SLOVENSKI STANDARD

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Karakterizacija odpadkov - Vzorčenje odpadkov iz industrije bogatenja mineralnih surovin

Characterization of waste - Sampling of waste from extractive industries

Charakterisierung von Abfällen - Probenahme von Abfällen aus der mineralgewinnenden Industrie

Caractérisation des déchets - Echantillonnage des déchets issus des industries extractives

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Characterization of waste - Sampling of waste from extractive industries

Caractérisation des déchets - Echantillonnage des déchets issus des industries extractives

Charakterisierung von Abfällen - Probenahme von Abfällen aus der mineralgewinnenden Industrie

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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.

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.

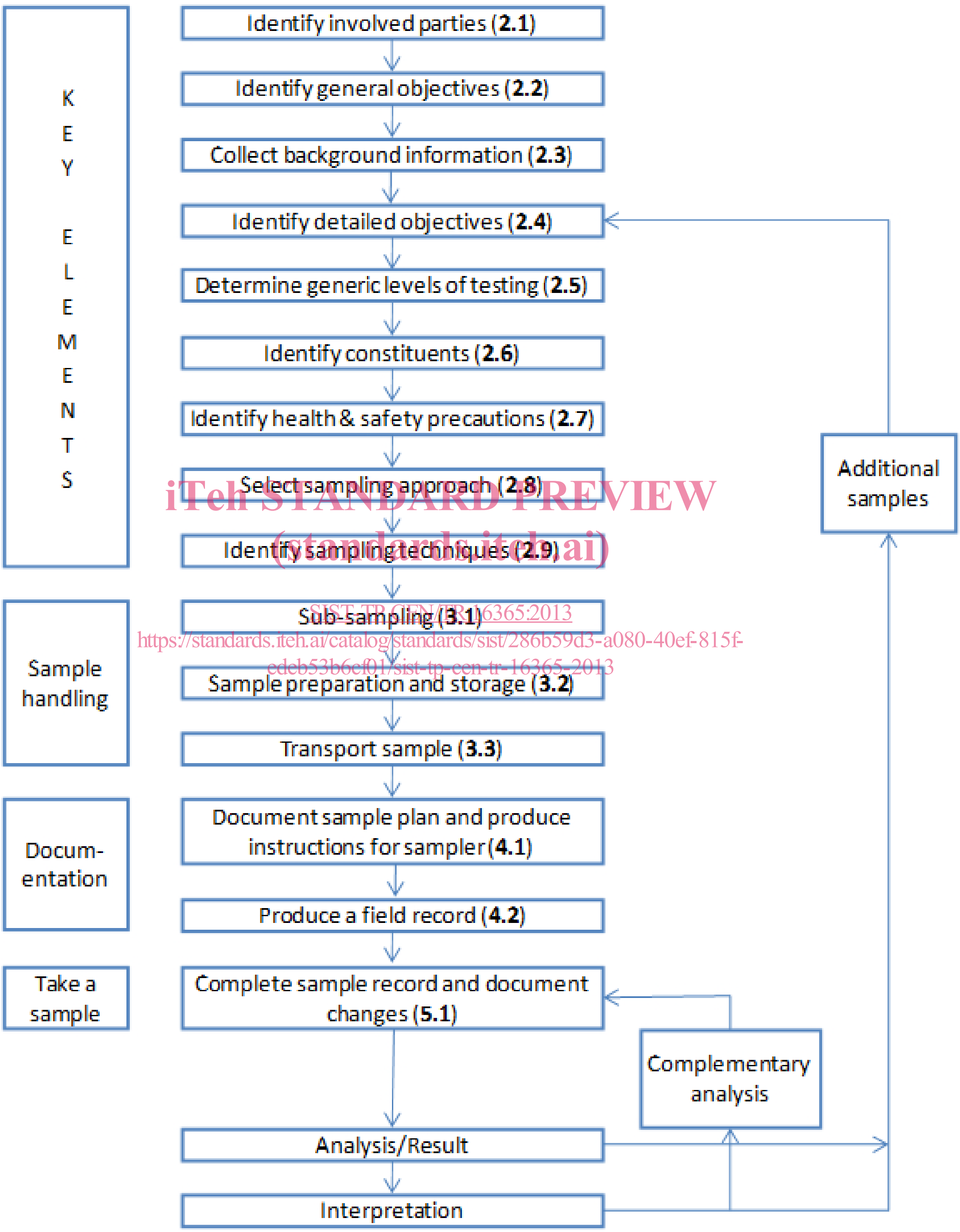
The framework standard EN 14899 identifies a process flow chart that defines the essential elements of a sampling plan and how those elements are linked. The basic steps identified in this flow chart have been followed in this supplementary guidance, with some minor changes (see Figure 1) to account 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.

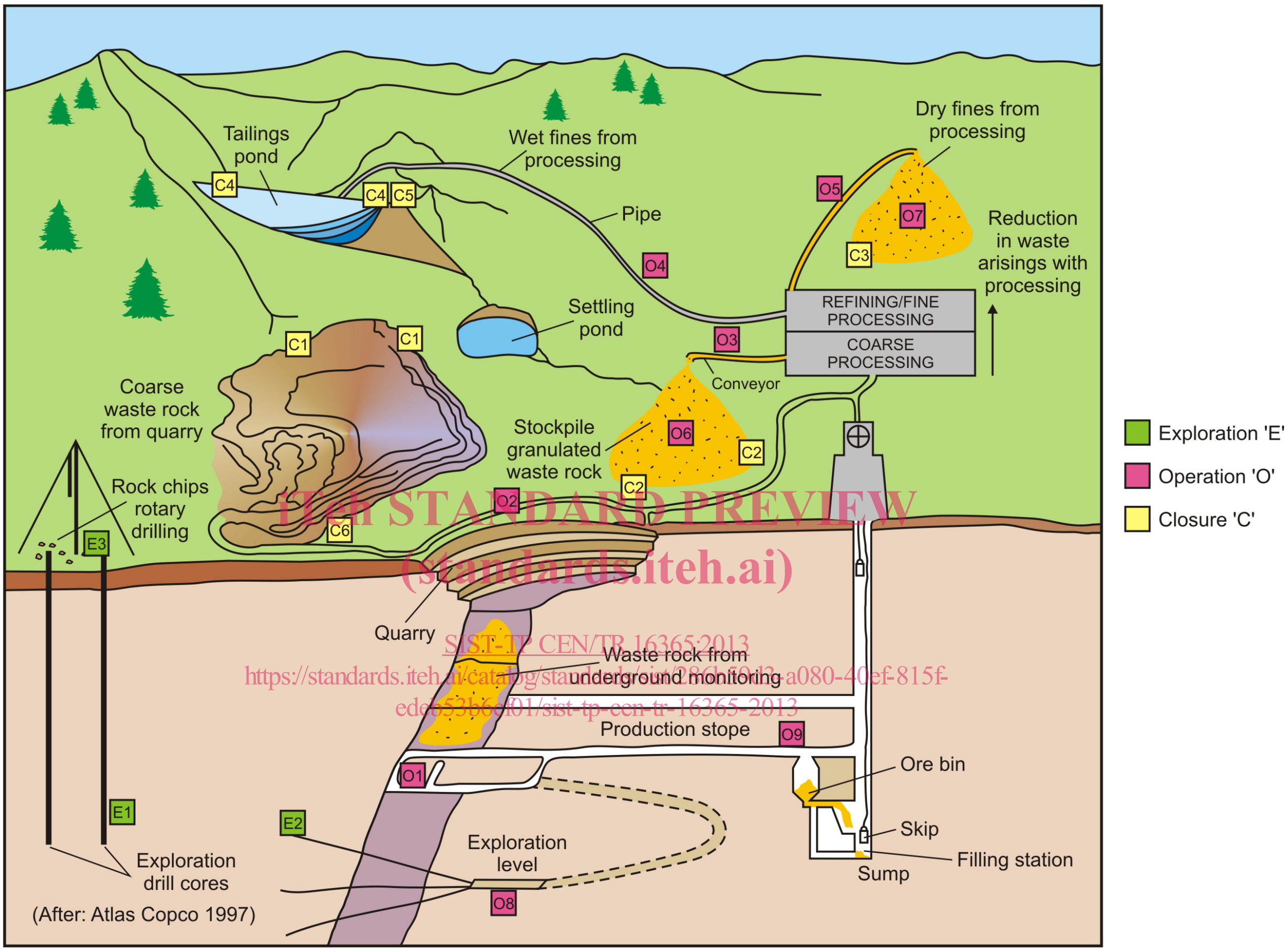


Figure 2 — Schematic of phased operation of mineral extraction linked to potential sampling objectives

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;