not generate acid/neutral rock drainage is high.

— In a massive sulfide deposit the future tailings material is found to contain 30 wt. % pyrite and high concentrations of other sulfides. The concentration of neutralizing minerals and the neutralizing potential of the material are low. In this situation we can be confident that the material will generate acid rock drainage (ARD) with a high metal content. The precision on estimating ARD potential by ABA is therefore not critical.

— It is found that arsenopyrite concentrations in a gold deposit are critical to the permit application due to the different waste management procedures that will be required with varying arsenopyrite content. A high precision is required from the sampling and test data to achieve an acceptable level of confidence for the waste management plans.

— Sampling and testing during the operation or production stage is commonly based on confirmation tests to check that results are still as predicted from the sampling undertaken in the exploration stage and that the criteria laid out in the design plan are being met. Tailings and waste rock dumps can be sampled on the surface of the out-slopes and on the top relatively easily without a large amount of equipment depending upon their design. However, sampling in this manner may lead to a biased result which is skewed towards the later part of the waste production phase and not very reliable if intended to represent the whole dump.

The most cost effective way to characterize geological materials (future waste material and waste in dumps and dams) for large and more complex sites is to undertake an iterative sampling and testing program. This approach will aid identification, amongst others, of an appropriate scale, sampling approach, and parameters to be analysed, and ensure an appropriate number of samples are collected to meet required precision and confidence levels. It may also be necessary sometimes to accept an increased level of uncertainty depending on the outcome of the analysis.

2.8.1.6 Choosing the statistical parameter

The statistical parameter is any numerical characteristic of a population - for example, the mean[2)] or 90-percentile. The chosen parameter has a critical bearing on both the type of sampling and the number of samples needed. For a number of commonly used parameters, CEN/TR 15310-1 provides methods for estimating the parameter and calculating the associated uncertainty. The latter is a critical piece of information, because it provides the quantitative link between the number of samples and the achievable reliability (see above).

The choice of parameter will often depend on the sampling objective. For comprehensive characterization, for example, measures of variability (e.g. relative standard deviation[2)]) and extreme behaviour (e.g. 95-percentiles) are likely to be required.

2.8.2 Determine the practical instructions

2.8.2.1 General

This element of the sampling plan describes the practical field instructions on when, where and how many samples are to be collected. The practical instructions are in part dictated by the identified objectives, the selected approach and the required analytical methods. The material available for sampling will provide a practical constraint on the final sampling programme. The practical instructions represent a condensed set of actions to be followed by the sampler and are distilled from the more comprehensive sampling plan that has been developed, with justification, by working through the flow chart in Figure 3.

If judgmental sampling is adopted, it is important to describe the criteria used to select the sampling location, e.g. colour, grain size, secondary minerals. If, for example, the sampling approach is intended to be systematic, the grid system should be described. If probabilistic sampling (see below) is to be performed, there should be a description on the approach that is selected.

The practical instructions should, furthermore, describe how many samples that are needed to be collected both spatially and over time, for example how often a sample should be collected from a conveyor belt or pipeline. The instructions should include the following (as discussed in 2.9.2.5):

- discussion of the required sampling pattern;
- increment and sample size considering coverage of different material types;
- use of composite and discrete samples;
- sample numbers;
- sample frequency.

2.8.2.2 Identify the sampling pattern

The sampling pattern defines where, when and how the required samples will be selected from the population. The sampling pattern may be:

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2) If the distribution is not normal or log normal, statistics other than arithmetic mean and arithmetic standard deviation should be used (Price, 2009).

- simple probabilistic sampling – although every part of the population has an equal chance of being sampled, the resulting samples may not be very evenly spread across the population, and other more structured patterns of sampling may therefore be preferred;

- stratified probabilistic sampling – strata are identified within the population, and specified numbers of samples are spread randomly within each stratum. Benefits of this approach are that each stratum within the population is sampled adequately, whilst the advantages of random sampling are retained within each stratum;

- systematic sampling – as per stratified probabilistic sampling except that the samples are taken at the same time or location from each of the identified strata or batches. This approach could lead to bias if there is a systematic component of variation within the process that runs in step with the chosen sampling interval;

- judgemental sampling – this type of sampling can follow any type of sampling pattern or frequency. Where possible, however, it should follow as many as possible of the principles of the probabilistic approach; i.e. undertake a systematic sampling approach for an identified sub-population (chosen, for example, because access restrictions may limit sampling to the outside perimeter of a tailings dam). If it can be assumed that this sub-population is likely to be little different in the characteristics of interest from the overall population, then this method of sampling can still provide information on the entire population of interest, despite the fact that access limits sampling to a small part of the population.

Table 3 lists issues that need to be taken into account with the selection of the sampling pattern. Further information with illustrations is given in CEN/TR 15310-1 and ISO 10381-1.

Table 3 — Potential issues for selected sampling patterns

Sampling pattern	Rationale	Concerns
Probabilistic sampling	Unbiased sampling, any location can be sampled.	May lose information on variability because the material is commonly systematically tested rather than focussed on changes in geological units or zones of mineralisation. Difficult to sample tailings and waste rock areas in this manner.
Systematic sampling	Set up a grid based on known variations. Combining random sampling on a grid system is possible. Easy to set up and locate on a map.	May lose information on variability if the selected grid coincides with any systematic changes in the properties of the waste. Use of expert judgment in the field is important.
Judgmental spot sampling: rock types; alteration types; colour variations; grain size ore/wall rock; and layering	Sampling based on known variations. Can select a random approach within each area selected on judgement.	Incorrect identification of the population or areas selected on judgement may mean that samples are not collected from areas that are later shown to be important. Samples may need to be re-allocated to a different population or area, as better knowledge is gained (which is one reason why discrete sampling may be favoured over composite sampling).

Exploration of a mineral deposit is commonly based on a systematic sampling approach. Drill holes are set by a grid system to define the economically viable area of mineralization where grade and volume are key parameters. Samples of future waste rocks are then defined as the material likely to be excavated but which lie outside the area of mineralised material to be processed. Stratified random sampling can then be undertaken on this material for each ‘sub-population’ or geological unit. Smaller zones (relative to the total mineralization) with different mineralogy and chemistry or grain sizes may be ignored or not detected depending on the adopted grid system if they are systematic or where the grid spacing is too large. However, these smaller zones may in some incidences be a subsequent cause of waste management problems. To avoid this issue a mix of judgemental and probabilistic sampling may be adopted especially where there is a limit on the samples that can be collected. Drill cores or chips are commonly divided into different sections and probabilistic sampling may be collected from e.g. defined different rock types and alteration zones. This will reduce the possibility of sampling bias due to systematic variations in the material properties.

Efforts may focus on sampling of material that is close to the zone of economic mineral extraction as this transition zone may be of higher environmental concern. Judgemental spot sampling may also be used in this context to deliberately select samples that could give rise to the greatest management risk.

Sampling active tailings dams is often difficult due to their high water content. For geochemistry, new tailings material is best sampled from the discharge system rather than post deposition as this can then be sampled using a probabilistic approach. Sampling the full depth of deposited tailings can usually only take place near the embankment, if they are accessible at all, but identified sub-populations can still be sampled using a probabilistic approach.

If the tailings discharge point is moved around the dike, it may be possible to sample the surface material that has been exposed to weathering over some weeks or months. This material may be of interest if it contains sulfides. In addition to the limited area available, sampling of this surface material may be judgmental, with probabilistic sampling of identified sub-populations (e.g. geotechnical units or lenses within the profile). Adoption of a probabilistic surface sampling may be advantageous to evaluate the variability of the near dike material, commonly the coarser fraction due to the design of most discharge systems.

Older tailings dams built as a flow through (permeable dyke) are usually fully accessible and can be sampled by probabilistic, systematic, and/or judgemental sampling. Judgemental sampling is common, using visual variation on the tailings, design criteria, and discharge system for guidance together with the objectives of the sampling. It is advisable to sample tailings dams several times, using the results to redefine the required number of samples.

Probabilistic sampling is used primarily to evaluate the variability of the tailings material on the surface and with depth. Judgemental sampling is used predominately for evaluating the processes taking place within the tailings. Discrete sampling is commonly used both at the surface and at depth for variability evaluations. Discrete sampling is also used for surface sampling for process evaluation; whilst interval sampling (e.g. mix of a material from a certain core length) is sometimes used for process evaluation at depth. However, the objective of the sampling programme will, to a large degree, control the sampling approach, for example, sampling of different geotechnically important units over the profile.

Waste rock piles are harder to sample than tailings dams due to particle size variability. Larger composite samples for mineralogical/geochemical testing are more common for both variability evaluation and process evaluation. Due to the difficulty of obtaining samples at depth, most samples collected from drilled waste rocks are interval samples. For variability evaluation, random sampling from the collected intervals is common. For process and geotechnical evaluation it is more common to select all intervals from each drill hole.

Drilling tailings and waste rocks or sampling from conveyer belts and pipelines are also commonly performed on a systematic basis. However, the grid spacing, samples sizes and interval length are commonly chosen based on the variability of the material to be sampled and on the parameters to be analysed. In complex systems it is therefore commonly necessary to have several sampling rounds or selecting more samples for analysis from the material collected after the first round of analysis. This is performed in order to reduce the analytical errors induced through the sampling programme.

2.8.2.3 Analytical requirement for size of samples

Table 4 provides some guidance on preferred sample sizes.

Table 4 — Recommended sample size by measurement parameter

Measurement parameter	Application	Sample size
Hydraulic conductivity	Tailings, waste rocks, soils	Small sealed cylinder for tailings and soils; field tests for waste rocks
Moisture content	Tailings, waste rocks, soils	Small sealed cylinder; for tailings and soils; *in field* for waste rocks
Grain size	Tailings, waste rocks,	Tailings – 1 kg to 2 kg; Waste rocks – 3 kg to 5 kg or much more depending upon the grain size distribution
ABA – Static testing	Tailings, waste rocks, soils	1 kg is required in EN 15875:2011.
Mineralogy	All stages and situations	100 g to 200 g
Sequential extraction	All stages and situations, most common pre and post deposition	100 g
Leaching tests	Tailings, waste rocks, contaminated soils	2 kg to10 kg
Kinetic tests	Tailings, waste rocks	2 kg to 5 kg for laboratory tests; can be 100's of kg to several tons with on-site testing.
Field leach tests	Tailings, contaminated soils and waste rocks	weight as for kinetic tests or based on rapid in situ rainfall simulation of old waste material of commonly 1 m^2 to 2 m^2
Geotechnical properties	Tailings, contaminated soils, waste rocks	Sample size dependent on the property to be measured and also on the grain size distributions (see further details in e.g. EN 1997-2).

2.8.2.4 Determine the use of composite or individual samples

The following types of sample and sampling activity are used to describe the procedures used in the field to produce a sample for onward testing and may be used for mineralogical/ geochemical testing, but commonly not for geotechnical testing:

— A *grab sample* is an initial large, snapshot sample collected from a specified amount of waste or from a conveyor at specified intervals during operation;

— A *composite sample* is a combination of a number of grab samples, which should be thoroughly mixed. The grab or spot samples could be taken at the same sampling location, for example, a conveyor or discharge pipe over time or be from a number of different locations. When dealing with spatial variable statistics, composite sampling can often be advantageous to increase the representativeness of each sample analysed, whilst at the same time reducing the number of samples requiring analysis. A disadvantage is that information on variability over time or distance can be lost. The use of composite samples requires good field notes in which the variation within each composite sample is described.

— *Interval sample* (from a trench, or drill material) provides a sample that is representative of an entire section, this effectively evens out hotspots (i.e. you lose information on variability) but is useful to evaluate mass transport.

- *Discrete samples* (trench, drill material) are a series of single samples that can be used to evaluate the variability of the core or trench.

- *Primary sample* is a composite made up from all the grab samples of the particular waste stream collected during the specified time period (scale of sampling).

- *Sub-sample* is taken from the primary *sample* to produce the *laboratory sample* for onward testing (sample splitting).

Sampling for geotechnical analyses are described in ISO 10381-2 and EN ISO 22475-1 and testing procedures are presented in A.2 of the overall guidance document (CEN/TR 16376:2012).

For sampling of tailings and waste rocks from drill material, samples can be collected as discrete samples or as interval samples. By taking discrete samples the variability and potential hotspots are more likely to be exposed if sufficient samples are collected. However, by collecting interval samples it is possible to evaluate transport of metals within waste dumps. The purpose will therefore dictate which approach to take.

The use of composite samples prepared from material from different locations will improve the prediction of the overall central tendency, but may mask significant variability of material parameters like, ABA, mineralogy, grain size, chemistry. If drainage chemistry needs to be predicted smaller "*hot spots*" can create the largest problems and it is therefore important to avoid compositing (Price, 2009):

- at different times;

- over wide distances; and

- from different geological units or waste dumps.

The use of composite samples is also controlled by the same factors as described for the amount of samples:

- stage of the project;

- complexity of the geology;

- questions to be answered (objectives);

- degrees of uncertainty required or accepted;

- variability of the parameters to be analyzed.

Compositing should be avoided for materials that have a significantly different geologic composition, structure and weathering and leaching conditions. Where differences in potentially important properties occur irregularly or along continuums, samples should be taken from geochemically similar units (Price, 2009).

When dealing with random variable statistics, compositing saves cost. When dealing with spatial variable statistics, compositing ensures representitivity and is often a relatively cheap way to reduce error if field costs are high (Price, 2009).

Where there are concerns about the use of composite samples a split of each sample that comprises the composite should be stored, allowing for analysis later on in the project. The use of composite samples can therefore, be a useful tool in an early stage of the project while, used to less extent in the design and permitting process.

2.9 Identify the most appropriate sampling technique (EN 14899:2005, 4.2.8, and CEN/TR 15310-2)

The second technical report supporting the framework standard EN 14899 (CEN/TR 15310-2) provides guidance on the choice of sampling techniques. Furthermore, ISO 10381-2 gives information on appropriate sampling techniques and equipment for different soil sampling situations and EN ISO 22475-1 on sampling methods particularly for geotechnical testing. This report provides additional information that is relevant to sampling geological rock formations and extremely large stockpiles of waste rock or tailing dams.

A generic look-up table illustrating suggested applications for sampling equipment is provided in Table 5. Sampling techniques are partly determined by the objectives of testing, partly by the characteristics of the deposit or waste and parameters to be tested and partly by the approach selected for sampling. Additionally, field measurements provide valuable information and indication of presence of specific properties. This information can be used in judgemental sampling for selecting samples for laboratory testing. Examples of field measurements are pH, portable X-ray fluorescence analyzers (XRF) and near infrared spectrometer. Field measurements may also be valuable by itself and increases the amount of information for characterization of the waste.

Table 5 — List of example sampling techniques that may be used in the different settings and phases

Sampling scenarios	Method of sampling	
Exploration		
Economic material and waste definition (early exploration phase)	Drilling Trenching Surface samples (rocks and sediments) Water sampling to locate ore deposit	
Waste site location (specify e.g. by example)	Drilling Trenching Surface sample (rocks and sediments) Hand auger Machine-driven auger Continuous flight auger Split-spoon / SPT Sampler Shelby Tube Sampler (for undisturbed samples) Water sampling	
Production		
Production Front	Drilling material (from blast holes, etc.) Handpicking Shovel	
Conveyer belts	Shovel Advanced systematic sampling selection system	
Pipelines	Bucket Advanced systematic sampling selection system	
Dump trucks	Shovel	
Closure and closed sites		
	Surface sampling	**Depth sampling**
Tailings dams	Spoon Shovel Hand auger Machine-driven auger Continuous flight auger	Trenching Backhoe Hand-auger Machine-driven auger Drilling Split-spoon / SPT Sampler Shelby tube sampler (for undisturbed samples)
Waste rocks piles	Shovel Hand picking	Backhoe Drilling Trenching
For many geotechnical analyses special equipment is necessary or field tests (not included on the list) are performed instead. Tailings dams and waste rock dumps may also be sampled during production.		

The selection of an appropriate sampling technique will depend on a combination of: the different characteristics of the material; and circumstances encountered at the sampling location. These determining factors are:

— type of material / the physical state of the material (e.g. monolithic, granular, liquid, paste or sludge);

— sampling scenario / the way in which the material occurs (e.g. as a geological formation beneath the ground, on a conveyer belt, in a stockpile or in a lagoon);

— degree (expected) of heterogeneity (e.g. homogeneous particulates, segregated tailings sludge, mixtures of solid materials);

— sampling objective, which may influence the approach to the selection of composite or individual samples as detailed in 2.6.

An undisturbed sample is needed to test hydraulic conductivity, consolidation, shear stress, etc. in the laboratory. The undisturbed sample is commonly obtained by pushing a smaller metal cylinder into the material to be collected (for tailings). This type of cylinder is often combined with a split spoon. The result of hydraulic conductivity testing will be biased if the material is disturbed. However other methods may be required to obtain an undisturbed sample for investigation of layering effects within a tailings deposit resulting in anisotropic permeabilities (e.g. cutting out of block samples and recording orientation).

Drilling tailings dams and waste rock dumps may result in a conduit for air and water. It is therefore necessary to follow correct drilling procedures, sealing holes from cross-contamination and possible increased oxidation of sulfides. Proper closure of the hole may include filling the hole partly with gravel and bentonite layers or if water monitoring is to be performed this should take place within wellpipe using standard procedures for installing groundwater wells (ASTM D5092 - 04(2010)e1).

It is important to clean (and decontaminate) equipment between samples. The use of water can be problematic where this needs to be carried in to the field. An alternative is to clean the equipment using material located adjacent to the next intended sample and wiping it clean with a towel. It is important to collect some additional QA samples to specifically evaluate cross contamination issues. If it is not possible to clean the sampling equipment between samples, this should be documented in the sampling record.

The sampling approach for different sampling scenarios is commonly very different for the different phases of a mine operation. A few scenarios are listed below:

a) Exploration phase (geochemical analysis only):

   1) Drilling and extracting material either from drill cores or chips depending upon the type of drilling used during exploration. Drill material is commonly logged by a geologist based on the needed information for the type of mineral deposit and stored as interval samples if drill-chips or in core boxes. Samples for environmental evaluation/waste characterization are commonly available from stored leftover material in drill core boxes, chip boxes or sample bags.

   2) Trenches in the exploration area are also common depending on the type of mineralization. Samples may be collected from these trenches for waste.

   3) Surface sampling in the exploration area may also be performed to characterize the future potential waste material depending on the mineralization. If the operation will become an open pit, the surface material may represent the overburden. If the deposit outcrops it may represent the mineralization and material may be test processed to obtain a representation of future tailings material.

b) Operation phase (for geochemical analysis and some geotechnical tests where appropriate):

   1) Conveyer belts are used in many operations to remove waste rock, either as part of waste transportation or all the way from excavation to final deposit. Sampling from conveyer belts are relatively easily performed and allow a statistical approach to sampling.

   2) Pipelines or channels are commonly used to transport tailings from the processing plant to the tailings dam. A sampling system can easily be set up to systematically collect samples from these transport systems.

   3) Dump trucks are commonly used in large operations, especially with high percentage of waste rock production. Systematic collection of samples from dump trucks can be very cumbersome and lead to operational difficulties. Dump trucks also tend to be used where the waste rock material is very

coarse (boulders) and conveyer belts are difficult to use. Sampling from dump trucks is, therefore, not recommended.

4) Production front is the area where new material is excavated or waste rock and/or overburden is removed. In operations where drilling and blasting is necessary it is relatively easy to set up a sampling system from drill cuttings/drill mud from the blast hole drilling.

5) Waste rock dumps have commonly large grain size variability**.** Particles should ideally be collected from all particles sizes with a weighted percentage of the different sizes. However, in many cases most of the reactions are taking place on the outside of the particles and, therefore, small particles have per weight, a larger effect on drainage water quality than larger particles. It is also not practical to collect samples with particle sizes above 10 cm. Price (2009) therefore recommends that the sampling strategy for waste rock sampled near surface should be to separate the material in three grain size fractions with diameters: > 12 mm; 2 mm to 12 mm; and < 2 mm. Furthermore, Price (2009) recommends analyzing all three fractions to evaluate if there are mineralogical/geochemical differences between the fractions, and then mainly focus the analysis on the fine fraction. This does depend on the material characterized and the objective(s) of the testing program.

6) Tailings dams that are in operation are commonly relatively wet making it difficult to access the inner and deeper parts, both with surface samples and with drilling equipment. In these cases sampling should be focused in/on the outer parts.

c) Closure phase and closed sites (for geochemical analysis and geotechnical analysis):

1) Waste rock dumps are commonly relatively dry consisting of coarse material. Depending on the objectives of the sampling, it may be necessary to collect large samples to be crushed and split in order to get representative samples. In some cases, depending on the objectives and the background information, it may be advantageous to collect only the finer material from the waste rock deposits. Use of a backhoe or drilling is also advantageous to obtain deeper samples and possibly place monitoring equipment in the waste dump.

2) Closed tailings dams are often more accessible than operating dams due to draining of the material and possible hardening of the surface (depending upon dam design, climate, and mineralogy) and can be accessible with heavier equipment. This increases the possibility to sample anywhere in or on the tailings dam and with the use of auger drilling it is possible to place monitoring equipment in addition to extracting solid samples. Operating tailings are on the other hand commonly not very accessible except near the embankment.

3) Leach piles (heaps) become waste rock piles when the valuable metals have been leached out (to below economic concentrations). Leaching is performed on coarser material, commonly on crushed material. The same approach as for waste rock dumps applies, however, there may be hazardous leaching agents left in the leach pile and a larger amount of secondary minerals that may need a different handling and preservation than regular waste rock piles.

4) Leachate, seepage, runoff can often be collected from the toe of tailings and waste rock piles if the underlying bedrocks and sediments are not too permeable. Collection of water samples can be valuable in evaluating how to best close a waste deposit.

Further information and worked examples using the decision making process are provided in Annex B, in the following subclauses, and in EN 14899, CEN/TR 15310-1 and CEN/TR 15310-4. It is necessary, as part of selecting a sampling approach, to develop the technical goal from the objective and define the practical instructions. These are discussed in the following two subclauses.

Operation phase: If the sampling and testing undertaken during exploration was sufficiently comprehensive, the intensity of sampling during the operational phase of the mine can commonly be reduced. Probabilistic sampling should be undertaken wherever possible i.e. from a moving conveyor (whole width sampling) or discharge pipe or from the production front using drill mud from blast hole drilling to provide data about the whole population of waste being produced.

Framework Standard EN 14899 and more specific CEN/TR 15310-2 also suggest sampling during the loading or unloading of trucks used to transport the waste. However, this approach is not recommended for wastes from the extractive industry where the size of the truck effectively leads to exclusion of some very large particle sizes.

As described earlier, sampling of active tailings dams can be difficult due to the large amounts of water used in transporting the tailings material to the dam. One possibility is to sample tailings from the pipeline leading to the dams or the outlet from the flotation plant /concentrating plant (this usually requires special arrangements (a sampling valve). There may be some opportunity to collect samples near to the edge of the dam or embankment using a hand auger, split spoon auger, or from the excavation of pits using a shovel. Running larger drill rigs onto the tailings is generally not recommended due to the risks involved.

Active waste rock dumps can be sampled using a backhoe or track-hoe when the sampling plan requires representative composite samples from the near surface. Drilling into waste rock can be performed using air flush reverse circulation drilling. This technique results in a crushed, mixed, interval sample. The use of water or bentonite should be avoided to limit alteration in the physico-chemical properties of the material to be sampled.

Closure phase and closed sites: Surface or near surface sampling on closed tailings can be performed using a shovel, backhoe or hand auger. To obtain samples at greater depths, drilling will be required. The preferred technique is drilling with a hollow stem auger with split spoon or roto-sonic drilling. These techniques also allow for the installation of single stem boreholes or nested piezometers and probes for long term monitoring.

Deposition of tailings may result in several types of particle segregation. First of all, there is a lateral segregation where larger grains deposit close to the discharge point and smaller clay size fractions the furthest away. In addition, segregation can also take place based on the specific weight of the material, where sulfide minerals will deposit closer to the discharge point than lighter minerals. This horizontal segregation results in a layering of the tailings, and commonly due to variable discharge pressure and moving of discharge point, resulting in laminated. Depending upon the sampling objective these variations of mineral distribution and grain size distribution needs to be recognised when selecting the sampling techniques and deciding the sampling locations

Larger waste rock dumps may be very difficult to sample using simple equipment due to the size of the material and the segregation that takes place during dumping. A waste rock dump whether it is filled using conveyer belt or a dump truck will have segregation of grain sizes from the top to the bottom of the out slope as it is being constructed. There will, therefore, be a layering of the waste dump with an angle equal to the angle of repose. If there is mineral segregation based on the grain size variation it will therefore, be difficult to get a representative sample.

Overburden samples are usually collected by air rotary (auger drilling is not recommended by GARD, 2009). Samples collected using air rotary will generate rock chips, problems associated with this approach include:

— contamination of material during the transport to the surface;

— incorrect logging due to uncertainties about the depth of origin of the chips;

— the potential for segregation of the material coming to the surface during the drilling.

3 Sample handling in the field

3.1 General

The following clause, discusses the issues on splitting, conservation, packaging, storage, and transportation of any samples to be collected. Sample labelling should be performed immediately upon sampling before any further handling. The labelling should include project identification and a sample number (most common). All samples should be labelled using a permanent marker or a label (sometimes provided by the laboratory to be used) fixed to the sample packaging. It is also often useful to include a label inside the packaging if the packaging allows for this. If the sample is being split, the split sample packaging should have similar labelling as the original sample with an additional sub-number (CEN/TR 15310-4).

3.2 Sub-sampling

In sample preparation and sub-sampling prior to analysis, care should also be taken to collect samples that are large enough to limit “nugget effects” due to the non-uniform distribution of minerals in clusters (Price, 2009).

Sub-sampling/splitting of a field sample is a common practice in the extractive industry, due to the often-large amount of material being handled to get a representative sample. In addition it is often necessary to produce a number of sub-samples for each of the laboratories involved in analysis. Sub-sampling is easily performed on dry material of <10 mm particle size using splitters.

The use of splitters reduces the need for homogenizing the sample before production of sub-samples for the different analytical procedures. Larger and coarser samples can be split on the ground by dividing the primary sample pile into different slices, as with a pie. If crushers are available, it may be beneficial to first perform crushing before splitting unless the coarse/original material is needed for some of the testing.

During the advanced exploration phase, it is not uncommon for larger companies to have a small field laboratory that can perform some of the simpler preparation work. Smaller companies commonly have to rely more on off-site laboratories or the sampler to perform this preparation work. Exposure to air should be avoided for samples collected for microbial or red/ox reaction analysis.

Core drilling or reverse circulation drilling is common for exploration projects. The cores are often split in two, and one half is crushed and milled for chemical and mineralogical analysis. The remainder is commonly stored for later analysis. It is common for pulp or rejects from the laboratories to be returned to the operator/exploration company. This material is commonly fine powder and can be used for chemical analysis for waste characterization. In this instance sample splitting to produce sub-samples for waste testing may be a simple process.

If drilling methods are used for exploration, the samples collected are commonly interval samples stored in larger bags. The exploration company would split off material from these bags for their mineralization evaluations. There is commonly a reasonable amount of material available that will be defined as waste rock, whilst there may be relatively smaller amounts left for waste characterization of material close to or within zone that has the economic value for the operator.

The size of the sub-samples produced post splitting may be less than the minimum sample size required by the laboratory, although the degree of homogeneity may mean that a smaller sample size is acceptable.

Sampling during the operational stage commonly results in large samples that need to be split into smaller sizes following the splitting techniques described above. Samples from pipe lines and conveyer belts can easily be large and sub-sampling is recommended before submission to the laboratories. When required many laboratories can undertake this preparation work.

Special attention is needed for multi-phase samples, e.g. tailings samples contain a water phase. A general recommendation is that the water phase is not separated from water-rich samples. The samples should be sent to the test laboratory intact.

Tailings and waste rock samples from drilling, whether hand auger or large auger, commonly gives a smaller amount of samples and splitting will take place only to save a portion of the sample and where several analytical techniques are required. Surface sampling of tailings and waste rocks on the other hand usually presents no limit of the availability of the material. For waste rocks it may be a challenge to collect a representative sample due to large grain sizes that may have to be split before collecting the sample.

Further general information on sub-sampling is provided in CEN/TR 15310-3.

3.3 Sample preparation and storage (CEN/TR 15310-4)

3.3.1 General

Requirements for sample packaging, preparation and storage will depend upon the sample type and the analyses to be performed. The packaging and storage should be such that it maintains the field (natural) conditions as far as possible. The following paragraphs describe some of the packaging and storage measures that may be relevant to waste from the extractive industry.

3.3.2 Packaging

A wide range of sample storage systems can be used for sampling during exploration and production. These storage systems should be sized appropriately to meet the objectives of the sampling programme and grain size of the material and may include:

- paper bags;
- plastic bags;
- sealed containers;
- drums;
- plexiglas or metal columns.

Paper bags in particular are a simple and cheap means of collecting soils and sediment during exploration. They have the advantage that they allow the samples to dry easily. They are resistant to water, but an alternative should be sought if the samples are radioactive or contain low pH material to avoid decomposition of the bag.

Plastic bags are the most commonly used material. However, the use of sealed plastic bags should be avoided unless the sample is first dried as this can lead to conditions that promote or increases sulfide oxidation. Sealed containers should be used where an airtight environment is required, for example for bacteriological analysis, analysis of hydraulic parameters, and analysis of nitrogen compounds to evaluate explosive residues. During the operational stage of the mine plastic and metal drums are commonly used for collection of larger samples (Table 6).

Collection of undisturbed column samples and sampling directly into columns may be preferred if laboratory column experiments are to be performed. Collecting directly into columns preserves some of the structure of the material and may improve the understanding of flow and mineral reaction rate processes. Examples where this type of sampling is preferred are:

- undisturbed samples for column leach testing;
- undisturbed samples for hydraulic analysis.

Such practices will be insufficient for accurate measurement of physical parameters requiring undisturbed samples. In these cases, a Shelby tube sampler or similar should be used and precautions taken to minimise vibration during transportation, drying out of wax seals, rust during storage and tensile breaks in the sample during extrusion from the tube.

Table 6 — List of parameters relative to container types and storage conditions

Measurement parameter	Containers	Storage conditions
Porosity (if possible/appropriate)*	Sealed containers (e.g., Shelby tube)	Preferably 4°C ± 2°C
Void ratio (if possible/appropriate)*	Sealed containers (e.g., Shelby tube)	Preferably 4°C ± 2°C
Hydraulic conductivity (if possible/appropriate)*	Sealed containers (e.g., Shelby tube)	Preferably 4°C ± 2°C
Moisture content	Sealed containers (e.g., Shelby tube)	Preferably4°C ± 2°C, frozen
Grain size, specific weight; Atterberg limits	Fabric or paper bags Plastic bags**	No specification
ABA - Static testing	Fabric or paper bags Plastic bags** Sealed container	Dry, 4°C ± 2°C
Mineralogy	Fabric or paper bags Plastic bags** Plastic bags** Sealed container	Dry, dark
Sequential extraction	Fabric or paper bags Plastic bags**	Dry, dark
Leaching tests	Fabric or paper bags Plastic bags** Sealed container	Dry, dark
Kinetic tests	Fabric or paper bags Plastic bags** Sealed containers (undisturbed)	Dry, dark
Shear strength (if possible/appropriate)*	Sealed containers (e.g., Shelby tube)	No specification
Compressibility (if possible/appropriate)*	Sealed containers (e.g., Shelby tube)	Preferably 4°C ± 2 °C
Bacteriological analysis	Sealed plastic containers	4°C ± 2 °C, dark
Ecotoxicity tests	Polyethene, polypropene, polytetrafluorene or glass containers	transport time less than 48 hours and/or 4°C ± 2°C

*It may prove more worthwhile to determine these parameters by field-testing if possible.

**For dry samples.

3.3.3 Preparation and storage

During sampling it is important to have appropriate knowledge of sample requirements and the critical factors influencing the waste properties of interest in order to take measures to minimise changes in samples. Changes in sample properties may occur due to various environmental factors, e.g. oxidation, carbonation or due to sampling approach (e.g. loss of sample integrity in taking the sample). Especially for biological testing and evaluation of the acid generation potential it is crucial that samples are not deteriorated due to use of inappropriate sampling methods, improper sampling handling and storage conditions. Sampling for geotechnical properties may set special requirements on the sampling approach and sampling equipment (e.g. need for undisturbed samples), which may in some cases drive a decision to determine them indirectly by a different test method or by field testing instead.

The majority of samples from the extractive industry do not require extensive material preparation or preservation to protect the integrity of the samples prior to analysis. The most commonly used preservation technique is that of sample drying to minimise mineralogical changes in the sample such as sulfide oxidation.

In the case of secondary minerals containing water or hydroxyl ions, drying should only be performed at relatively low temperatures, normally < 30 °C (CEN/TR 15310-3).

Where ecotoxicity test is required on a sample then EN 14735 should be followed.

Where bacteriological analysis is required on a sample, the procedures undertaken are specific to the type of analysis and the guidance should be followed in ISO 10381-6.

Waste from the extractive industry is as a general rule unaffected by long term storage if the material is in a dry environment. However, sulfide oxidation may take place in environments where the humidity is as low as 20 % (Borek, 1994), although the oxidation becomes slower at these low humidity levels.

Long term storage of samples containing hydrated minerals should be avoided to minimise losses of water of crystallisation and formation of dehydrated minerals. Waste rock samples should be analysed within two weeks, or if necessary, stored in a dry, 4 °C ± 2 °C.

3.4 Transport the sample to the laboratory (EN 14899:2005, 5.2, and CEN/TR 15310-4)

The basic procedures required to transport waste samples from the extractive industry to the laboratory requires no additional procedures over those outlined in CEN/TR 15310-4 and in EN 14899.

A chain of custody form should always accompany the samples.

4 Documentation (long and short form of sampling plan)

Proper documentation of the sampling activity is very important. It is also important that the sampler is informed about the documentation procedures used for the specific project.

4.1 Document the sampling plan and produce instructions for the sampler (EN 14899:2005, Clause 6)

Examples of the documentation needed for the sampling plan are given in Annex B. This clause specifies that the project manager shall document the sampling plan.

During consideration of all of the issues required to develop a sampling plan the user will produce a plan that contains all the data and decisions made on the required approach to sampling. Once this is finalised it is necessary to produce a 'short-form' sampling plan that provides the basic instructions to the sampler that effectively implement the requirements of the comprehensive sampling plan. Examples of both a comprehensive and short form sampling plan are provided in the worked examples in Annex B. Any changes to the final sampling plan in the field shall be documented in the sample record, following agreement of the change with the project manager.

4.2 Produce a field sampling record (EN 14899)

Prior to the field sampling a sampling record template should be produced to record all the information of relevance to the site and sampling activities. A basic list of information is specified in 6.2 in EN 14899:2005 but has been supplemented for recording of information of activities at mining sites and should allow for recording of:

a) all procedures and observations during the sampling;

b) all variations from the intended sampling plan;

c) unique sampling number;

d) date and time of sampling;

e) place and point of sampling;

f) persons present;

g) difficulties of access, including information on those areas or volumes of the material that are sampled or not sampled;

h) condition of the material:

   1) colour;

   2) consistency/ homogeneity/ grain size;

   3) observations during sampling (odour, gassing out, development of heat);

   4) mineralogy;

   5) sulfide oxidation products;

   6) seeps/ seep colour/odour, etc.;

i) details of onsite determination (pH, Eh, conductivity, temperature, etc.);

j) identify sampling amount (estimated volume and mass);

k) sub-sampling methodology;

l) name of sampling personnel;

m) place, date and signature.

The sampling plan shall specify that any measurements taken in the field shall be recorded in the field data form and appended to the sample record.

The sampling record should be completed after collection of each sample to provide an accurate record of activities undertaken on site and observations that relate to the sampling location and its surrounding.

If available, it is useful to document the location of all samples in the geological block model for the operation. This results in a better understanding of the waste variability and the waste management especially when combined with the geological data.

An example of a sampling record is found in Annex B.

5 Sampling (EN 14899:2005, 5.1)

Before sampling begins, all items of the sampling plan should be checked with the sampler, project manager and the operator. Adjustments of the sampling plan may be necessary due to field conditions or placement of the waste that were not foreseen during the development of the sampling plan. Sampling should only be performed by qualified personnel.

If changes affecting the objective of the sampling plan are required, it is recommended to request a written authorisation of this change prior to proceeding with the sampling. If the changes do not affect the objective(s) of the sampling plan, recording the changes and reasons for the changes in the sample record form is sufficient.

6 Sampling uncertainty and other issues

Uncertainty is commonly a much larger problem for the sampling aspects of a programme than for the laboratory/analytical testing. Relatively small samples are collected when compared to the whole population or sub-population and rocks are commonly not homogeneous. This represents a challenge, and for that reason comprehensive, but flexible sampling plans should be developed. Some of the issues regarding uncertainty are described throughout this guidance document, but specific reference is required on a number of issues that are highlighted in this clause.

NOTE These are also described within the overall guidance document (CEN/TR 16376).

To evaluate the sample variations within waste dumps it is common to collect duplicate samples adjacent to each other. The analytical performance of the laboratory can be checked by splitting a sample into two sub-samples and deliver them to the laboratory as two separate samples.

Appropriate sampling for the specific tests required for characterization is important. Particular care is required for collection of samples for kinetic or geotechnical testing, to ensure sample viability. The number of samples that require kinetic testing is commonly much lower than the number submitted for other chemical/mineralogical tests due to the long duration time and high cost of the kinetic tests. It is of utmost importance that the objectives of the kinetic and/or geotechnical testing are identified to be able to define an appropriate sampling methodology.

If there is a large variability, it may be beneficial to collect samples from the expected *worst case* scenario within the population, e.g. picking out sample areas with high sulfide content.

The sample size for column tests is commonly 2 kg to 5 kg. If material is taken from core material, this is routinely all that would be available and would usually be a composite sample. However, it may often be useful to run larger kinetic tests in drums that may take 100 kg to 200 kg. This will result in a more representative sample and would not necessarily increase the costs. Typically this is commonly done in the last phase of exploration where bulk material is being excavated, or as a part of the operation where it is necessary to perform confirmation analysis of smaller columns run during the exploration phase.

If the samples are not collected and used immediately for the column tests, they should be dried at maximum 30 °C. This results in the halting of oxidation and avoids destroying secondary hydrated minerals.

Statistical analysis of test results is advisable to confirm that a representative data set has been obtained. For example, histograms may be used to ensure that the entire distribution has been captured in sample selection (Runnells et al, 1997) and samples with “extreme” characteristics have not been overlooked. The number of samples will increase as the heterogeneity (e.g. particle size and composition) of a material type increases. For this reason, characterization of process tailings typically requires fewer samples than characterization of waste rock. Sample representativeness shall continually be assessed during the mine life. For example, a change in ore type over the mine life may produce process tailings with different characteristics. Operational monitoring should include a program of systematic on-going tailings testing to identify changes and implement alternative waste management practices, if required.

Annex A
(informative)

Example sampling plans for waste characterization for exploration, operation and closure stages of extractive industries

A.1 General

A number of sampling scenarios have been developed for a range of mining wastes to illustrate the range of approaches that may be required for the commonly used phases of sampling: screening and detailed characterization; and compliance testing. The examples are summarised in Figure A.1.

The detailed sampling plan is intended to lay out all the issues pertaining to a specific site that shall be taken into account in the design of the final sampling plan. This approach will ensure that the sampling plan meets all the requirements of the intended sampling and testing programme and provides clear instructions to the identified organisation or individuals undertaking sampling. It effectively forms a record of the reasons why a given approach is selected and the decision making process. The worked examples should not be used as a standardised approach and have been developed to illustrate key issues and assist the reader in developing a sampling plan to meet their specific requirements and site conditions.

— Example A.2. Exploration stage. Large open pit mine containing sulfides;

— Example A.4. Operation stage. Simple system aggregate quarry.

A.2 Example detailed sampling plan for exploration stage

A.2.1 Large open pit mine containing sulfides

The most extensive waste characterization will typically be performed during the late phase of the exploration stage. There is commonly only relatively small amount of material available during this stage. This makes it challenging to predict the environmental effects from future waste disposal with any degree of confidence. Waste management plans and closure plans shall also be developed based on the results of the waste characterization testing; these will have to take into account the confidence level achieved in the waste characterization testing.

The following sampling plan is developed as an example of how the sampling plan could look like for a potential large scale open pit mine containing sulfide minerals.

A.2.2 Background

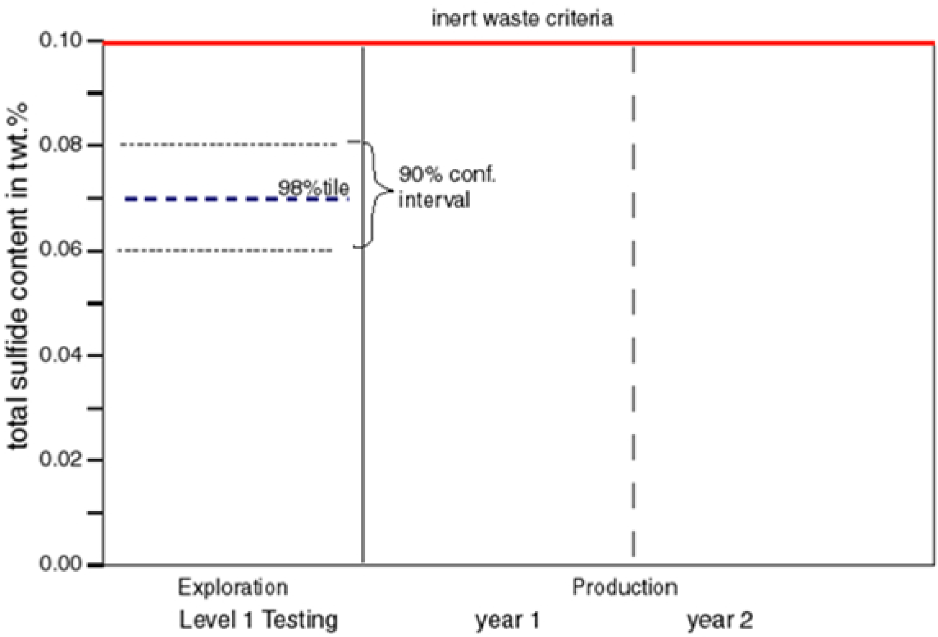
The exploration project is adjacent to an existing operational porphyry copper mine. There is therefore a lot of background information on mineralogy that can be utilised to develop the sampling plans. However, even with this knowledge, it is likely that several rounds of sampling will be needed to supplement this information in the permitting/exploration stage, with follow up sampling during operation, if a permit is granted. The host rocks are granite gneiss which commonly exhibits a low neutralizing potential. The total reserves are 200 million tonnes and it is estimated that operation will produce approximately 400 million tonnes of waste rock. Exploration drilling has been performed during the last two years with 10 drill holes available for ore and waste definition. The project is in the feasibility/permitting stage.

	Step	Outcome
Defining the key elements of a sampling plan.		
1	**Identify involved parties** 2.2	Mine owner, Permitting authorities Independent consultant Stakeholders
2	**Identify the general objectives** 2.3	Overall sampling objective: To characterize the future waste rocks and tailings from a porphyry copper deposit containing sulfide minerals for a production of 35 million tonnes of ore per year to provide information for the proposed mine permit application. The characterization exercise is required to: — guide decisions on deposition methods and appropriate techniques, and — evaluate closure options.
3	**Background information and field inspection** 2.4	This example is has been developed based on a large scale open pit mine that is currently operating a tailings and waste rock facility. The mine is also performing exploration as a part of on-going activities together with new and updated mine permits. The mine has through exploration identified increased reserves and wishes to apply for increased production. There is already a large amount of information about the waste material from earlier investigation and from compliance testing of the waste material from the on-going operation. This sampling plan has been developed to facilitate the design of a management plan of the future waste from the exploration stage of the operation. There is a lot of information about the waste produced during current operation and it is intended that this is used to support the current sampling and testing programme. The mine has an existing waste rock management plan, but it is unknown if this will be appropriate for the new areas of extraction as this has a different ore type and will require a different processing system.
4	**Identify detail objectives** 2.5	The following specific objectives are defined for the project: 1) undertake testing to define the intrinsic geochemical properties of the waste and define the acid generation and/or leaching potential of each potential waste stream to ensure a disposal facility is appropriately designed; 2) evaluate if there is inert waste within the waste rock units; 3) develop waste management plans; 4) prepare a closure design for the waste facility for two types of waste rock and tailings; 5) evaluate/calculate the reduced leaching-A/NRD potential based on the closure design suggested.

5	**Determine generic level** **2.6**	The material to be characterized is the future waste to be generated during an exploration project. Comprehensive characterization testing is required to supplement background information from earlier studies and the results from a previous screening programme. If the new areas have different chemistry/mineralogy, etc. than the background data indicates, the sampling number should be intensified to obtain enough data for the specific objectives.
6	**Identify tests and constituents to be tested** **2.7**	The different tests suggested for the different objectives are listed below. In this exploration stage of the porphyry copper deposit, the same tests should be performed on both tailings and waste rocks material. However, due to the limited amount of material for testing material representing future tailings, the number of samples will be different as discussed under point 19. Commonly only a few samples but larger samples are sent for testing process method. **Comprehensive characterization/waste disposal category** The following tests should be performed for this specific exploration project containing sulfidic waste as a minimum for the comprehensive waste characterization. 1) EN 15875 will be the main method used, however analysis using modified Sobek will also be performed to verify similarities in these two methods since Modified Sobek was used before. NAG test, will also be used for evaluating this method as a run of the mine method for later quick testing. 2) Total sulfur and sulfide sulfur testing. Both methods are advised since there are sulfate minerals identified in the material. 3) Leach tests (test method to be chosen based on total metal content, and the A/NRD potential). 4) Metal concentration of elements using 4 acid dissolution and ICP-MS, for a suit of elements likely in this type of deposit, to evaluate the potential of metal leaching and used to evaluate how to set up possible kinetic testing. 5) Mineralogy analysis by microscopy to support the information and evaluation of data from the other tests specified above. NOTE A similar list should be developed for all of the identified objectives (for the purpose of this example a list is limited to only one of the objectives. **Inert waste evaluation** Input from the comprehensive characterization. If other tests are needed, these should be specified here. **Acid generation and leaching potential** Input from the ABA and the other tests from the comprehensive characterization. **Develop waste management plans** The waste management plans should be developed based on the result from the comprehensive characterization and the inert waste evaluation. **Design closure plans** In order to design the closure plans, the area of disposal needs information of the characterized soil types, bedrock groundwater, water shed, etc. Potential additional tests needed should be specified. **Evaluate/calculate leaching-A/NRD potential after closure**

		The data used for evaluating the leaching rate and A/NRD potential after closure will primarily depend upon the data from the comprehensive characterization and the acid generation and leaching potential data combined with the closure design. Potential additional tests needed these should be specified.
7	**Health and safety precautions** **2.8**	The mine has developed health and safety policy and procedures for onsite activities. The sampler is to comply with this policy and will go through a one hour training to be informed about this policy and what to do in an emergency situation.
Select/develop the sampling approach, 2.9.		
8	**Identify the technical goals** **2.9.2**	There are five main objectives that are addressed by this sampling plan and the technical goals of these objectives overlap. All material for the characterization programme will come from exploration drilling (core drilling and reverse circulation drilling). Samples are being logged for mineralogy, colour (mineral indicator and oxidation stage of sulfide minerals), and fracture density. Special attention should be paid to sulfide content, pyrite and pyrrhotite content and carbonate mineral content. Sampling is restricted to the material available from the drill holes. The drill holes give an approximately even distribution of the waste rocks and the ore. The result of the first testing round will inform whether more drill holes are required. The comprehensive waste characterization exercise should be based on an iterative approach, where a smaller amount of samples are selected in the first round and depending on the results of this sampling there may be a need to enter into a second round of sampling and testing. Samples of waste rock should represent material near to and distant from the ore deposit to define the variability in the waste material with increasing distance from the mineralization zone and evaluation of the tonnages of potentially different waste types. Samples can be taken as this material is processed to represent future tailings material. In the first instance the waste material are divided into the following categories: 1) Waste rock with low sulfide, copper content and high neutralisation potential; 2) Regular waste rock; 3) Low sulfide tailings; and 4) Concentrate pyrite tailings. The sampling will be undertaken on logged drilling material, Therefore, before selection of samples it will be necessary to evaluate the borehole logs to ensure samples are taken from an appropriate range of future waste rock. Material that is likely to be representative of future tailings will come from discharge material from the mineral processing tests being undertaken by the mine. The goal of initial waste characterization sampling and inert waste evaluation is to produce sufficient data to undertake preliminary development of the waste management plan and closure plan. The results of these evaluations will inform the design of the kinetic tests which may be needed if it is determined that further evaluation of the A/NRD potential and leaching potential of the waste rock is required.
9	**Identify the overall**	The overall population is approximately 250 million tonnes of waste rock that will be produced over an 8 year production period, together with approximately 150

	population **2.9.2.2**	million tonnes of tailings. Approximately 3 wt. % of the mined material will be removed from the mine site as construction material. It is intended to attempt to sell some of the remaining waste material for road construction etc. to reduce the overall amount of waste to be deposited. In the initial first round of sampling the waste rock sub-population will be chosen based on the following field observation (this is based on experience from the operating mine at the same location): 1) < 0,5 % sulfide content; 2) > 2 % calcite content; 3) High biotite and actinolite content.
10	**Choose the sampling population** **2.9.2.2**	Samples are available from the whole population based on the exploration drill holes. If needed, and based on the results obtained in the first round of sampling, new drill holes will be set to fill in information gaps or further confirm the initial results. As previously stated samples are only available from the drill-hole material not the whole population.
11	**Assess variability** **2.9.2.3**	Temporal variation could be significant during operation. The variability in the ore deposit is likely to lead to changes in the waste rock composition. However, although initial information can be gathered from the exploration characterization sampling and testing, this will need to be assessed further during the operational stage of the mine and appropriate sampling plans developed for compliance testing. The waste management plans will be developed with the potential variation in mind.
12	**Identify the scale** **2.9.2.4**	Each sample for the waste rock characterization will represent 40 million tonnes. This is based on the first evaluation assuming two relatively homogeneous sub populations of the waste rock. Each of the waste rock units are based on early exploration activity estimated to be equal in volume. The results from the screening will indicate if this scale is acceptable.
13	**Select the statistical approach** **2.9.2.5**	The selected sampling approach is judgemental - systematic. Judgement will be used to select samples from both waste rock types, at which point a systematic approach will be used to collect samples across the area of rock to be excavated. The judgemental selection will use the information in the drill logs to make sure that the two types of waste rock are proportionally represented. Twelve samples will be selected and submitted for processing tests. All of these will be sampled for the comprehensive characterization. In addition, a further 20 samples selected from the ore (targeted at typical and low and high grade ore and analysed for the comprehensive characterization methods).

14	**Choose the desired reliability (i.e. precision and confidence)** **2.9.2.6**	The required parameters (i.e. 98-percentile) are to be estimated to a precision of 20 % with 90 % confidence. The data variability will be calculated for the different analysis when results have been received and used in developing the percentile for the different criterions used in the waste management plan. This width of confidence interval is depicted by the pair of dotted lines in Figure A.1.  **Figure A.1 — Illustrative data for a Level 1 (comprehensive) characterization** NOTE In this example, that the estimated 98 percentile (for total sulfide as an example) is not much more than half of the inert waste criteria, and even its upper confidence limit is only about three-quarters of the inert waste criteria of being non-acid generating > 0,1 wt % pyrite.
15	**Choose the required statistical parameter** **2.9.2.7**	If there is testing for inert waste the 98th percentile will be used for sulfide content and the constituents of interest for leaching tests together with a 10 % confidence level. NOTE It is impossible to plan a sampling program to estimate the maximum, as the true underlying population maximum will always exceed the observed maximum by some unpredictable amount however the higher number of samples are taken thus lower is the unpredictable amount. The recommended approach is therefore to plan to estimate a high percentile (the 98 percentile in this example) to a specified level of precision and confidence, and then check that there is a reasonable safety margin between this and the inert waste limits. The data will be evaluated for the management plan and closure plan and from those evaluations confidence level and percentile will be assigned.
Determine the practical instructions, 2.9.3.		
16	**Identify the sampling**	At minimum 50 waste rock samples, 20 ore samples and 12 samples from discharge material of processing tests should be collected.

	pattern **2.9.3.2**	The sampling pattern will be selected based on evaluating the distribution of sulfides in three dimensions within and around the ore body. A key focus should be the transition areas between the ore body and high sulfide rocks and the transition between high and low sulfide zones. From the drill log data it has been determined that the drill holes intercept the variation of the geological strata. For that reason it is assumed that no bias will be introduced by adoption of a systematic increment sampling. Ten drill holes are set in the deposit which intercepts the waste rock areas. The drill holes are commonly between 100 m and 200 m long before interception of the ore body. The sulfide content, from background information, is linked to the ore formation and the host rock. The low sulfide waste rock has high actinolite and biotite content and somewhat higher calcite content than the host rock of the ore deposit. Distance between the ore body and the host rock is variable. A minimum of fifteen samples should be selected from the innermost 25 m (waste rocks nearest the ore body) the other 35 samples selected at different distance from the ore body within the host rock with a minimum of four samples from each drill hole. Within the total amount of 50 samples from the potential waste rock (excluding tailings and ore), the samples from the low sulfide waste rock should be collected on the basis of at least one sample from each drill hole and at least five samples from within 25 m of the host rock.
17	**Determine minimum increment size and sample size** **2.9.3.3**	**Increment size** Not relevant. **Sample size** If kinetic testing is required it will be necessary to collect a minimum of 10 kg. 3 kg to 4 kg for the kinetic tests and 2 kg to 3 kg for the remaining tests. The spare material should be stored in case follow up tests are required.
18	**Determine required number of samples** **2.9.3.3**	The total amount of core available is approximately 1 500 m. 50 samples representing the waste rocks (excluding tailings and ore samples) should be collected from the 10 drill holes. This will represent 1/3 of the total amount of material available in the first sampling round. There will be 12 mineral processing tests. The solid discharge from all of these tests will be analysed for the detailed characterization. These tests represent 6 samples of 10 m composites and run for two different processing set up. In order to evaluate the variability of the ore and hence the variability of the tailings samples will be collected from the ore as well. There will be taken from the 20 samples of the ore that will be analysed for the comprehensive characterization tests. The result of these tests will guide in further sampling for detailed characterization testing of the waste material.
19	**Decide on the use of composite and incremental samples** **2.9.3.4**	The exploration drilling will be undertaken using reverse circulation, samples will be collected that represent 2 m interval. All of these composites will be available for sampling. The mining operation will blast lifts that are approximately 10 m high. To produce waste rock samples that are 2 m interval will require mixing of samples from five consecutive bags. The same 10 interval samples should also

		be used for samples from the drill cores.
20	**Identify appropriate sampling techniques** **2.10**	**Waste rocks and ore from the storage building** Samples should be collected from the drill cores that are geologically, mineralogically and geotechnically logged by the mine staff geologists. Samples are to be collected from near ore and from distant ore near the edge of the future pit walls. It is estimated that the future waste will be approximately 400 million tonnes. The amount of the two waste rock types is unknown.
Sampling management in the field, Clause 3.		
21		All samples should be collected from the storage sample house. The samples should be dry. The samples should be stored in solid fabric bags within a large plastic bag to avoid contamination. The test laboratory will perform splitting of the samples before analysis and/or submission of samples to other laboratories. The remaining split of 2 kg to 3 kg should be returned to the mine for dry storage.

A.3 Example short form sampling plan for exploration/ permitting stage

The short-form sampling plan is intended to provide a statement of the activities that need to be undertaken in the field by the sampler. It should provide sufficient additional information that the sampler is informed of the purpose of the sampling activities and reasons why deviations from the plan might be appropriate or inappropriate in particular circumstances.

Short form sampling plan for the sampler	
General information	
Sampling plan completed by:	On behalf of:
Client (Company): Contact: Tel:	Material producer: Contact: Tel:
Other involved parties: Regulators of copper county, NGO´s, land-owner	
Sampling to be carried out by (Company):	Specify name of sampler:
Specify the objective of the testing programme	
Overall sampling objective: To perform characterization to fulfil the requirements of the Directive on waste for from the extractive industry, defining if parts of the waste can be identified as inert waste, developing waste management plans and closure plans. **Technical goal:** Collecting samples to evaluate initial information that there are two waste rock types and evaluate the variability of the ore and the waste.	
Developing the practical instruction	
Material	
Type of material: Material located in drill core boxes and rock chip bags — Waste rocks — Ore	**Location (address):** County Drill materials storage house
Form and nature of arising: Drill core and rock chips from reverse circulation drilling	
Background information: The quarry consists of a porphyry copper deposit containing low amount of	

sulfides (0,1 to 2 wt. % pyrite). There are ten drill holes, five of which are from core drilling and five are from reverse circulation drilling. The core boxes each represent 2,5 m of drilling and each bag 2 m.

Identify access problems that may affect sampling programme: None

Sampling methodology

Scale: 1 sample represents approximately 8,5 million tonnes.

Five consecutive bags (representing 10 m drilling) are to be collected from the reverse circulation drilling, split in two using an on-site splitter following the equipment cleaning instructions in the sample storage room. After splitting the samples are put into sample bags.

Each sample to be collected from the whole 10 m core interval. The core is to be split in two and the material placed in the sample bags.

Sampling population: 400 tonnes future waste material, drilling material from 1 500 m

Specify detailed sampling location based on the geological log data samples are to be collected from the following intervals of the different drill holes.

WC 102
10-20, 60-70, 110-120, *150-160,180-190*, 220-230, 280-290
WC 105
20-30, *80-90, 110-120, 140-150*, 210-220, 260-270,
WC 108
10-20, 40-50, 80-90, *110-120, 130-140*, 180-190 250-260
WC 109
30-40, 70-80, 130-140, *160-170, 220-230*, 290-300
WC 111
10-20, 60-70, *80-90, 110-120*, 240-250,
WR-115
0-10, 30-40, *60-70, 90-110, 130-140, 170-180*, 220-230, 290-300
ER-130
20-30, 80-90, *140-150, 170-180, 210-220*, 260-270, 310-330, 380-390
ER-132
10-20, 40-50, 70-80, *110-120, 150-160, 180-190*, 210-220, 280-290, 320-340
ER-140
0-10, 30-40, *90-110, 130-140, 170-180*, 220-230, 290-300
ER-142
0-10, 20-30, 40-50, *80-90, 130-140*, 170-180, 220-230,

Italic sample numbers are low sulfide waste; underlined are ore samples. C in the drill-hole name indicates core drilling and R indicate reverse circulation drilling.

Specify date and time(s) of sampling:

Specify persons to be present:

Identify equipment:

Specify no. of samples to be collected:

Specify no. of increments per sample:

Specify increment size/sample size:

Description of sampling event:

Cores should be cut in the sample storage house at the mine. ¼ of the core should be collected in the sample bags.

The splitting will be performed using a 16 shut, two way splitter, with 12 millimetre opening. One split goes back in the original sampling bag and placed back in the storage location, the other split goes into the fabric sampling bag. The total weight of the sample will be approximately 10 kg.

Detail requirements for on-site determinations: Identify at site the following items from the samples being collected:

— Mineralogy especially visible pyrite and other sulfide minerals

— Calcite and dolomite — Possible oxidation of the sulfide minerals (colour and secondary products)
Identify sample coding methodology: The samples will be coded according to the mine core drilling coding system with drill hole depth interval + sample data.
Identify safety precautions: Sampling shall only be carried out during regular office working hours at the mine. Mine safety regulations are to be followed and at a minimum using safety glasses, hardhat and steel toe boots inside the mine perimeters.
Sub-sampling: none
Packaging, preservation, storage and transport requirements
Preservation: none
Packaging: Heavy duty fabric bags placed in heavy duty plastic sample bags.
Storage: Store in the mine sample storage house until all the samples are shipped.
Transport: Deliver the samples to the laboratory using a proprietary courier.
Laboratory Instructions: Samples will be crushed and milled at the laboratory and split into 4 equal sub-samples. Two sample splits should be returned to the sampling contractor. One sample should be used at the laboratory performing the crushing and milling Lab X for ABA, NAG testing, total carbonate, and total chemistry, and leach test. One sample should be shipped to the chosen laboratory for mineralogy analysis.
Analytical laboratory
Company details: Contact: Delivery date:

A.4 Operation stage – Simple system – Aggregate quarry

Background: The material to be extracted from the ground consists of topsoil, overburden and the rock formation which is almost exclusively composed of limestone. All of the materials are considered to be inert. The topsoil and overburden will be used to backfill the excavation void. The limestone will be washed and the resulting tailings (which are made of dust and clay particles) will also be disposed into the quarry pit.

NOTE In several Member States, materials backfilled in the excavation void are not considered to be wastes.

	Step	Outcome
Defining the key elements of a sampling plan.		
1	**Identify involved parties** **2.2**	Quarry owner, Permitting authorities Independent consultant Stakeholders
2	**Identify the overall testing objectives** **2.3**	Comply with the Mining Waste Directive and thereby Identify the waste generated by limestone extraction (size, type, physico-chemical characterization).
3	**Background information and field inspection** **2.4**	The geological and pedological provide thorough information on different types of rock formation and soil layers covering them. The formation containing the prospected limestone has been subject to extensive studies and both its mineralogy and chemical composition are well known. Sulfide sulfur test and heavy metals analysis have been done on the overburden and topsoil showing very low levels. Ground and surface water analysis data are also available.
4	**Specific objectives** **2.5**	Ensure and verify that the materials are in accordance with the background information at hand (mainly geological and pedological description). Prove compliance with previous waste characteristics using a visual inspection by an experienced geologist. Verify and confirm compliance with inert waste criteria. The characterization exercise is required to: — optimise the waste management; — guide decisions on deposition methods and appropriate techniques; — evaluate closure options; — compliance with the available data.
5	**Phase of testing** **2.6**	Phase of testing : Confirmation testing (visual inspection) Depending on the results from on-site confirmation testing, further laboratory testing could be required if the test data indicates abnormal and unidentified differences between observed and expected material. As both the deposit and the overburden and topsoil are relatively homogeneous, the results of a previous screening and detailed characterization testing programme are deemed to provide sufficient data. The deposit has been shown to be homogeneous, and free of metal or sulfide bearing minerals.
6	**Identify tests and constituents to be tested** **2.7**	The drill cores samples will be visually inspected and analysed by an experienced geologist to ensure that the layers are in accordance with the background information and earlier test data. The visual determination of the material has proved to be very reliable as any occurrence of an unidentified layer would show from a difference within the colour and texture of the material.

	Step	Outcome
		Depending on the outcome of the analysis, the geologist may require that further testing be carried out. Further testing would then envisaged as follows: — total sulfur testing; — static test; — analysis of harmful metals; — leaching test if metal content significant; — mineralogy analysis by microscopy.
7	**Health and Safety precautions** **2.8**	Compliance with the Company or MS health and safety policy applied to core drilling.
Select/Develop the sampling approach, 2.9.		
8	**Identify the technical goals** **2.9.2**	The technical goal is to sample from rock cores, overburden and topsoil to confirm the geochemical characteristics as provide in the background information and quantify the amount of the different type of materials to be extracted.
9	**Identify the overall population** **2.9.2.2**	The deposit along with the future waste material within the boundary of the quarry to be deposited.
10	**Choose the sampling population** **2.9.2.2**	The sampling population is defined according to the pedological and geological maps with as a target the qualitative and quantitative evaluation of the underground material. The sampling population will cover both the rock formation and the overburden/topsoil.
11	**Assess variability** **2.9.2.3**	There are no relevant geochemical variations with regards to the compliance with the inert waste criteria. Furthermore, the extracted materials are very homogeneous (within given layers).
12	**Identify the scale** **2.9.2.4**	Considering the important size of the deposit, the scale is assumed to be 4 ha.
13	**Select the statistical approach** **2.9.2.5**	The sampling does not follow a specific statistical approach. Cores are drilled in selected places identified following detailed analysis of the geological map (e.g. around faults). Accessibility is an issue in some places due to the resilience of land owners.
14	**Choose the desired reliability (i.e. precision and confidence)** **2.9.2.6**	The purpose is to check that the material within the geological and pedological available descriptions.

15	**Choose the required statistical parameter** **2.9.2.7**	Not applicable: the drill cores are logged and analysed by a geologist.
Determine the practical instructions, 2.9.3.		
16	**Identify the sampling pattern** **2.9.3.2**	Cores are drilled in selected places identified following detailed analysis of the geological map (e.g. around faults, etc).
17	**Determine minimum increment size and sample size** **2.9.3.3**	Not applicable: the drill cores are logged and analysed by a geologist.
18	**Determine required number of samples** **2.9.3.3**	One drill core per 4 ha.
19	**Decide on the use of composite and incremental samples** **2.9.3.4**	Not applicable: the rock formations and soil layers are very homogeneous.
20	**Identify appropriate sampling techniques** **2.10**	Samples are to be collected from the drill cores that are geologically, mineralogically and geotechnically logged by the staff geologists. It is estimated that the future waste will be approximately 8 million tons (considering 30 years exploitation).
Sampling management in the field, Clause 4.		
21		The drill cores are deposited on a paddle and are transported by truck to the laboratory.

Annex B
(informative)

Example sampling record

(Example sampling record taken from EN 14899:2005:)

Sampling record	
Sample code: **(R** Reflect **site location, material type and date of collection)**	
Date of sampling	
Signature of sampler:	
General information	
Waste producer: Contact:	Client (Company): Contact:
Location of sampling:	Carried out by (Company): Sampler:
Sampling objective	
Material	
Type of material:	Estimated moisture content:
Description:(colour, odour, consistency/homogeneity/grain size – uniform or diverse)	
Sampling methodology	
Describe/define batch or consignment sampled:	
Place and point of sampling:	
Access problems that affected areas or volumes of material sampled:	
Date and time of sampling:	
Persons present (record name and address of witnesses present where appropriate):	
Procedure (describe procedure adopted):	
Equipment used:	
Number of increments/samples collected:*	Increment size/sample size:*
Observations during sampling: (e.g. gassing out, reactions, development of heat)	
Details of on-site determinations: (if undertaken complete field record sheet and append to sampling record, see Table B.2)	
Safety measures taken:	
Sub-sampling and pretreatment	
Identify location: e.g. on-site or fixed laboratory facility (describe whether open air or enclosed)	
Procedure:	
Packaging, preservation, storage and transport details	
Packaging:	Storage:
Preservation:	Transport:
Deviations from sampling plan	
Detail:	
Delivery to analytical laboratory	
Company:	Delivery date:
Received by:	Signature:

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