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Characterization of waste — Overall guidance document for characterization of waste from the extractive industries

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National foreword

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Characterization of waste - Overall guidance document for characterization of waste from the extractive industries

Caractérisation des déchets - Document guide pour la caractérisation des déchets issus des industries extractives

Charakterisierung von Abfällen - Leitfaden zur Charakterisierung von Abfällen der mineralgewinnenden Industrie

This Technical Report was approved by CEN on 24 June 2012. It has been drawn up by the Technical Committee CEN/TC 292.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (CEN/TR 16376:2012) 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. The target audience of this guidance document includes all stakeholders concerned with the management of extractive waste including authorities, regulators, waste producers, consultants and testing laboratories.

The overall guidance document is one out of three guideline documents (TR, technical reports) and one technical specification (TS) on aspects related to the characterization of extractive wastes developed by CEN/TC 292. These four documents are:

- overall guidance document for characterization of waste from extractive industries (CEN/TR 16376);
- guidance on sampling of wastes from extractive industries (CEN/TR 16365);
- kinetic testing for sulfidic waste from extractive industries (CEN/TR 16363); and
- sampling and analysis of cyanides (WAD) discharged into tailings ponds (CEN/TS 16229).

In addition to these four documents, CEN/TC 292 developed a European Standard (EN 15875) for static determination of acid and neutralization potential of sulfidic waste (acid-base accounting).

The overall guidance document applies to waste from extractive industries according to the waste definition in Art. 3 para 1 of the Directive 2008/98/EC on Waste. Therefore, as far as residues from the extractive industries are mentioned in this document they are only covered if they fulfil the criteria of the above mentioned waste definition.

This document provides guidance and is not a required procedure. It gives recommendations on what to evaluate during characterization of waste from extractive industries. It provides a tool box with many different methods that may or may not be applicable in a specific case, and it is not a legally binding document.

Introduction

Waste from the extractive industries can only be managed properly if sufficient knowledge about its geochemical and physical properties and behaviour is available. Such knowledge may be obtained through characterization of the waste. Consequently, Directive 2006/21/EC of the European Parliament and of the Council of 15 March 2006 on the management of waste from extractive industries and the associated Commission Decisions on waste facility classification, inert waste definition and waste characterization include several requirements related to characterization of waste.

In one of its decisions the European Commission states that: “The purpose of the characterization of extractive waste is to obtain the relevant information on the waste to be managed in order to be able to assess and monitor its properties, behaviour and characteristics and thereby ensure that it is managed under environmentally safe conditions in the long term. Furthermore, the characterization of extractive waste should facilitate the determination of the options for managing such waste and the related mitigation measures in order to protect human health and the environment.”

A multitude of methods and tools are available for various waste characterization purposes – some are standardized and some are not. Often several methods that differ only slightly from each other are available for the same purpose. Tradition and geography often determine which method is used. In some cases, the use of specific methods is required by legislation. Within EU legislation, European (CEN) Standards and methods are generally preferred if they are available. The implementation of Directive 2006/21/EC and the COM decisions calls for appropriate waste characterization which may be achieved by the use of several characterization methods and standards. Some of these methods and standards have been applied in the extractive industry for many years, while others have been less commonly used in this context. In a few cases, it has been necessary to develop or initiate development of new CEN standards for the purpose.

This overall guidance document has been developed by CEN under mandate M/395 by the European Commission to support stakeholders in the EU Member States involved in the characterization and management of extractive waste in selecting the appropriate waste characterization tools (standards or methods) for a given purpose related to the management of extractive wastes and to the requirements of Directive 2006/21/EC and the associated COM decisions. Furthermore; it is meant to provide information on the possibilities and limitations of the methods and to provide some guidance on where to find further information on the interpretation and application of the waste characterization results. The overall guidance document is intended to cover all the different waste categories produced by the wide range of sectors within the extractive industry and to reflect state-of-the-art with respect to waste characterization methods. It is the purpose of the document to provide the stakeholders with an overall summary of the specific aspects of characterizing waste from the extractive industries, but it will not replace the in-depth expertise required in most cases. Stakeholders include authorities, regulators, operators/waste producers, consultants and test laboratories.

1 Scope

This Technical Report gives guidance and recommendations on the application of methods for the characterization of waste from extractive industries[1], i.e. wastes resulting from the prospecting, extraction, treatment and storage of mineral resources and the working of quarries. The document covers characterization methods for both physical and geochemical properties and also other significant aspects, from planning to interpretation and reporting.

The main purpose of the document is to aid the extractive industry and regulatory agencies in the member states in understanding how to perform waste characterization for planned, active and closed extractive operations.

The document includes a discussion on when and why characterization may be needed and on the contexts 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.

The extractive industry covers many different sectors with very different waste categories and characterization may be carried out with many different objectives. For this reason, a guidance document on characterization cannot be prescriptive or provide generally applicable instructions on how waste characterization should be performed in each and every case.

2 Principles and procedures

2.1 Definition and role of waste characterization

Waste characterization is generally understood as the determination of waste properties and behaviour in terms of geochemical characteristics (e.g. composition, reactivity, thermodynamic stability, mineralogy, leaching properties) and physical properties (e.g. particle size distribution, density, permeability, compactibility, physical stability) and the interdependence and changes of these properties under varying conditions.

Whereas the above more general definition of waste characterization is fairly straightforward, the Commission (COM) decision 2009/360/EC on waste characterization applies a broader definition which includes a substantial amount of additional information. The COM decision also addresses background information on the extractive operation in question, geological background of the deposit to be exploited and on the origin and amount of wastes occurring during prospecting, extraction and operation as well as information on the classification, transport and management of the wastes produced[2]. These issues are discussed in Clause 4.

Waste characterization is primarily a management tool. In the extractive industry, waste characterization is often carried out to determine or estimate the present and future behaviour of a given type of waste under specified conditions to facilitate proper management of that waste. One cornerstone of the European legislation on extractive waste is the development of waste management plans, and one key component of a waste management plan is the waste characterization. The waste management plans will cover many aspects related to the waste management. Waste characterization may thus provide important information in many different contexts. For example, it may constitute an important part of an environmental impact or risk assessment, it may be used to assist in the definition of the most appropriate waste management solution in order to achieve physical and geochemical stability of the waste or it may be used to assess the suitability of an extractive waste for various construction purposes.

2.2 The waste characterization process at a glance

The starting point when designing a plan for waste characterization would normally be a definition of the general objective of the characterization exercise and the related questions that should be answered or may need to be answered at some time in the future.

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[1] as defined in Article 2 of Directive 2006/21/EC

[2] Sections 1, 2 and 3 of COM decision 2009/360/EC

Waste characterization is related to the short and long-term physical and geochemical stability and environmental performance of the waste facility and is carried out to understand the relevant waste properties in order to predict the behaviour of a waste material in a specified scenario. The properties that are of interest will depend on the situation at hand and the decisions to be made. In many cases, the leaching behaviour of the waste will be a key concern. Short term and medium term leaching potential will be an important input in the decision process with regards to the location and design of dumps or tailings storage facilities and the need for collection and treatment of drainage. The long term leaching potential will be the critical factor in the development of closure plans.

Sulfidic wastes, primarily from some metal and coal mines, require special attention due to the potential weathering of sulfides that may lead to the production of acidic (or neutral) and metal laden drainage, commonly known as acid rock drainage (ARD) or acid/neutral rock drainage (A/NRD).

When the waste management plan includes the construction of waste dumps, or the use of waste for dams or other construction purposes, stability is an issue and physical properties of the waste become a key concern.

To obtain a permit for a new or existing operation it is necessary to characterize the waste. The complexity of this characterization depends on the type of material. For a geologically/mineralogically uncomplicated case, with access to existing information, a field inspection and comparison with other sites with the same type of material may provide sufficient information. For geologically/mineralogically complex sites there may be many samples run for different types of analysis. During operation, sampling and testing may be carried out to check that the initial characterization remains valid and in some cases to check compliance when there are certain criteria that need to be met.

When the general objectives of the waste characterization have been defined the next step will be to define the relevant site specific scenario.

It is only then that decisions can be made on relevant waste properties, which tests to carry out, which methods to use, how many samples are needed and so on.

The sequence of characterization is illustrated in Figure 1.

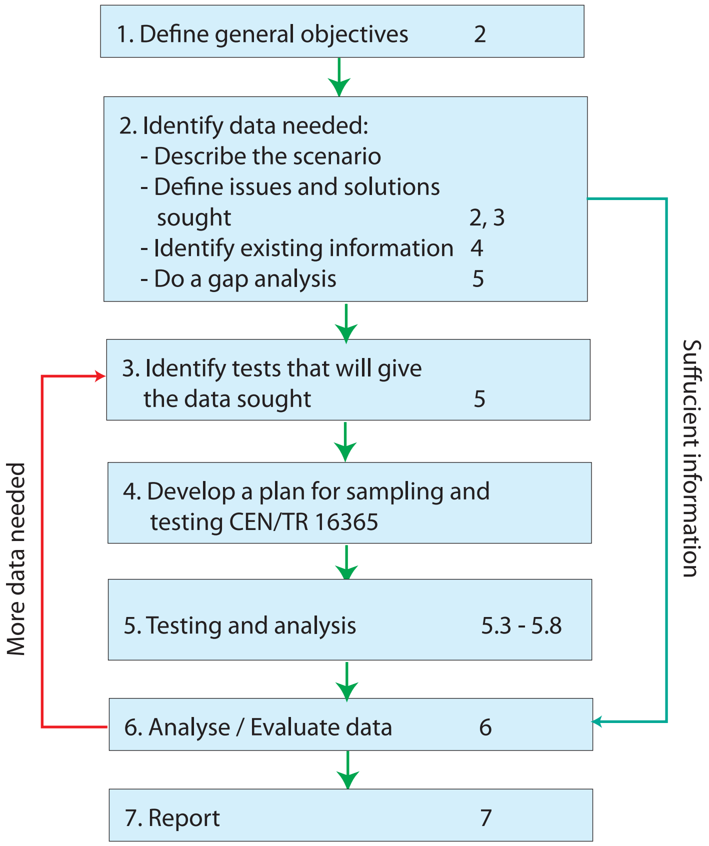

Figure 1 — Flow chart of the characterization process, with references to the clauses within this document

Below follows a simplified, stepwise description of the whole characterization process:

Step 1: Define general objective

As a first step it is important to define and understand the general objective of waste characterization in the specific case. Characterization should always be done with a purpose. Without keeping that purpose clearly in mind it is very easy to end up with an overly ambitious characterization program, or, with results that are too uncertain to allow any conclusions.

Examples of general objectives (most characterization programs will include multiple objectives) with references to current European legislation include:

- development of waste management plans (Dir 2006/21/EC, Art 5.3); including
  - design of waste facilities (Dir 2006/21/EC, Art 11);
  - assessment of leachate generation and design of treatment measures (Dir 2006/21/EC, Art 13);
  - development of closure plans (Dir 2006/21/EC, Art 12);
  - classification of waste facilities (COM decision 2009/337/EC).
- classification of inert waste (Dir 2006/21/EC, Art 3 and COM decision 2009/359/EC);
- calculation of financial guarantee (Dir 2006/21/EC, Art 14 and COM decision 2009/335/EC);

- classification of waste categories according to the European waste list (COM decision 2000/532/EC and subsequent amendments).

Step 2: Identification of data needed

The following four steps will have to be carried out in parallel.

- Describing the scenario

It is useful to document a general description of the scenario at hand (Clause 4) e.g.:

a) the kind of operation, existing or planned (e.g. metal mining, lime stone quarry, salt mining, open-pit or underground, size of mineral deposit);

b) the phase of operation during which the study is conducted (exploration/pre-mining, on-going operation, existing waste dump/disposal site);

c) the character of the surroundings (e.g. urban, industrial, farmland, forest, distance to receiving waters, existence of protected areas, other industrial activities);

d) the kind of waste management foreseen (e.g. disposal in dams, dumps, stockpiling, saturated/unsaturated, separation/mixing).

- Definition of the issues and the solutions sought

Based on the general objective defined in step 1, information about environmental aspects associated with different categories of extractive waste (Clause 3) and given the scenario at hand, specific questions that the operator (or the competent authority) wants to answer by characterizing the waste should be defined.

Examples of questions;

- Will there be a release of drainage from the waste facility? If so, how will the drainage quality develop over time? Will drainage water require collection and treatment to meet water quality standards? If so, what treatment would be appropriate?

- What kind of closure of the waste facility will be needed?

- Will the waste meet short and long-term specifications for construction purposes?

- Is there a potential to optimize the waste management by separation into different categories?

- Identifying existing information

It is not in anyone’s interest to carry out characterization work to produce information that is already available. Given the questions that need to be answered, relevant existing information should be identified (Clause 4). This could include:

- mineralogical and geochemical information from exploration/mapping;

- data from previous waste characterization;

- relevant information from comparable operations;

- information on the disposal site, topography, hydrology, geological and geotechnical;

- other local conditions, e.g. climatic data.

- Gap analysis

Does existing data (e.g. available characterization data on the mineralization and on future extractive waste, background information) give sufficient information to answer the relevant questions and to support the management decisions that have to be taken? If that is the case, no further testing is needed at this stage. However, there may still be a need to verify these results over time.

If the conclusion is that more information is needed, these needs should be specified in as much detail as possible to guide the choice of test method and the development of a characterization plan. Part of this specification should be an evaluation of the data quality needed, i.e. what level of uncertainty is acceptable (6.2). This will be important when deciding on a sampling strategy and might influence the choice of test methods.

The gap analysis might also lead to the conclusion that there is a lack of information regarding local conditions or other background information.

Step 3: Identify tests that will give the data sought

There are a large number of test methods for determining different waste properties, but also different methods for determining the same property. Some methods are standardized, some are established industry practice and some are under development. Some methods are applicable to specific types of extractive waste, but not to other types. When considering the use of any test method, its applicability and limitations should be well understood.

Clause 5 gives guidance on what methods are available and may be relevant for the determination of specific properties of extractive waste. Additional information is given in Annexes B to F.

Based on the information needs specified in earlier steps and information on available methods the most appropriate test methods should be identified. In some cases, a visual inspection may be sufficient to confirm that the waste material at hand corresponds to existing information while in other cases a whole package of tests will be needed.

See also 2.3 for some further comments on the choice of test methods.

Step 4: Develop plan for sampling and testing

The development of sampling plans is briefly described in 5.2. EN 14899 describes how the sampling plan for waste characterization should be compiled. Additional information specific for the extractive sector is available in CEN/TR 16365.

It is not possible to give general guidance on the number of samples needed as this will depend, amongst other things, on the amount and quality of background information and on the acceptable level of uncertainty of the results. In general, a more heterogeneous geology will require more samples than a homogeneous rock mass. Typically, exploration data will be used to design a sampling plan that is representative of the whole ore body and potential waste. In the case of sampling of waste from the process, the required frequency of sampling will depend on the variability in the waste stream. A statistical approach is recommended to avoid a situation where the characterization results are inconclusive due to too few, or non-representative, samples (for more information on statistical approaches see CEN/TR 15310-1).

Getting representative samples of future tailings will in many cases require processing of the ore in a pilot plant.

It is important that plans for sampling and testing are coordinated as e.g. the size of samples needed and potential pre-treatment or conservation requirements will be given by the test method(s) chosen. A general recommendation is to always collect and store extra material during sampling to allow for additional tests without renewed sampling (when testing is done on stored samples potential ageing effects will have to be considered).

Step 5: Carry out sampling and testing

Guidance regarding field sampling is given in CEN/TR 16365. It should be noted that sampling of extractive waste may present some very specific health and safety risks that shall be taken into account and managed (2.4).

When a high accuracy of the test results is needed it is recommended to use laboratories accredited for the test methods in question (not applicable for methods or within jurisdictions that are lacking accreditation schemes). General quality assurance and quality control (QA/QC) procedures should always be applied.

Step 6: Analyse/evaluate

When test results are reported they will have to be analysed and evaluated by a suitably qualified person with experience and knowledge of the behaviour of mineral wastes (Clause 6). It should be assessed whether these results allow the questions defined in the beginning of the process to be answered with an acceptable level of certainty. If not, additional sampling and/or testing will be needed (i.e. back to step 3 or 4).

There may e.g. be a need to take more samples because the waste proves to be more heterogeneous than expected, or the testing program may need to be complemented with more sophisticated methods to allow conclusions to be drawn.

In some cases the evaluation will be quite simple and straight forward: e.g. a comparison of total content with given criteria.

In other cases, the evaluation will involve interpretation of results, sometimes in combination with sophisticated modelling. E.g. when making assessments of drainage quality based on leaching tests. The scope of this document is limited to the characterization as such. Further guidance on how to carry out modelling, other than the overview of available modelling tools in Annex H, will have to be found elsewhere.

Step 7: Report

The whole characterization procedure should be documented and reported (Clause 7). It is important that the report covers not only final results and conclusions, but also the previous steps in the procedure, i.e. what decisions were made and why. What was the objective of the characterization, what background information was used, what assumptions were made etc.

Additional reports may be considered for specific audiences.

2.3 Choice of test methods

This guidance document roughly groups test methods (and properties) into:

- mineralogical analysis (5.3);
- geotechnical methods (5.4);
- geochemical analysis (5.5);
- leaching tests (5.7).

In the case of waste containing sulfide minerals, methods to evaluate acid generation and buffering potential have been developed (5.6). They can be divided into static (Acid-base accounting) and kinetic tests.

Figure 2 and the following text give an overview of categories of test methods and how they relate to potential waste management issues.

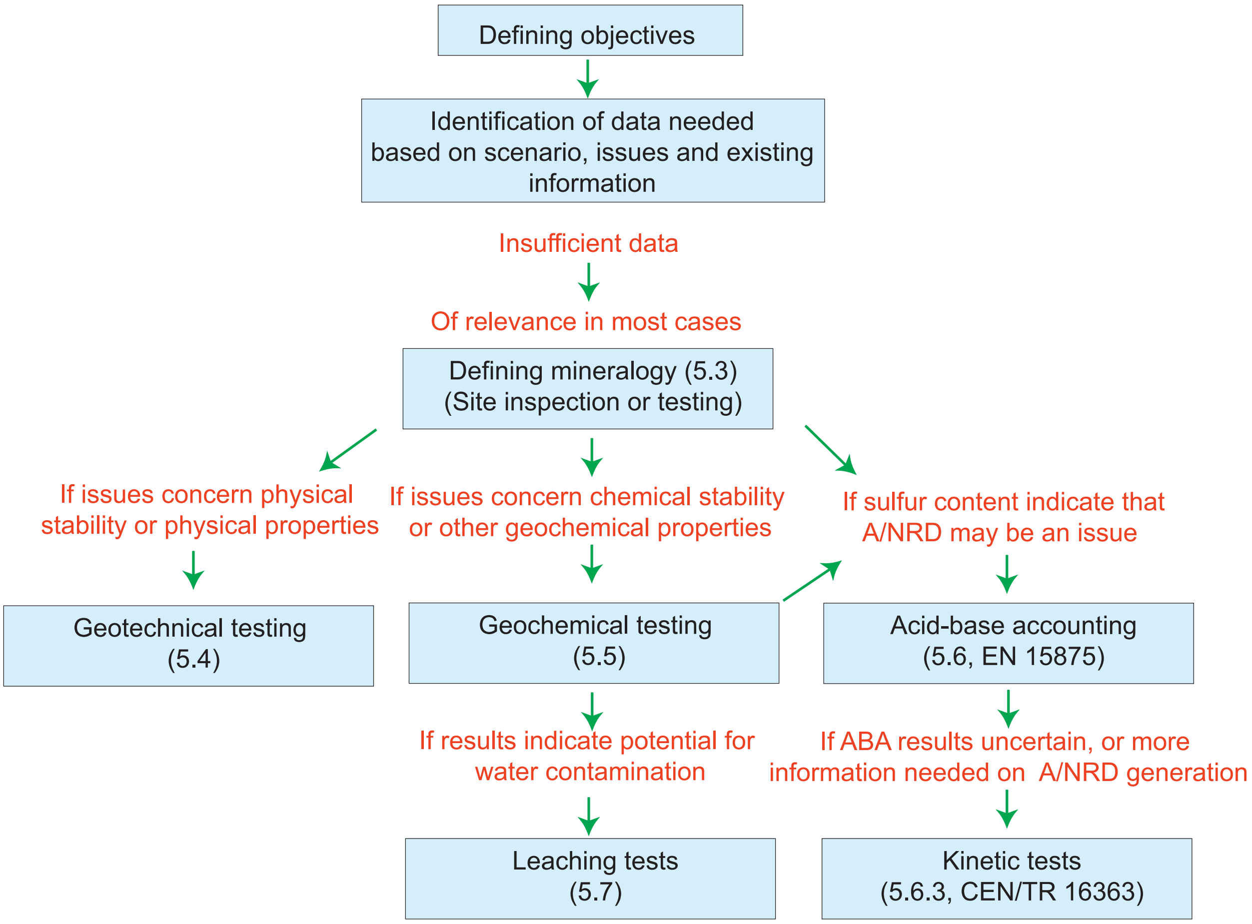

Figure 2 — Examples of sequences of testing in extractive waste characterization

For an assessment of the properties of extractive waste, a good knowledge of the mineralogy of the waste material is generally necessary. This information is essential for the evaluation of results from chemical analysis and leaching tests as well as for the assessment of A/NRD potential. Mineralogical information may be available from geological mapping and/or exploration efforts. An analysis of the mineralogy may take different forms. Depending on the quality of existing information and the complexity of the geology a visual inspection may be sufficient, while in other cases, advanced laboratory equipment such as electron microscopes or X-ray may be needed.

The physical properties of the waste relate, in particular, to two main aspects:

— Physical stability; e.g. slope stability of waste dumps or tailings used in dam construction, including changes of the stability due to physical or chemical weathering;

— Hydrology of the waste facility; e.g. infiltration rates, residence times, water saturation. Hydrology is a key factor in assessing the performance of any given waste facility design.

Chemical analysis to determine total concentrations of relevant substances will be part of most characterization programs. The substances of relevance are different for different types of waste from the extractive industry. Many test methods detect a whole range of substances, which may be useful for screening. However, characterization should focus on those substances that can potentially exceed critical levels in future discharges or, if the characterization is aimed at classification of the waste, those substances where relevant thresholds might be exceeded.

For an assessment of environmental impacts the total content is not representative of what may be released from the waste. There are different leaching tests available for this determination. Some tests will give total leachable amounts; others will be more relevant for an assessment of drainage quality.

Leaching tests should be carefully chosen depending on which data is needed to satisfy the defined objectives of the characterization exercise.

For waste containing sulfide minerals, the issue of potential A/NRD (acid/neutral rock drainage) has led to the development of a number of specially designed test methods for the assessment of whether or not a material will produce acidic drainage. Standard chemical or leaching methods are not sufficient to assess element release related to, or driven by, sulphide oxidation. For screening purposes, or as a first step, so called static testing, e.g. Acid-base accounting (ABA), is applied. The ABA test is a simple approach of comparing acid production potential based on sulfur (sulfide) content with readily available buffering capacity based on a standardized titration with acid. EN 15875 describes a European standard procedure for a 24 h ABA test. The ABA test results in a ratio of buffering capacity over acid production potential leading to one of three conclusions; “potentially ARD generating”, “not ARD generating” or “uncertain”. The ABA test is evaluating the potential of the waste to generate acidic drainage. It should be noted that if the waste contains sulphides that are oxidising, even if the drainage is well buffered, the drainage may still contain constituents of concern, i.e. what is often called Neutral Rock Drainage. If the ABA test does not give a clear answer, or if the waste is potentially A/NRD generating and there is a need for a deeper understanding of processes in the waste, there are a number of long term tests available, so called kinetic tests. These tests can be run in different scales, from lab columns to test dumps in the field. The set up will allow oxidation, weathering and drainage processes to take place in the waste, either under controlled lab conditions or under natural field conditions. Depending on the set-up the kinetic test results may form the basis for an evaluation of, among other things, A/NRD potential, reaction rates and drainage quality. Different kinetic tests are described in more detail in CEN/TR 16363.

2.4 Health and safety

The specific health and safety issues in the context of waste characterization are mainly related to field sampling. During sampling there are physical and chemical risks to be aware of. The physical risks may be related to:

- movement of large vehicles, e.g. trucks;
- collapse of waste rock heaps/slopes during sampling;
- active waste dumping;
- sink holes and cavities;
- inhalation of dust (e.g. silica, asbestos fibre);
- unstable wet tailings etc.

Chemical issues are mainly related to the use of potentially hazardous chemicals in the process (e.g. sodium cyanide) or off-gases resulting from processes in the waste (e.g. hydrogen sulfide or carbon monoxide).

The sampling plans should identify all relevant physical and chemical health and safety risks and safety measures should be specified. Health and safety issues should be streamlined with the health and safety procedures of the operator. Good practice when planning a characterization program would be to develop a project and site specific health and safety plan. It is recommended that, at an operating site, waste sampling should never be performed on your own and that at least one member of the sampling team should be a local employee. These issues are further discussed in the sampling standard EN 14899.

2.5 Applications

2.5.1 Development of waste management plans

For any operation producing and managing significant volumes of extractive waste the development of a waste management plan is good practice and is also required under current European legislation (Directive 2006/21/EC).

An appropriate description of waste properties will be a key input to many aspects covered by the waste management plan, including:

- design of waste facilities;
- development of closure plans;
- classification of waste facilities (e.g. according to COM decision 2009/337/EC), see 2.5.2.

Depending on the situation at hand, selected parts of what is presented in this overall guidance document are relevant in the development of waste management plans. It is recommended that a plan for the characterization work is developed based on the steps defined in 2.2.

The preamble to COM decision 2009/360/EC on technical requirements for waste characterization (point 3) specifically notes that characterization should be done primarily “on the basis of existing relevant and appropriate information or, if needed, by sampling and testing.” It further notes (point 4) that “the level of detail of information to be gathered and the related sampling or testing needs should be adapted to the type of waste, the potential environmental risks, and the intended waste facility.” The level of detail should also be correlated to whether or not the characterization results may change the waste management procedures.

2.5.2 Waste facility classification

EU legislation requires certain extractive waste facilities within the EU to be classified in one of two categories. The methodology and criteria for classification are described in more detail in COM Decision 2009/337/EC. One key factor to consider is the relevant waste characteristics.

The classification should be based on an assessment of:

a) the consequences of a potential failure due to the loss of structural integrity or incorrect operation;

b) the percentage of the waste that is either classified as hazardous waste or the content of substances or preparations classified as dangerous.

The COM Decision specifies a number of factors to consider when assessing potential consequences of a failure (Articles 5 and 6), one of them being the physical and chemical properties of the waste in the facility.

The most important physical properties for this assessment will be those related to the potential spread of the waste (see 5.4).

The chemical properties that will be of interest relate to both total content and mobility of constituents in the short and long term (see 5.5, 5.7). According to Article 4 of the COM decision, this information will be used as input to establish the source term in the context of a source-pathway-receptor chain.

Article 3 of the COM decision further requires an assessment of potential hazards related to reactive waste. Hence, it will be necessary to determine if the waste is “reactive”, i.e. if it is thermodynamically unstable under present or expected future conditions and therefore may react (for example oxidize) and cause the release of significant amounts of contaminants or heat. Potential self-ignition is one such property that indicates “reactivity”, A/NRD production capacity is another (see 5.6).

An analysis of total chemical composition (see 5.5), an assessment of self-ignition properties (see 5.5) and of the potential for A/NRD production (see 5.6) of the extractive waste may be required to establish whether or not the waste, or particular sub-categories of waste, is hazardous in accordance with Directive 91/689/EEC and subsequent amendments.

Finally, according to the COM decision the assessment of dangerous substances content is based on an inventory of substances and preparations used in the process and does not require any characterization of the waste. However, in the case of operating tailings storage facilities this inventory can be replaced by a chemical analysis of the water and solids contained in the facility.

2.5.3 Inert waste classification

All of the criteria given in COM decision 2009/359/EC on the definition of inert waste are dealt with in the characterization options and tools that are described within this document (see Clause 5). A couple of the criteria are judgemental, i.e. “sufficiently low” and “substantially free”. It is not a part of the mandate to CEN to make those judgements. In order to evaluate “sufficiently low” content the COM decision states that “the content of these substances [substances potentially harmful to the environment or human health] shall not exceed national threshold values for sites identified as not contaminated or relevant national natural background levels”. Methods described in this guidance may be used to evaluate the speciation of metal-bearing minerals in order to accurately determine the content of substances potentially harmful to the environment or human health. This information may be considered in the assessment of “sufficiently low content”.

In addition, Article 1(3) of COM decision 2009/359/EC states that the Member States may draw up lists of waste materials to be regarded as inert in accordance with the above criteria. In such cases, the characterization required by authorities may be limited to verifying that the waste at hand corresponds to the listed waste material.

2.5.4 Prediction of discharge water quality

The results of waste characterization play a key role in assessments of the potential impact of extractive waste on groundwater, surface water and surrounding soil due to leaching from the waste and subsequent attenuation and transport of released substances and in the design of measures to control such impacts. Leaching behaviour is a key property in this context. However, there are different test procedures for leachability that will be more or less suitable depending on the scenario at hand. Moreover, there is still a long way to go from characterization data on leachability to an assessment of what might in fact be the discharge from the waste facility. There are modelling tools (batch modelling or reactive transport modelling) available with the ability to simulate different chemical and physical reactions that will influence the drainage water quality on its way through the waste deposit (see Annex H).

A risk to the environment caused by leaching may be described as a chain of events (Figure 3). In order for a negative effect to occur at the receptor or target, the chain shall remain unbroken. An assessment of the resulting impact is made based on knowledge of the nature, size and behaviour of the source, the pathway and the interaction between the released substances and the pathway (soil, groundwater, surface water) and the nature and vulnerability of the receptor (or based on legal requirements at a point of compliance, e.g. expressed as a concentration at the point of discharge or as an environmental quality standard, EQS).

In a waste characterization context, however, only the source term in the impact assessment chain will be addressed. Guidance on impact assessment as such, including determination of the transport and effect of contaminants shall be sought elsewhere.

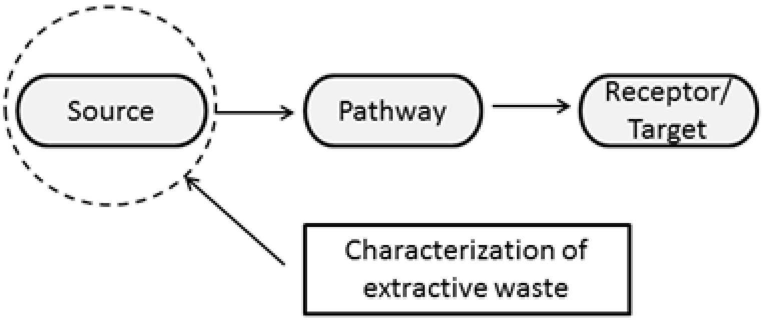

Figure 3 — The concept of impact illustrated as a chain of events.

NOTE For an impact to materialize, the chain remains unbroken. The figure also indicates that characterization of waste only applies directly to assessment of the source term

To define the source term information will be needed both on the leaching behaviour of the waste (including potential effects of weathering), the physical properties of the waste, the scenario (design) and the water balance, calculated or measured, for the system in question.

For this type of analysis the leaching behaviour may be described as a function of different parameters such as pH, redox, liquid-to-solid ratio (L/S) or time, water saturation, mineral reaction rates etc. This will generally require more than one test and careful interpretation of the results (e.g. by means of modelling).

The water balance will depend on external factors such as climate and rainfall, on design parameters such as surface area and slopes, covers etc. and on waste specific properties such as particle size distribution and hydraulic conductivity. It is only the waste specific properties that will be part of the waste characterization.

2.5.5 Assessment of closed waste facilities

Waste characterization is an essential part, but not the only one, of the assessment of a closed waste facility. Closed extractive waste facilities may in principle be evaluated in the same way as active waste dumps and tailings ponds. Ideally, there will be enough information available through records and from field inspections to evaluate the risks related to the physical and geochemical stability of the waste material. If available, monitoring borehole data (level and quality) may provide very useful information.

A field inspection will in many cases give a lot of valuable information on the properties and status of the waste. Things to look for include general design, signs of leakages, erosion and cracks. Further guidance on field inspections can be found e.g. in CEN/TR 16365.

If there is not enough data to evaluate the facility after reviewing existing information and the field inspection, further waste characterization may be performed by carrying out geotechnical and/or chemical/mineralogical analysis following the same structure as described in 2.2 and the flow chart in Figure 1.

In evaluating the chemical-mineralogical aspects of closed sites containing sulfidic tailings it is important to evaluate the oxidation stage and the oxidation front in the tailings. This can be done by field observations in combination with sampling and testing of waste material and pore water.

Most of the information on investigations of existing facilities given in 5.8 is relevant also for closed facilities.

2.5.6 Planning for monitoring

Monitoring of seepage water quality and/or groundwater quality is commonly performed during operation and after closure of facilities. The need for monitoring should be evaluated based on the result from the characterization, the type of facility and other local factors. If the waste characterization does not indicate any concerns regarding water quality, further monitoring may not be needed or justified.

Data from monitoring of seepage water quality and/or groundwater quality can be used for verification of predicted water quality and be considered as in-place field scale leach/kinetic tests. The level of monitoring (e.g. number of monitoring points, frequency and constituents analysed) should be proportionate to the potential for significant environmental impacts.

It may also be advisable to verify within the waste facility that predicted mineral reactions and weathering are in fact taking place. This type of information together with the water quality monitoring could give valuable input to the planning of progressive or final closure.

3 Waste categories, potential environmental impacts and related characterization methods

3.1 General

Within the extractive industry there are a number of greatly differing sectors. A distinction is often made between construction minerals (minerals mainly intended for the construction industry), industrial minerals (intended for the manufacture of products such as glass, ceramic and paper), metal ores (intended for the production of metals) and energy fuels (intended for energy production).

The environmental issues vary from sector to sector, within each sector, and even for different wastes at the same site, depending upon the mineralogy of the material and surrounding rock, the processing type (including chemicals added during processing), the size of operation and the location for deposition of the waste.

This means that the appropriate set of characterization methods to be applied will differ between sectors and for different waste types.

With respect to the types of wastes falling under the term “wastes from the extractive industry”, a substantial difference in potential impacts exists between acid producing waste, highly alkaline wastes, salty waste and fines from aggregate extraction. This ultimately translates in the required level of testing needed for the different types of waste.

More details on the different sectors, their processes and related waste categories can be found in the BAT document on mine waste management (Reference Document on BAT, 2004). However, it should be noted that, due to the limited risks associated with the sector, the BAT document does not include the extraction of construction materials/aggregates.

It should also be noted that, while this document is focusing on the characterization of solid wastes, the basic principles and many of the methods presented may be applied to other waste categories within the sector. E.g. the process water being discharged together with tailings is also a part of the waste stream and may contain, besides elements dissolved from the minerals, process chemicals and chemicals from blasting. An extractive operation may also produce wastes in the form of sludge from the treatment of water or gases. During drilling, especially for on-shore oil exploration there can be considerable amounts of drill mud that also need to be discarded in a managed way.

3.2 Characterization during the different phases of extractive operations

In general, the life of an extractive operation can be divided in three (or more) phases:

- exploration (including feasibility and design);

- operation (including transport and deposition);
- closure (including after-care).

While waste will mainly be produced during the operation phase, waste characterization will have to be considered in all phases of the mine life.

The exploration phase starts with the first evaluation of the potential for economic mineral deposits. This may be done by evaluating existing geological information such as geophysical and geological mapping. Drilling may follow if the first evaluations seem promising. When the exploration phase moves into feasibility study, the development of a permit application becomes a part of the project. It is normally in the permitting process that the waste characterization becomes more focused, even though the environmental issues should be a part of the project from the very beginning.

Most of the characterization will normally be done during the exploration phase. However, at this stage the amount of material available for sampling and characterization is limited. In the early stage of exploration, there may only be a limited amount of drilling or surface sampling performed to define the mineralization (pre-feasibility stage). In the later stage of exploration (feasibility stage) there is often a larger amount of drill cores or surface samples collected. There may also be test pits or shafts excavated in order to evaluate the quality of the material, test the processing methods or evaluate the ore grade. In this stage of exploration the amount of material available for characterizing the future waste would normally not be a limitation. It is commonly in this later stage that the characterization needed for the waste management plan and the permit application is carried out. When defining the objective of the characterization program the information needs for the permit application, for the waste management plan, for the design of waste facilities and for the closure plans, will be important to consider.

Exploration drilling will normally continue throughout the life of the operation.

For a new mine, the operation phase starts when the permits have been issued and excavation and construction works can commence. During the operation phase characterization is primarily performed in order to confirm the results of the earlier characterization, performed during the feasibility study and preparation of the permit application. Confirmation characterization will normally be performed primarily on the waste streams (conveyer belts, haul trucks, pipelines etc.) but may also be based on samples from drilling (exploration or production) or from the production front in the mine/quarry itself. However, if the verification does not agree with the previous characterization and the criterion set up for production, a renewed detailed characterization may be needed.

For mines that were already in operation when EU Directive 2006/21/EC came into force the requirement to develop a waste management plan may include the characterization of waste already deposited on waste rock dumps and tailings dams, if that information does not already exist.

Expansions or other changes of the operation may also lead to the need for a renewal of the permit, including requirements to carry out characterization of expected waste streams.

A conceptual closure plan will be required before start-up of a new mine or quarry. The plan will be based on the early waste characterization. During the operation, and particularly as part of the detailed closure planning towards the end of the mine life, there is a need to verify and update those early characterization results and, if needed, modify the closure plan. At this stage, valuable information (confirmation) can also be obtained by field investigations of deposited waste and of actual pore water and drainage water quality. However, towards the end of mine life the available management/closure options have usually become more limited.

Where possible, closure/reclamation is carried out as an integrated part of the ongoing operation.

Table 1 — Illustration of characterization efforts and closure options during phases of an extractive operation

Phases of an extractive operation	Characterization	Access to information	Management Options (incl. closure)
Exploration (incl. design and permitting)	Initial characterization followed by detailed studies.	Usually limited to drill cores. Representativeness a major issue.	Many options.
Operation (incl. construction)	Follow up characterization, testing for closure alternatives.	Easy access to samples, gradual build-up of data base including field data.	Reduced amount of options, but equipment available, can make adjustments and changes to the closure plans.
Closure and after closure	Verification of the models, waste management plans, long term mineral weathering etc. set up during operation. Verification of effectiveness/function of implemented closure measures.	Access to full scale, in situ, information.	Very limited. Location given, Only variations within the options chosen earlier.

EU member states are required to keep an inventory of closed waste facilities. The purpose of characterizing the wastes at closed and/or abandoned sites/facilities would commonly be to evaluate if they constitute a risk to human health and safety and to the environment and if they do, to determine what is needed to reduce this risk to acceptable levels. Aspects to be studied would normally include physical stability of dams and dumps and leaching potential/chemical stability of the waste.

3.3 Types of waste facilities

Within the extractive sector there are different types of waste facilities. The two main types are dumps/heaps and ponds/lagoons. The methods of deposition, the properties of the underlying soil/bedrock and the local topography are examples of other important factors that will differ from site to site. In combination with different waste properties this means that waste facilities will represent different engineering, environmental, economic or human risks or challenges and therefore the appropriate characterization approach will vary.

Overburden and waste rock are typically deposited on heaps (if not back-filled or used for construction purposes). This material is characterized by diverse grain size, from boulders to clay fraction, heterogeneous composition and varied water content. In some sectors tailings are dewatered and deposited on heaps. This is common practice e.g. in the potash industry.

Washing plants at quarries are designed to remove fines and generate a clean mineral product from crushed rock or sand and gravel using wet processes. Fines are removed from the water phase in settling lagoons or by thickener/ filter press systems. The settling lagoon becomes the final deposit for these fines while filtered fines may be dry-stacked. Water is typically recovered from these systems and recycled to the washing plant in order to minimize water consumption.

Tailings from flotation, or other processing, deposited in tailings ponds present different properties and risks. Tailings are normally composed of fine-grained material with relatively uniform grain size distribution. Tailings are commonly separated by hydrocyclone in fine-grained and coarser-grained fractions at the concentrator to produce back-fill material or dam construction material at the disposal site.

In this way, the coarser fraction may be utilized while the water-saturated, fine-grained material is deposited into the tailings pond. Even though tailings are normally more homogenous than waste rock and thus easier to characterize, tailings in general represent a higher risk of failure, and a higher risk to cause external harm in case of an accident. This is why tailings and tailings storage facilities are in the foci of geotechnical characterization of extractive wastes and waste deposits. A current trend in tailings management is the increased application of paste technology. Paste makes it possible to use un-separated tailings for backfill or to design a tailings storage facility with much less free water. In most cases this would increase the physical stability but introduces other challenges.

3.4 Associated environmental aspects

3.4.1 General

The environmental aspects of extractive wastes can in general be split in two categories: physical and geochemical. These aspects are briefly discussed in the following sub-clauses. Further information can be found in the BREF document (Reference Document on BAT, 2004), Chapter 1.6 “Key Environmental Issues”.

3.4.2 Risk of dam / slope failure

3.4.2.1 General

One of the primary risks is that of physical movement of the waste, either through failure of the deposited waste itself or of its confining structure. The risk of dam or slope failure is relevant for most mines and quarries. In the context of slope/dam stability the physical properties of the waste are relevant for dry-stacked tailings / waste rock dumps, for the use of residues (waste rock and/or coarse tailings) in dam construction and for the calculation of loadings on dams. The geochemical characteristics of the waste may also have an impact on stability as they might lead to a change over time of the physical properties.

In addition, both physical and geochemical properties of the waste will be relevant for the assessment of the potential consequences in case of a failure.

This guidance document does not deal with the design of dams or give guidance on the assessment of dam/slope stability or on the assessment of consequences of a dam/slope failure. However, waste characterization will provide essential input to such assessments and, therefore, a brief introduction to these issues is given here.

3.4.2.2 Waste dumps/tips/spoils

The placement of waste materials such as waste rock on dumps is generally done either by vehicle or conveyor at a natural angle of repose. Under some circumstances such waste materials can both fail and liquefy resulting in the footprint of the slide extending far beyond the toe of the waste slope. Though such occurrences are rare, and the risk usually confined within the operation site, they can and have happened and the risk needs to be recognized and appropriately addressed in the design of all waste dumps.

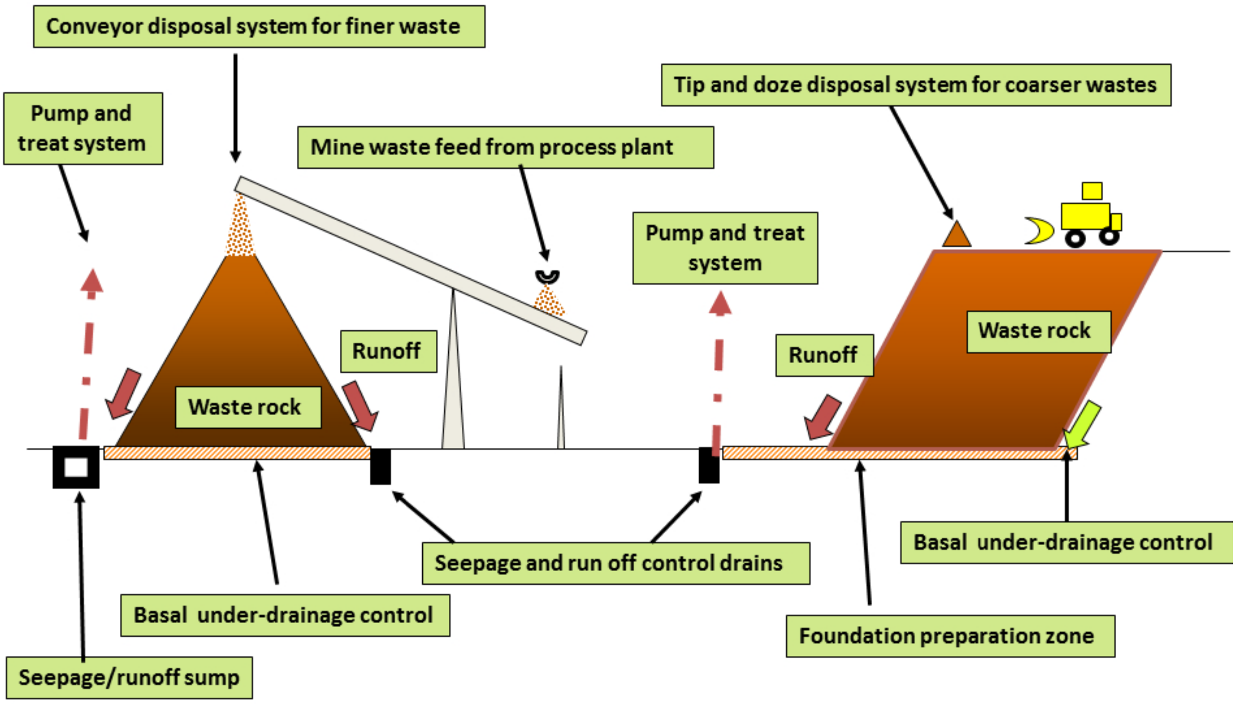

Figure 4 — Two typical sections of waste rock dumps (from Cambridge 2010)

The construction of the waste dump/tip should normally follow established geotechnical design procedures associated with slope stabilization, e.g. material selection, controlled placement/compaction and control of underlying ground. During its active lifetime the dump may receive a wide range of material types and sizes dependent on the geological setting of the ore body. The physical and geochemical properties of the different waste materials should be evaluated and characterized as appropriate to allow for a design and operation of the tip recognising the properties of the varying materials over time.

3.4.2.3 Tailings ponds/silt lagoons

Tailings are generally produced and discharged in a slurry form (as a mixture of particles and water) to a facility where sedimentation takes place. The aggregates and industrial minerals sectors tend to refer to these as silt lagoons and the metal mining industry as tailings management facilities (TMFs). The purpose of a TMF is the disposal of tailings from an extractive activity in a way that is designed to remain stable for an infinite amount of time (Bjelkevik 2005a). In many cases, these facilities are also designed to provide a cost effective and environmentally appropriate means of recycling process water.

A typical section through a tailings management facility is shown in Figure 5 including a number of key features of a complex facility. It should be noted that, depending on the setting, not all the features shown may be required or relevant. Depending on the water balance/water management there may be a need to discharge excess water. This water may need treatment before discharge.

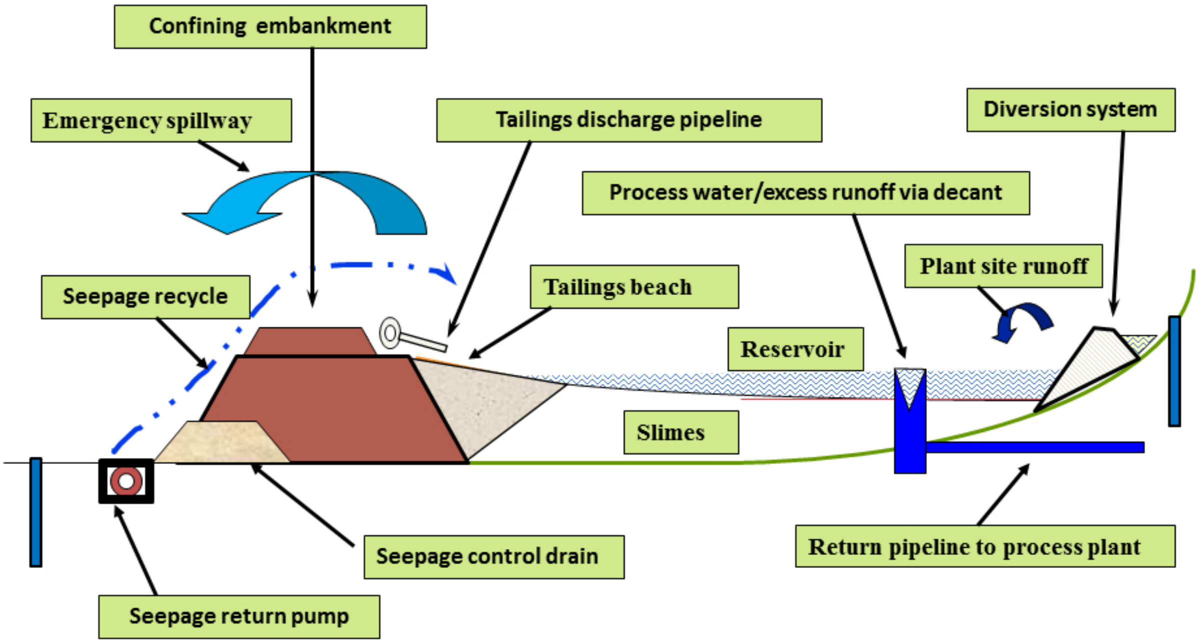

Figure 5 — Typical section through a tailings management facility, TMF (from Cambridge, 2010)

The embankment may be constructed using a wide range of construction techniques and materials, including locally won borrow materials, waste rock derived from the mineral extraction operation or the tailings themselves. A good understanding of the different properties of the materials available for construction and how those properties may change over the life of the facility is important. In each case the intrinsic physical and geochemical properties of the materials to be used shall be characterized and the design prepared accordingly using recognized good practice.

Dam failures (total or partial) and incidents related to the stability of the tailings dam may be caused by several factors related to the properties of the construction material, e.g.:

- internal erosion;
- external erosion;
- seepage;
- changes in physical properties of the dam over time, e.g. due to sulfide oxidation;
- overloading of the facility;
- frost heave and thaw weakening;

as well as by external factors, not related to material properties, e.g.:

- inadequate foundations;

- overtopping of dams;
- seismic events.

(Ref: e.g. ICOLD 2011, 2009, 2001)

The failure of a tailings facility could potentially lead to significant consequences due to the immediate impact of any spillage and the resulting short, medium and long-term effects (e.g., contamination of soil and water bodies, loss of animal life, etc.). Disregarding hazardous properties, any fine grained waste may cause an environmental impact due to the formation of high levels of suspended materials in the receiving waters. The potential long-term negative effects on the affected ecosystem will depend on the hazardous properties of the waste, but also on the remedial actions taken after an accident.

The risk to life and the environment downstream shall be assessed in order to classify the facility and to determine the appropriate factors of safety to be used in the design. An appropriate characterization of the waste will be essential for these assessments.

Some potentially useful tools for waste characterization to produce input data for the design and/or classification of waste facilities are discussed in the following clauses of this guidance, including:

5.3 Mineralogical analysis;

5.4 Physical and hydraulic properties;

5.5 Chemical Analysis (for solids and water);

5.6 A/NRD prediction tests (static tests and kinetic tests, laboratory and field scale); and

5.7 Leach tests (short term for materials that are not affected by reaction rate or are in a stable release setting).

3.4.3 Risk of dusting and siltation

The risk of significant erosion or dust emissions is relevant for most extractive waste facilities and most waste management techniques.

Run-off from waste rock dumps, quarry waste tips or quarry fines stockpiles can cause erosion and contaminate local watercourses. Suspended solids (siltation) may harm freshwater ecosystems and impact on other water users.

Dust can be picked up by the wind from the ground or a stockpile. Dry sections of tailings storage facilities as well as large quarry waste tips or quarry fines stockpiles can be a source of airborne dust, which can be exacerbated if the facilities are elevated above the original ground level. Generally, the impacts diminish greatly as distance from the source increases and the most acute impacts are likely to occur in close proximity to major sources.

Transportation of waste (or products) on conveyer belts and/or trucks is potentially another significant source of airborne dust.

Examples of key properties that would feed into an assessment of these risks are:

5.8 Particle size distribution, density;

5.5 Chemical composition.

3.4.4 Risk of contaminated drainage

3.4.4.1 Risk of acid/neutral rock drainage

The risk of environmental impacts related to acid/neutral rock drainage is related to the sulfide content of the extractive waste.

When sulfide minerals are exposed to oxygen, they are likely to oxidize and may have the potential to generate sulfuric acid. Due to the oxidation (mineral weathering), there is a potential for metal release from the same sulfide minerals. This process is called acid rock drainage (ARD) or acid mine drainage (AMD).

Not all sulfide minerals generate acidity during oxidation, but they may still release metal ions. If acidic conditions are generated, metals may also be released from the non-sulfide minerals.

There may be minerals present that have the potential to consume the acid and, thereby, raise the pH. The most efficient of these minerals are some of the carbonate minerals, particularly calcite, while dolomite and siderite have a lower reaction rate.

When the acid produced from sulfide oxidation is being neutralized, it is called Neutral Rock Drainage and the combined processes are referred to as acid/neutral rock drainage (A/NRD).

The transport and/or attenuation of released metals will among other things depend on site hydrology and on material properties.

This is a complex system and predictions of drainage water quality will in most cases require geochemical and transport modelling. While waste characterization alone will not predict actual drainage quality, it will provide essential input data to these models.

The A/NRD concepts are further discussed in Annex G, A/NRD processes and Mineralogy.

Some potentially useful tools for waste characterization for the purpose of A/NRD prediction and management are discussed in the following clauses of this guidance, including;

5.3 Mineralogical analysis;

5.4 Physical and hydraulic properties;

5.5 Chemical Analysis (for solids and water);

5.6 A/NRD prediction tests (static tests and kinetic tests, laboratory and field scale).

3.4.4.2 Other impacts on drainage quality

Other wastes from the extractive industries may provide other stresses to the environment than those caused by acid production.

Alkaline drainage in the extractive sector is commonly an effect of the processing agents used, e.g. soda ash and lime used in processing bauxite ore for Al production and sodium cyanide used in gold leaching.

Another potential impact is high salt loads in the discharge from salt (e.g. potash) extraction and waste facilities.

Most operations need to blast to excavate the material. This may result in contamination of water from undetonated explosives. Nitrogen compounds from undetonated explosives may be leached into groundwater from the production site or from the waste rock dump, or it may follow the ore to the processing plant and end up in the process water in the tailings pond.

During processing there are many different chemicals that may be needed as part of a mineral separation process, e.g. frothers or suppressing agents, and during settling of fine grained material. These chemicals are transported in the water phase or adsorbed to the solid material and end up in the tailings dam. The process may also result in saturation and precipitation of thiosalts (sulfate containing precipitates).

Some potentially useful tools for waste characterization for the purpose of drainage quality prediction and management are discussed in the following clauses of this guidance, including:

5.3 Mineralogical analysis;

5.4 Physical and hydraulic properties;

5.5 Chemical Analysis (for solids and water).

3.4.5 Risk of spontaneous combustion

The risk of spontaneous combustion is mostly relevant for stacked waste rock at coal and lignite mines.

Despite extensive research having been carried out on spontaneous combustion of coal wastes, the process is still not fully understood (Banerjee, 1985; Pone et al, 2007). Spontaneous combustion takes place when the rate of heat generated by the oxidation of organic matter exceeds the rate of heat dissipation. The main driver of the process is the oxidation of organic matter, but other factors could also favour or hinder spontaneous combustion. It is likely to occur when fresh coal waste is suddenly exposed to the air for a variety of reasons (side cutting of stream, landslides, rapid erosion, mining or dumping, among others). The waste can also start to burn from forest fires or lightning.

Susceptibility to self-heating depends on the rank of coal, surface area exposed (particle size), moisture content, oxygen content, mineral content (especially pyrite), organic matter type, storage time and climatic conditions, mainly the maximum temperature and prevailing wind directions (Chesnokov, Shscerbakova, 1991).

In the case of stockpiles and waste piles the structure or shape of the piles and the level of compaction also play an important role in self-heating (van Krevelen, 1993). The risk of spontaneous combustion can be reduced or prevented by a reduction of coal content in the waste and by reducing the permeability to air in the disposed material through improvements in the production process, dump shaping and compaction of the waste in thin layers (Szczepanska and Twardowska, 2004).

Some potentially useful tools for waste characterization for the purpose of predicting spontaneous combustion are discussed in the following clauses of this guidance, including;

5.3 Mineralogical analysis;

5.4 Physical and hydraulic properties (surface area, porosity, moisture content, conductivity);

5.5 Chemical Analysis (for solids and water);

3.4.6 Naturally occurring radioactive materials (NORM)

The directive 2006/21/EC on the management of extractive wastes states that “while covering the management of waste from the extractive industries which may be radioactive, this Directive should not cover such aspects as are specific to radioactivity, which are a matter dealt with under the Treaty establishing the European atomic energy community (Euratom)”.

Sources of ionizing radiation include natural radiation sources from terrestrial origin and may, therefore, be present in extractive wastes. Even if the directive and this guidance do not deal with specific aspects of radioactivity, other characterization aspects of these materials are covered. There may be a need for extra precautions during sampling, storage and sample transport of wastes that may have naturally elevated levels of radioactivity.

The risk of nuclear radiation is mostly related to uranium mines, but is also relevant for any waste with sufficient radionuclide content (e.g. radium, radon, thorium or uranium). The potential for radiation impacts can readily be determined from a basic elemental analysis of the waste.

4 Supporting information

4.1 General

In addition to waste properties there is other information that may give valuable input for the evaluation of waste characteristics and for an assessment of potential short and long term impacts on soil, surface water and groundwater. For example, the COM decision 2009/360/EC specifies five categories of information:

- background information;
- geological background of deposit to be exploited;
- the waste and its intended handling;
- geotechnical behaviour of waste; and
- geochemical characteristics and behaviour of waste.

The last two points cover what is generally considered as waste characterization and are discussed throughout this document. The first three categories cover supporting information that is discussed in this clause. In addition, information from the site of disposal is briefly discussed.

For new operations a lot of the information specified in the following sections will already be documented as part of the exploration activities. For ongoing operations, additional background data will be available from the continuous registration of operational data. Existing information may also include results from previously performed waste characterization.

Examples of useful information gathered during exploration and production may be:

- Geological mapping, commonly three dimensional and often combined with mineralogical and geochemical analysis, will normally be an essential part of the exploration results; and
- Groundwater and surface water information may be collected and evaluated before and during the operation, e.g. to evaluate access of process water or to assess the need of pumping capacity for open pits and underground operations.

Any use of existing information should normally be complemented with a field visit to verify and supplement the information gathered.

4.2 Background information

Information on general background and objectives of the extractive operation is helpful in order to put the waste and the waste characterization process into context. The background information would typically include general information about:

- ongoing or planned prospecting, extraction, or processing activity;
- type and description of method of extraction and process applied/planned;
- the intended product.

These points are rather straightforward and will normally be readily available in permit applications or other existing documents.

4.3 Geological background of deposit to be exploited

4.3.1 General

Information under this heading is aiming at the identification of waste units that will be exposed by extraction and processing by providing relevant information on:

- nature of surrounding rocks, their chemistry and mineralogy, including hydrothermal alteration of mineralized rocks and barren rocks;
- style of mineralization and morphology of mineral deposits including mineralized rocks or rock-bearing mineralization;
- mineralization typology, including physical properties such as density, porosity, particle size distribution, water content, covering worked minerals, gangue minerals, hydrothermal newly-formed minerals;
- size and geometry of deposit; and
- weathering and supergene alteration from a chemical and mineralogical point of view.

However, not all of this information is relevant for all types of operations or extractive industries. The level of detail of the information to be gathered should be adapted to the type of waste, the potential environmental risk, and the intended waste facility.

The information listed above is discussed in the following clauses.

4.3.2 Surrounding geology

Information about the surrounding geology (including hydrology) will often contribute to the understanding of the potential environmental impacts of the extractive waste and is commonly an integral part of both the exploration and production phases, but not for all sectors.

For the production planning, as well as for the waste management plan, it is important to establish the borders between the ore and the waste. In cases where the mineralization is defined by distinct rock types the borders are commonly easy to establish. In cases with a rock alteration type mineralization, like in disseminated deposits, the border is often defined as a cut-off grade based on production cost and product price. In these cases, the border may change over time.

Geological information may also be documented for the evaluation of the stability of mine tunnels and pit walls. This information is in some cases useful for the evaluation of the geotechnical stability of the waste.

Draw-down of the water table due to pumping during the operation may expose rocks and soils that were previously below the water table to air. If these rocks contain sulfides, the lowering of the water table may result in sulfide oxidation, acidic drainage and possible generation of secondary iron sulfate minerals. An understanding of these processes may provide valuable input to the waste characterization as the surrounding rock in many cases presents the same properties as the waste. In some cases, the surrounding rock is in fact the future waste.

Information regarding the surrounding geology may include:

- mineralogy and geochemical properties of the surrounding rocks;
- hydraulic properties;

- groundwater levels.

To carry out new investigations to obtain this type of information could be quite costly. However, for those operations where information about the surrounding geology will be important for the characterization and management of wastes this information will most likely already be documented.

4.3.3 Nature of deposit

The nature of the deposit together with the value of the product will to a large extent determine which extraction method is feasible (quarry, open pit, underground, block caving etc.). One key element of a description of the nature of the deposit would be the structure of the ore. i.e. if the deposit is a massive type, stock-work type, vein type or disseminated.

The nature of the deposit and the extraction method will have a significant impact on the types and volumes of waste produced. A quarry typically produces relatively small amounts of waste per tonne of product. Underground mining commonly means less waste rock produced per tonne of ore than an open pit. A massive deposit commonly means less tailings produced per tonne of product than a stock work or a disseminated deposit. However, in this context it should be mentioned that the economics of an open pit operation may mean a more efficient recovery of the resource overall through the inclusion of low grade material, i.e. a lower cut-off grade.

4.3.4 Mineral deposit

Knowing the nature of the mineral deposit is essential to the owner/operator in order to plan an efficient and profitable operation. The information about the mineral deposit may include:

- mineralization typology;

- textural descriptions; lithology, structural features, depth of oxidation;

- whole rock geochemistry and elements distribution in mineral phases;

- geochemical properties (e.g. reducing capacity, degradable organic matter content, thermodynamic stability, acid forming potential and acid neutralization potential);

- mineralogy (ore minerals, gangue minerals, hydrothermal newly-formed minerals etc.);

- physical properties such as density, porosity, particle size distribution, water content.

Relevant parts of the information described above may be obtained from existing plans and reports and be included as part of the waste characterization. The level of detail should be adapted to the mineralization in question and proportional to the size of the operation and risks to be managed.

4.3.5 Size and geometry

Information on the size and geometry of the ore deposit are important in the development of plans for the operation. The plan gives information on the estimated volume of ore and waste rock together with information on when different types of ore and waste rock will be extracted. This information is essential when developing the waste management plan.

The information about waste volumes over the planned lifetime of the operation needs to be combined with information about waste characteristics as discussed elsewhere in this document. This implies that during exploration not only the mineral resource should be analysed but also the volumes and behaviour of the future extractive waste.

In recent years, most resource calculations performed during exploration and in feasibility studies used a specialized computer package that deals with 3D data. The “block model” of the future operation can be considered as a 3D database of the potential mining units, based on which the definition of ore or waste is made. The block model is based on all relevant and available test results. Assuming that the environmentally critical and relevant parameters (acid producing capacity, mineralogy, leachability etc.) of future waste blocks are defined with a certain probability during the detailed exploration phases, this information can be fed into the block model and serve as a base for the waste management plan (an example of a cross section, 2D, waste model is shown in Figure 6).

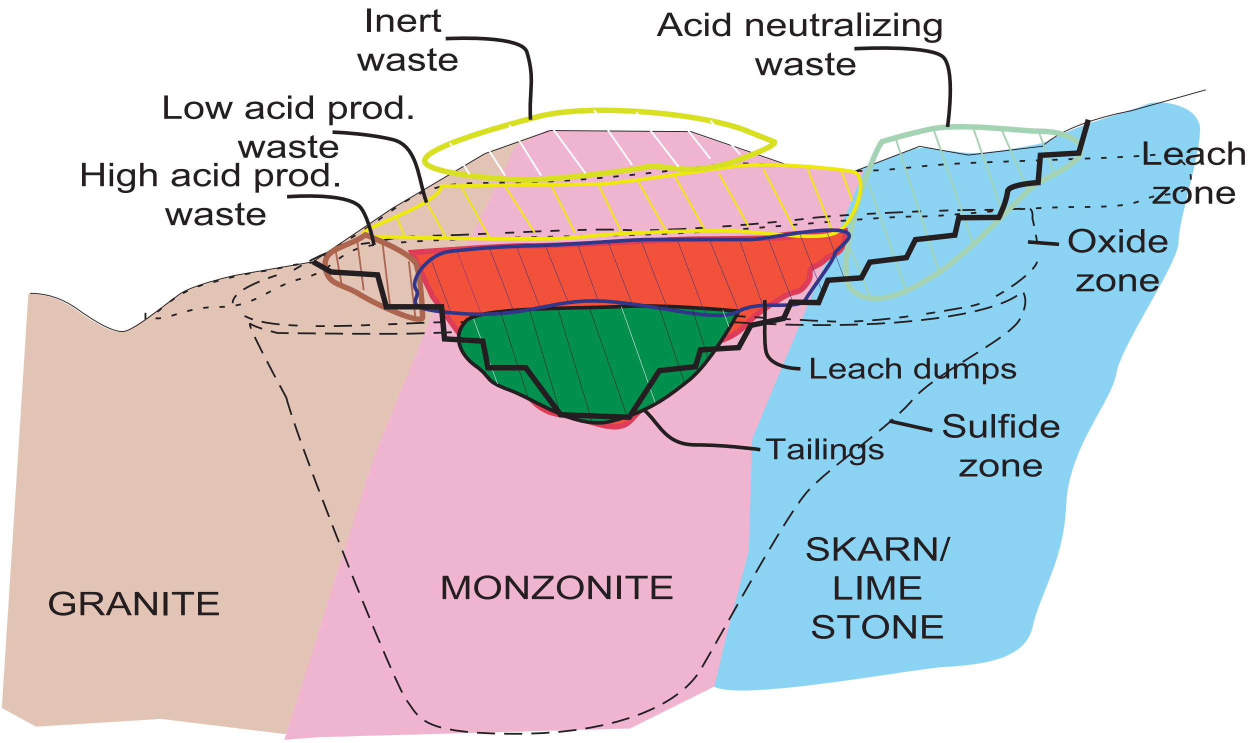

Figure 6 — Waste model linked to geology and production types are illustrated for a hypothetical porphyry copper deposit. The waste model illustrates different types of waste rocks in addition to ore that is producing tailings and ore that is producing copper leach rocks (Walder, 2001)

4.3.6 Weathering stage of deposit

The significance of weathering primarily relates to mineral deposits containing sulfide minerals with the potential to form A/NRD. Defining the weathering and alteration of a deposit is commonly a part of the information gathered during exploration and details about these processes can be found in text books (e.g. Robb, 2007; Barnes, 1989).

A sulfidic metal mineralization is commonly divided into:

— a hypogene mineralization, which relates to the original mineral assemblage, i.e. the mineral assemblage formed during the primary mineral forming processes.

— a supergene mineralization, which relates to mineral assemblage that has formed due to near surface processes (weathering processes).

The supergene processes do not only relate to metal ore deposits but to weathering in general.

In most of the case the supergene alteration can be enhanced by the hydrothermal alteration that develops a pervasive alteration increasing the porosity, changing the chemistry of the parental rock and leading to the deposition of newly formed minerals (e.g. pyrite, biotite), or the replacement of primary minerals by clay.

The supergene alteration (commonly associated with sulfide containing deposits) takes place when the mineralization is exposed to oxidizing conditions. This may occur if there is erosion of the overlying rocks and the water table is lowered within the sulphide mineralization. The sulfide minerals are then likely to oxidize with a transport of elements as shown in Figure 7. The processes illustrated here are natural ARD processes; however, due to limited access to oxygen, the processes may be relatively slow.

In some cases, the supergene alteration is crucial for developing an economic viable deposit where the supergene processes transport valuable metals downward and re-precipitate them on top of primary minerals with the same valuable metals. Understanding the processes of the ore deposit can often give an indication of how waste material from that deposit will react.

The hydrothermal alteration zones may also be found in deposits that do not contain sulfides. However, it is commonly less important to understand the hydrothermal alteration zones for those deposits. Sedimentary deposits such as limestone, sand and gravel will not have the hydrothermal alteration zones.

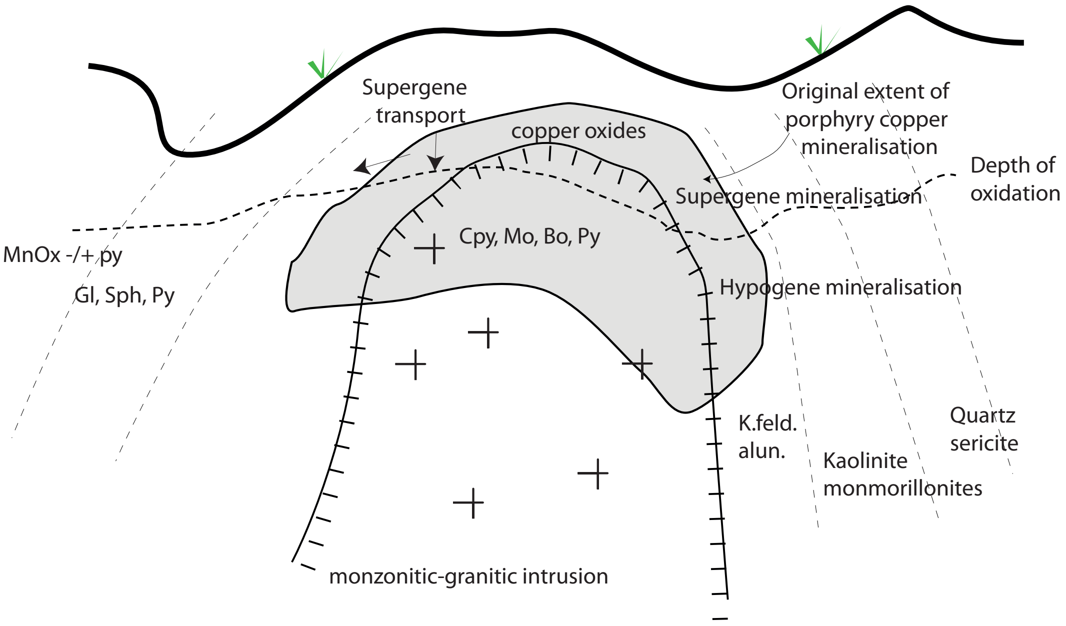

Figure 7 — Mineral distribution in and around a hypothetical porphyry copper deposit. The dotted semi-vertical lines represent hydrothermal alteration zonation, while the horizontal line represents the bottom of the supergene-oxidation processes. Cpy-chalcopyrite $FeCuS_2$), Py-pyrite (FeS_2), Bo-bornite (Cu_5FeS_4), Mo-molybdenite (MoS_2), Sph-sphalerite (ZnS), Gl-galena (PbS), MnOx-manganese oxides (Walder, 2003)

4.4 The waste and its intended handling

Information about the origin of the waste within the extraction site and the processes generating such waste could give valuable supporting information when evaluating waste characteristics.

A practical approach could be to document predicted quantities (annual and total) of site specific waste categories based on two factors: mineralogical characteristics relative to the mineralization and the geology of the extraction site and the type of process the waste has gone through before being deposited. For example:

— waste rock, type x, without processing;

- waste rock, type y, after crushing and sorting;
- tailings from ore type a, after froth flotation;
- tailings from ore type b, after gravity separation;
- tailings from ore type c, after leaching.

Waste handling includes transport, processing and disposal. It is recommended to document planned or existing waste transportation systems, e.g. transport by trucks, conveyer belts or pipe lines.

Mineral processing may involve the use of chemical substances, e.g. frothers, suppressing agents, flocculants and leaching agents. These substances may end up in the waste stream and it is therefore recommended that all process chemicals are listed.

In addition, under EU legislation, information should be provided on the classification of the waste according to the Commission Decision 2000/532/EC, including the hazardous properties (the so called European Waste Catalogue).

The COM decision also requires the waste characterization to include information on "type of waste facility, final form of exposure of the waste and method of deposition of the waste into the facility". It should be noted that for new operations, even though preliminary plans and information may be available at an early stage, these are management decisions that should be made based on the result of the waste characterization.

4.5 Baseline data for the site of disposal

For future waste facilities or expansions, information regarding baseline properties of the intended disposal site (or alternative sites where relevant) may provide useful supporting information. The appropriate level of detail will depend on the type of waste and the disposal scenario envisioned. Both normal and exceptional site conditions which may influence the properties of the future waste facility and the behaviour of the deposited waste could be of interest.

Relevant information regarding potential disposal sites may include:

- Site topography, hydrology and geotechnical data, e.g.:
  - water balance and degree of water saturation;
  - dimensions of the planned waste facility;
  - physical properties of the foundation soils and bedrock.
- Geochemical conditions, e.g.:
  - geochemistry of foundation soils and bedrock;
  - groundwater chemistry.

5 Characterization tools

5.1 General

The most common methods used for mineralogical, geotechnical and geochemical analysis in extractive industries are presented in this clause, together with a discussion of their applicability.

The methods described should be seen as a set of tools. Which tools to use in a specific case will depend on characterization objectives, type of operation, available disposal scenarios and other site specific conditions.

5.2 Sampling

When available data is insufficient for meeting the characterization objectives, a plan for further sampling and testing may need to be developed. A field visit will normally be part of developing a sampling plan.

A specific sampling guideline for the characterization of waste from extractive industries has been developed (CEN/TR 16365). This guideline builds upon EN 14899, “Sampling of Waste Materials – Framework for the preparation and application of a Sampling Plan”. The sampling guideline is focusing on the development of sampling plans but does also discuss some practical aspects such as sampling equipment, storage and transport. According to the sampling guideline a plan for characterization of extractive waste should normally cover the aspects listed in Table 2.

Table 2 — List of aspects to be covered in a sampling plan

Aspect	Explanation
Identification of stakeholders	List with contact information of all stakeholders
Identify general objectives	General objectives as described in Clause 2,
Background information	Information of the process type, waste types, known geology, mineralogy, previously performed characterization etc.
Specific objectives	Detailed objectives such as prediction of drainage quality, ARD potential, mineralogy
Determine generic level of testing	Sampling for screening, detailed characterization, confirmation etc.
Identify constituents and analytical methods	Elements/constituents of concern e.g. total metal content, pyrite content, and analytical methods to determine these constituents
Identify health and safety precautions	Health and safety issues for the sampler and the storage and transport of the samples
Select sampling approach	Judgmental, unbiased, etc.
Identify sampling techniques	Sampling from drill core, auger, shovel etc.
Sub-sampling	Splitting system of samples after collection for reduction of volume/mass
Sample preparation	Drying, splitting for different analysis, storage requirements
Transport sample	Transport system to the lab and transport documentation
Document sampling plan and produce instructions for the sampler	Details on sampling procedure for the sampler
Produce a field sample record	List of information to be included in the field sample record
Complete sample record and document changes	List deviations to the sampling plan and reasons for the deviation

5.3 Mineralogy

5.3.1 General

Knowledge of the mineral composition and mineral chemistry may be essential to interpret data on chemical composition or leach tests and to predict short and long-term drainage water quality from the waste material generated in the extractive industry. The mineralogy may further be of significance when interpreting acid production and buffering potential based on acid-base accounting.

Mineralogy in this context provides supporting information for the characterization of the waste material. Mineralogical information together with physico-chemical conditions may e.g. give indications about the occurrence of weathering processes such as:

- oxidation (sulfides);
- dissolution (carbonates, sulfates);
- hydrolysis (silicates);
- sorption;
- mineral stability/reaction rates.

This information in combination with geochemical data and leach test results can be used to evaluate leaching potential, metal transport, and attenuation.

Some key aspects of mineralogy and A/NRD processes are discussed in Annex G.

The following clauses discuss different approaches to obtaining relevant mineralogical information.

5.3.2 Analytical methods

There are many different mineralogical analyses that can be performed and their usefulness varies with the type of issues that need to be solved.

The mineral sciences, especially in the last few decades, have become very equipment-focused. The analytical methods are being developed towards high accuracy determination of small quantities. A pitfall to be avoided is to get too deep into details, losing sight of the integrated assessment that is usually what is sought.

Microscopic analysis is often the basic form of mineralogy analysis. The microscopy analysis can give information on type of minerals and if these minerals have undergone any alteration/weathering.

X-ray diffraction, XRD, methods are commonly used to identify minerals, but the method has detection limits of 2 wt % to 3 wt % (weight %, commonly used for mass fraction within the sector). This means that the method may not detect minerals that could be of concern even at lower concentrations. XRD methods are only semi quantitative and additional chemical analyses are necessary to quantitatively identify minerals using XRD (Rietveld method).

An electron probe micro-analyzer, EPMA, is a micro-beam instrument used primarily for the in situ chemical analysis of minute solid samples. EPMA is also informally called an electron microprobe, or just probe. The primary importance of an EPMA is the ability to acquire precise, quantitative elemental analyses at very small "spot" sizes (as little as 1 µm to 2 µm), primarily by wavelength-dispersive spectroscopy (WDS). The spatial scale of analysis, combined with the ability to create detailed images of the sample, makes it possible to analyze geological materials in situ and to resolve complex chemical variations within single mineral or amorphous phases.

The core concept of the Rietveld method as well as other full profile refinement methods is the comparison of the measured diffraction profile with a calculated profile. The latter one is based on calculated and refined crystal structures where certain elements can be substituted by others. Chemical information is therefore needed:

- to have the real composition of the mineral phases, which can be obtained by EPMA;
- to compare the measured whole rock composition with the calculated composition obtained from the quantitative XRD results.

In general the more additional information (microscopy, EPMA, whole rock chemistry) you have about the rock the better your refinement result will be.

The microscopic and XRD analysis give no information about trace element concentration (often substituting main elements in the mineral structures), which may be important for evaluating trace element sources and drainage chemistry. There are many different techniques that can be used to identify the mineral chemistry (e.g. scanning electrone microprobe (SEM), X-ray photoelectron spectroscopy (XPS)).

Mineralogical analysis is a stepwise procedure. How far to go will depend on information needs, time available and costs. See Figure 8 below.

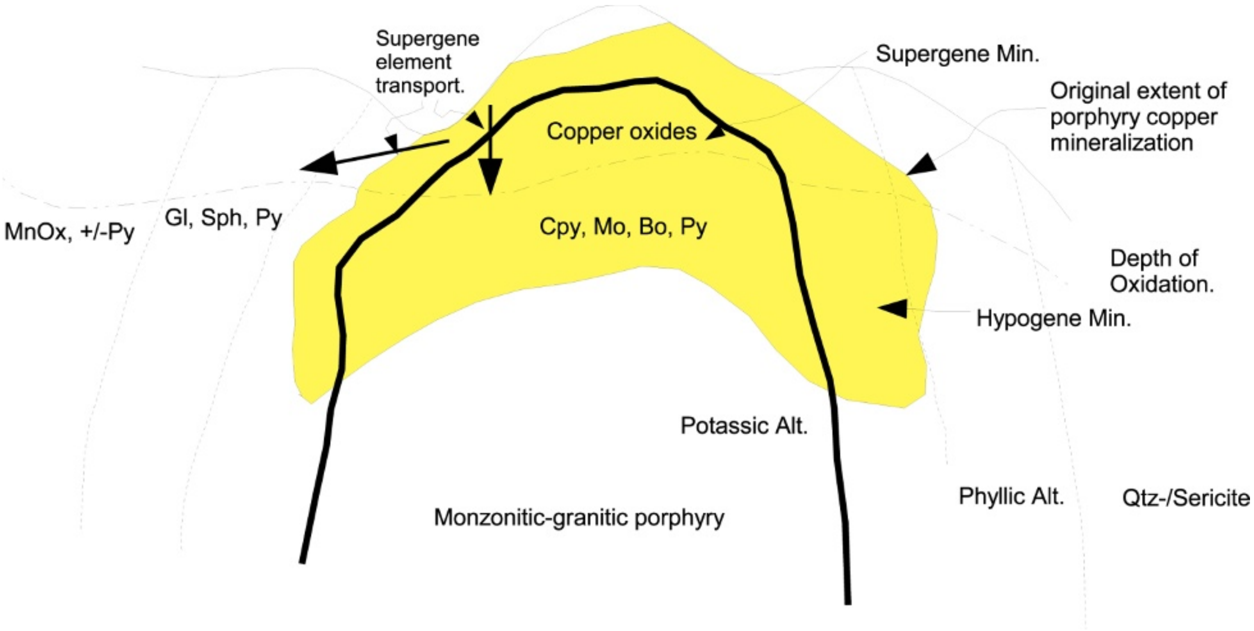

Figure 8 — Stepwise approach to mineralogical analysis

The most important analytical methods are discussed briefly in Annex E. These are methods which can provide reliable information on bulk mineralogical composition and especially on specific questions of minerals heterogeneity and alteration processes. The annex gives background information on capabilities and limitations of the analytical methods to facilitate dialogue with specialists.

5.3.3 Texture

The texture of the waste material may be important to assess when characterizing the waste material, because the texture of the rock gives information about the actual availability of the minerals to leaching and to generation and neutralization of acidity. If the reactive minerals are fine grained this will lead to a large surface area when crushed, i.e. high availability. If the reactive minerals occur only encapsulated within non-reactive minerals they may remain effectively immobilized.

It may also be relevant to assess the degree of liberation; i.e. if the surfaces of the mineral grains are available for surface reactions. This is relevant for neutralizing minerals, acid producing minerals, or leachable minerals.

Analysis of the texture of the waste material is commonly performed by classifying the texture of hand specimens and from microscopic analysis of polished thin sections (see Annex G).

5.4 Physical and hydraulic properties

5.4.1 General

Physical and hydraulic properties of extractive waste are important in the context of dam or slope stability assessment (as discussed in 3.3.1) and for the assessment of water balance and hydrology of a disposal site. Physical properties may impact the geochemical behaviour of the waste as well as being impacted by chemical reactions taking place.

Every mineral deposit is more or less unique with regard to waste properties as well as to its geological, geographical, hydrological, climatic etc. conditions. This in turn leads to site specific designs of waste disposal facilities. What information on physical properties is needed and relevant will have to be evaluated based on characterization objectives and site specific conditions. A complicating factor is that particle size and many other physical properties of the waste cannot be assessed in detail prior to extraction, which means that the initial characterization will have to be followed up during operation.

This clause gives an introduction to the general scope of geotechnical investigations and the reasons for their importance.

5.4.2 Overview of types of geotechnical investigations

Geotechnical investigations may be divided into laboratory and in situ investigations.

Laboratory characterization and testing of the waste materials may measure properties such as: compressibility, shear strength, angle of friction, grain size distribution, density (bulk density and specific weight), plasticity, fracturing (if appropriate), liquefaction potential, permeability and erosion potential.

While laboratory investigations in general make more accurate measurements possible, the average sample size is generally small. Therefore, an accurate sampling strategy to ensure representative samples is crucial. Up-scaling of the results should always be done with care.

In-situ tests may in many cases be less accurate, but they are of a more “integrating” character. This means that their results are generally valid for a larger volume. It should be noted that many in-situ tests will only give indirect information about the investigated material (e.g. in-situ measurements of the frictional forces or the peak resistance of the waste material may be used to get information on shear strength or angle of internal friction).

In-situ tests are in principle only applicable to waste already present in existing dumps and tailings storage facilities and their relevance for initial testing in the planning stage is limited.

Already this short introduction to field and laboratory investigations indicates that there is no perfect methodology for the characterization of physical properties of the waste material. There are different, more or less adequate, investigation strategies that all include some level of inaccuracy that can only be reduced using well educated and highly experienced professionals.

Geotechnical investigations of extractive wastes and waste facilities may include site specific information (e.g. hydrogeological, climate data, seismicity, topography, tailings and waste dump seepage) that are part of the general background information of the site, see Clause 4.

5.4.3 Test methods for physical and hydraulic properties

Different sets of testing may be considered for physical characterization of extractive wastes. These include:

- Physical characterization of the solid phase of tailings, which would normally include 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.

- Physical characterization of tailings used for dam construction, which would normally require additional physical characterization relevant for dam design. Additional tests that may be considered for this purpose include particle specific weight; natural water content; bulk density; Proctor compaction curves; variation of pore pressures; and swelling potential.

- Analysis of waste in existing dumps, which may also comprise estimation of the degree of aggregation; degree of compaction; amount of natural fines and amount of fines after compaction; weathering grade; initial and final moisture content due to drainage; effective cohesion; and angle of internal friction (see 5.4.4 below).

Compressibility and frictional behaviour are key parameters for the assessment of the behaviour of tailings and waste rock in impoundments and heaps. The internal friction is a key element of physical stability and is in turn strongly linked to the level of compaction and dependent on other processes taking place. Several factors are important to understand the compressibility and self-compaction properties of the waste materials, including: the overall compressibility; its dependence on moisture content; the possible migration velocity of moisture (seepage velocity); and the grain-size distribution.

It is important to consider the strong interdependence between physical and geochemical properties. While the physical properties of the waste may have a significant impact on the long term geochemical performance, chemical weathering of the waste will lead to changes of the physical properties.

Annex C briefly describes test methods for the categories of physical and hydraulic properties/parameters listed below (for each of the methods discussed in Annex C, existing international standards are listed).

- soil index properties including grain size distribution; moisture content; and plastic behaviour;

- density and compatibility;

- shear strength and bearing capacity;

- factors affecting permeability and subsurface flow (porosity, void ratio, saturation, hydraulic conductivity).

Some additional information on geotechnical test methods can be found e.g. in Annex 4 of the Mine waste BAT document (Reference Document on BAT, 2004).

5.4.4 In situ investigations of physical and hydraulic properties of deposited waste

Waste characterization at existing facilities may include an investigation of in situ physical and hydraulic properties of the waste and the resulting hydrogeological conditions in the waste facility. This may be important input e.g. to risk assessments of existing facilities or the development of closure plans.

Drilling is one way to get direct information. From boreholes disturbed samples can be collected or undisturbed samples can be taken using core samplers. The stratigraphy of the impoundment material may be described using the bore log. The disturbed samples are used to determine the Atterberg-limits, Proctor type, bulk density and compressibility. From these values bearing capacity, plastic and elastic soil parameter values can be estimated. Undisturbed samples can be used for triaxial or uniaxial tests to determine internal friction and cohesion of the materials. The mentioned properties of loose soils or of disturbed samples can also be investigated using shear tests. When using boring techniques the need to seal boreholes to avoid ingress of air and/or water should always be assessed. In specific cases it may be recommended to avoid boring.

Besides boring, Cone Penetrometer Testing and Standard Penetrometer Testing can also be used to characterize larger areas. Since they are quick and rather cheap testing methods they can be used to refine the information from the borehole investigations. Moreover, indirect information on bearing capacity, soil moisture distribution, etc. vs. depth can be recorded by special sensors.

To evaluate the hydrogeological conditions in operating tailings dams, monitoring wells can be established and the water levels regularly or continuously recorded. From the values the hydraulic gradient and the water flow direction can be determined. Wells or bore holes can be used to perform pumping tests (for highly permeable formations) or slug tests (for low permeability rocks) in order to determine the hydraulic conductivity (using steady state or transient data) and storage coefficient and specific yield (using transient data records).

5.5 Chemical composition

5.5.1 General

There is a variety of chemical methods that can be used to evaluate the chemical composition of water and solids. The solids can be analysed directly using e.g. atomic absorption with graphite furnace or subject to digestion followed by an analysis of the dissolved substances. There are different options for digestion and for the chemical analysis of the solutions. Which method to choose partly depends on the objective of the study, on analytical methods chosen in earlier studies (to produce comparable data) and on the laboratory resources available. There is a brief introduction to these methods in the following clauses. There are numerous publications on chemical analysis and Annex B lists applicability of CEN and ISO standardized tests methods for waste from extractive industries.

5.5.2 Digestion methods for inorganic substances

Before performing the analysis of elements by chemical analysis the solid waste material has to be digested resulting in an acidic aqueous solution. Generally, there are two approaches: either total digestion or a partial extraction. In environmental analysis often aqua regia is used, but (contrary to what is often attributed) this is a partial extraction and not a full digestion. Although aqua regia is a very strong acid, it is not able to fully digest silicates; therefore, all elements that are bound to that matrix are not digested and will consequently not be analysed. The digestion of silicates requires hydrofluoric acid which is used for total digestion. Alkaline fusion methods are applied to major components and to a selection of anions like chloride (Cl^-), sulfate (SO_4^{2-}) and fluoride (F^-), see Annex B.

5.5.3 Analysis of major, minor and trace elements

Following digestion, elements of interest are measured in the acidic aqueous solutions obtained using different detection methods. The choice of method depends on the specific element or elements and the range of concentrations of interest. When a large number of elements have to be determined, the first choice is inductively coupled plasma-atomic emission spectrometry (ICP-AES) or inductively coupled plasma- mass spectrometry (ICP-MS). These methods allow the simultaneous determination of: aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, lithium, magnesium, manganese, mercury, molybdenum, nickel, phosphorus, potassium, rubidium, selenium, silver, silicon, sodium, strontium, sulfur, tin, titanium, vanadium and zinc.

Direct analysis using flame atomic absorption spectrometry (F-AAS) or graphite furnace atomic absorption spectrometry (GF-AAS) is less frequently used nowadays These methods are mostly used for single element determination rather than for multi-element analysis, however, new instruments can also perform multi-element analysis. AAS requires larger samples than ICP. Mercury is an element that requires special analytical techniques due to its usually low concentration. Applicable are cold-vapour atomic spectrometry (CV-AAS) or cold-vapour atomic fluorescence spectrometry (CV-AFS). Low concentrations of arsenic, antimony, and selenium are measured by hydride-generation atomic absorption spectrometry.

X-ray fluorescence (XRF) is an instrumental method for total composition without digestion. The sample is either fused with borate or mixed with wax to obtain a tablet for further analysis. This method will also give the total content of elements in the sample.

Field XRF instruments can give a rapid analysis and it requires no pre-treatment. The detection limit, however, is higher than for conventional XRF.

5.5.4 Chemical speciation

So far, element concentrations have been determined without regard to chemical speciation. Concentrations are given as pure elements while in fact they are almost always bound in chemical compounds with different states of oxidation. This may be significant from a toxicological point of view. For example, the element chromium is found as chromium (III) as well as chromium (VI). While chromium (III) is benign, the latter is toxic at low concentrations and requires a dedicated analytical technique.

Direct methods for quantifying an element in a specific chemical form in a mineral are available only for a few substances. However, knowing in which mineral a substance occurs will, in most cases, indicate its chemical form. Mineralogical speciation (i.e. in what minerals each element is located in a rock) can be performed as described in 5.3. This will usually give a good indication for elements in higher concentrations, while trace elements can be hidden as adsorbed, ion exchanged or substituted atoms, or occur in the form of small quantities of separate minerals. The combination of mineralogical data, water quality data and modelling would be the most commonly applied approach to assess speciation (Lichtner et. al, 1996; Moritz et al, 2009).

5.5.5 Sulfur (total, sulfate, and sulfide)

For many wastes from the extractive industries the content of sulfur is of interest as it may lead to production of acid/neutral drainage. Sulfur can be found as sulfide or disulfide (e.g. pyrite), as sulfate (e.g. calcium sulfate = gypsum), or as organic sulfur. The latter would usually not be found in extractive wastes, the same holds for elementary sulfur. While most sulfides will contribute to acid production, sulfate and elementary sulfur will not.

Usually, the total content of sulfur is determined, which is sufficient for many applications. According to EN 15875, the preferred methods for sulfur determination are bomb combustion according to EN 14582 or high temperature combustion according to ISO 15178. When speciation is of interest, instead of total sulfur a more time and labour consuming analytical method has to be applied (see Annex F and EN 15875:2011, Annex C).

5.6 Determination of acid generation potential

5.6.1 General

Acid/neutral rock drainage (A/NRD) is the main long term environmental issue for the metal and coal mines sectors of the extractive industries. Different A/NRD prediction methods that are being used by the industry around the world include static testing and kinetic testing.

The static tests are designed for the single purpose of measuring the extractive waste’s capacities for producing acidity and its acid neutralization capacity. Accordingly, static tests are commonly used for screening purposes and to provide an answer to whether the extractive waste material has a potential to be an acid producer or an acid neutralizer. The static tests focus on the intrinsic properties of the material. They do not provide any information on reaction rates or release rates of weathering products.

5.6.2 Paste pH

Paste pH is the most basic and simple static test. While it can be useful to indicate ongoing acidification of waste that has been exposed to air and water for a few years it does not give any useful information for fresh or un-weathered waste. The method is usually performed in the field, but laboratories will also perform the test. A pH result below neutral indicates that acid production has occurred.

5.6.3 Acid-base accounting

Acid-base accounting (ABA) is the most common type of static tests. They are all designed to quantify the total acid producing potential and acid neutralizing potential of the material tested. However, they do not give any information on the rate of acid production or neutralization.

ABA-tests quantitatively balance the sulfur or sulfide-sulfur content (acid potential, AP) with the neutralization potential (NP) of carbonates and other alkaline material.

The quantitative balance is commonly expressed as the neutralization potential ratio (NPR),

$$\mathrm{NPR} = \frac{\mathrm{NP}}{\mathrm{AP}} \tag{1}$$

or as a difference between the acid potential (AP) and the neutralization potential of the host rock (NP), the net neutralization potential (NNP)

$$\mathrm{NNP} = \mathrm{NP} - \mathrm{AP} \tag{2}$$

The static test described in EN 15875 has been developed as part of the implementation of the Directive on waste from extractive industries and is the recommended static test procedure within Europe.

There are several other static tests that are used internationally. E.g.:

- acid-base accounting (ABA) the Sobek method (Sobek et al, 1978);
- modified Sobek (Lawrence and Wang, 1996).

In most methods (including EN 15875) the acid potential is calculated based on sulfur, or sulfide, content (see 5.5.5).

The net acid generation (NAG) test (Miller et al, 1997) determines resulting acid generation directly and is often used as a complement to ABA to resolve unclear cases.

Although the static tests share some common features the different protocols may produce different results for the same sample . Different mineralogy will manifest itself in different sensitivity to the choice of protocol parameters as described in EN 15875.

The protocol for EN 15875 specifies acid addition on the basis of the carbonate content (EN 13137) (Kaartinen and Wahlström, 2009) which is an upgrade of the most used ABA methods. In the standard ABA (Sobek et al, 1978), the modified ABA (Lawrence and Wang, 1996) and with peroxide siderite correction (Skousen, 1997) a fizz test is used for determination of the amount and molarity of hydrochloric acid added to the sample. Some concern has been raised regarding the rationale and appropriateness of this procedure (see for example Jambor et al, 2007).

As part of the standardization work, an evaluation of the different test methods for sulfur determination used for evaluating acid producing potential has been performed (see 5.5.5 and Annex F).

5.6.4 Kinetic testing

Kinetic tests are tests set up to allow for naturally occurring reactions to take place. These reactions will commonly change the behaviour of the tested material over time. The concept of kinetic tests could include any test that allows for, and measures, changes over time. Commonly kinetic testing refers specifically to tests run to evaluate acid generation behaviour of sulfidic wastes, i.e. to quantify reaction rates for acid producing and buffering reactions and to evaluate if the material actually will go acidic or not. The latter is the definition applied in this document. The more standardized kinetic test methods (ASTM 2001: USEPA 1978, Morin and Hutt, 2002) are designed to evaluate/calculate the maximum sulfide oxidation rate relative to neutralizing reactions and not to simulate waste behaviour under field conditions. They are not set up to provide information needed to evaluate leaching rates (Lapakko, 2004). Adjustments to the standardized methods are being used to evaluate leaching rates (Price, 2009), adjustments that take into account more site specific conditions such as airflow, grain size and climate. Information on leaching rates and drainage chemistry is also achieved from scaled up experiments and field tests.

Kinetic testing is briefly introduced in this clause. For more detailed information, see the kinetic test guideline CEN/TR 16363, and references therein.

In kinetic tests, the release of elements is assumed to be driven by sulfide oxidation and consequent neutralising reactions. Most commonly the kinetic tests are set up to allow the products from the mineral oxidation and dissolution to be transported away from the reactants in such a way that they are not a hindrance to further reaction, e.g. by forming a coating on the mineral surfaces. This is one of the reasons why the leach rate in the test is not applicable to field conditions.

The most well-known kinetic test for acid rock drainage is the humidity cell (Caruccio, 1968; ASTM D5744-96). This is a simulation test where the measured weathering and release is a combined effect of operation and various parameters, such as the exposed surface of the sample.

The humidity cell is intentionally operated to accelerate sulfide mineral oxidation and will predict if a given sample will produce acid leachate, but gives no information on when acid leachate may appear for the boundary conditions encountered in the field (EIPPCB, 2004). The accelerated rate of oxidation and acid production will also result in an accelerated rate of generation of oxidation products such as dissolved metals and/or precipitated metal compounds.

A variety of column tests have been used to characterize acid rock drainage (Lapakko, 2003). Column experiments can be used to determine the kinetic behaviour in tailings when exposed to atmospheric weathering (sub-aerial storage), or stored under water cover (sub-aqueous storage). A large number of column test protocols have been reported, representing a large variety with respect to material particle size, sample mass, water infiltration or flow rate and degree of oxygenation.

In-field reaction and leaching tests are used for the prediction of acid generation and drainage chemistry. This group of tests includes rainfall simulated leach tests performed in the field on waste rocks or rock surfaces, and pilot scale tests under controlled field conditions.

Lysimeter and field scale testing may be the type of testing that is closest to practice, as it considers natural exposure conditions. This type of test includes pilot scale tests with exposure of waste rock or exposed rock surfaces to the atmosphere (O_2) and local weather conditions or imposed rainfall regimes. However, these types of tests are costly and have to be run for a long time (years) before meaningful results are obtained.

5.7 Leaching behaviour and leaching tests

Constituent leaching from waste materials is a natural part of an ARD evaluation, however, leaching can also be of environmental significance where there are no sulfur, sulfide or iron-sulfide minerals. E.g., wastes from the extractive industry can be very alkaline where strong bases are used in processing (e.g. alumina refining or gold leaching) or very salty (salt mines). Leaching tests may in principle be applicable to any type of residue from the extractive industry, whether appropriate or not will depend on whether they present an efficient way to produce the information sought in a specific case.

The percentage of the content of a specific constituent that can be leached from a waste at a relevant pH is a measure of the potential leachability. The leachability varies strongly for each material and each element. It may range from close to 100 % to 0,001 % demonstrating that total composition is a poor measure to predict the potential environmental impact of an element.

There are a large number of standardized leaching tests available. The characterization leaching tests comprise methods for measuring (i) solid-aqueous partitioning as a function of pH, (ii) solid-aqueous partitioning as a function of liquid to solid ratio (L/S), and (iii) mass transfer rates for monolithic or compacted granular materials. Most of these tests have been developed for the characterization of waste, but not for extractive waste specifically. The preferred and most commonly applied leaching test for waste characterization is the CEN/TS 14405 column leaching test.

The column leaching test (CEN/TS 14405) is an up flow percolation test designed to resemble common percolation scenarios. The liquid to solid ratio is related to a time scale through the infiltration rate, density and height of the material. Through modelling, the effect of preferential flow can be quantified. The first eluate from a column test reflects the pore water conditions of the material considered. Control measures can be taken for testing of materials that are sensitive to oxidation to avoid changes in initial conditions.

It should be noted that, in the case of oxidising waste material, this test does not capture the change in leachability over time.

For more information on leaching tests, see Annex D.

5.8 Field Investigations

In the context of waste characterization, investigations of existing extractive waste facilities may be part of the monitoring/confirmation that is done to follow up on an earlier comprehensive waste characterization and predictions of future drainage quality or it may be part of characterizing the waste in closed facilities, with no or insufficient existing information.

Investigations of existing facilities may be seen as the ideal field scale test set up. Characterization and predictions from the early planning stages of an operation all have uncertainties based on issues of representativeness and on a number of assumptions of future conditions. In the existing facility, it may be possible to evaluate the sub-surface hydrogeological conditions, geotechnical conditions, and geochemical processes, much depending on how long the facility has been in place. This information may then be used for the evaluation and verification of models and predictions.

While most of the characterization tools discussed in this guideline in principle may be used on samples from existing facilities, the tests in 5.4.4 (in situ investigations of physical properties) and some of the tests in 5.6.4 (acid production potential – kinetic testing) are in fact only applicable to existing facilities.

Besides some of the geotechnical tests that are performed in the field, there are also field methods available for mineralogical and chemical tests that may be used when relevant:

- bulk chemistry performed by portable XRF (giving lower detection limit than laboratory equipment, but rapid result);
- direct measurements of soil pH using special probes;
- mineralogy observation in hand specimens;
- near infrared spectrometer (IRS);
- adding chemicals to observe reactions indicating mineral types;
- the advanced space borne thermal emission and reflection radiometer (ASTER) for evaluating mineral alteration products on tailings and waste rocks;
- thermal image camera to evaluate heat generation (sulfide oxidation).

There are many similarities between tailings and waste rock characterization. However, with regard to field investigations there are also many differences. Annex I gives some further details on field investigations of tailings storage facilities and waste rock dumps.

6 Evaluation and interpretation

6.1 General

In the early stages of the characterization different objectives are specified. To meet those objectives the data produced through the application of different test methods will have to be evaluated and interpreted.

This clause will discuss interpretation methods for the characterization of the waste material by discussing some of the contexts within which characterization data may need to be applied. However, it does not cover information on how to apply the results of characterization, 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 throughout this guidance document.

The extractive industry covers many different sectors with very different waste categories and characterization may be carried out with many different objectives. For this reason, a clause on evaluation and interpretation of characterization data cannot be prescriptive and provide generally applicable instructions.

Mineralogical analysis is usually the first analysis performed (see Figure 2) in order to identify potential environmental impacts from the waste material due to weathering and leaching processes. This initial mineralogical analysis is also performed in order to evaluate the level of geochemical characterization needed. The initial mineralogical analysis may be performed by identifying the mineralogy of hand specimens in combination with supporting information about the mineralization, surrounding geology etc. If there are no indications of minerals that can release potentially harmful concentrations of specific elements no further mineralogical/geochemical characterization should be required. This may be the case for example for quarries with a well-known geology while e.g. metal mines will always require some level of further analysis.

6.2 Uncertainties – limitations

Assessing current physical and geochemical properties and predicting how these will develop in the future under the influence of weathering, with possible different closure alternatives, is a complex undertaking. Especially the prediction of future behaviour is no exact science and will always include a level of uncertainty. The level of accuracy and precision that is needed/acceptable will vary depending on the context. In situations where the test result and predictions will be the key factor in the choice between different management options a high level of accuracy will be needed (alternatively high safety factors will have to be applied). On the other hand, in clear cut cases, whether clearly benign or clearly problematic material, there may be no need for a high precision. This may be the case when assessing if drainage collection and treatment will be needed (while dimensioning of a treatment system will still require a higher level of precision).

Data quality and uncertainty can be understood in several ways. It can be the uncertainty related to the representativeness of the samples that are collected, the uncertainty of a specific test result (e.g. leaching and analysis), the variability in materials taken at different times or locations and it can relate to the uncertainty in the prediction of release behaviour under field conditions. These uncertainties are not easy to measure or calculate, but it is important to get a sense for the magnitude of uncertainty when trying to draw conclusions based on test results and predictions.

In many cases, predictive modelling will be part of the evaluation. The inherent uncertainty in model predictions is rarely stated or recognized. Considerable uncertainty is inherent in determining many of the parameters that are required for modelling drainage quality from waste rock or tailings. Predictions are commonly based on an extrapolation of test data outside the range of tested conditions which will lead to a significant increase of the uncertainty.

The uncertainties of modelling outputs may stem from incomplete characterization or incomplete knowledge of the geochemical conditions, lack of data on thermodynamic properties and/or insufficient knowledge of the hydrological conditions at the site. Methods used to evaluate or account for model uncertainty include Monte Carlo analysis, stochastic methods, and an evaluation of a range of model parameters to develop a range of deterministic outcomes. Rather than parameters being described by a single value as required in the model, parameters are better described with a probability distribution (i.e., a mean value with standard deviation, etc.).

Model uncertainty should be acknowledged in predicting drainage quality from waste rock or tailings. When this is performed by taking into account uncertainty in model parameters, this will still not address uncertainties in the conceptual model. Field verification is commonly needed to reduce uncertainties in the conceptual model, as omission of an important release controlling process may lead to a faulty prediction. Keeping an open mind and being prepared to rethink a conceptual model is important. In this context the interrelations between laboratory test results, lysimeter studies or test pads and actual measurements at full scale operations in the field can provide a higher level of understanding and thus increase the reliability of data that are developed to support management decisions.

Other recent guidance documents on characterization of waste from the extractive industry discuss uncertainties in different stages of the characterization process, e.g. the GARD Guide (2009) and the MEND prediction manual (Price, 2009). The MEND prediction manual highlights that there is never a complete understanding and perfect prediction, and the Manual emphasizes the use of sensitivity analysis and risk assessment as part of the characterization program. The GARD Guide also recommends using characterization and modelling results cautiously.

Quality control/quality assurance (QA/QC) is important in this context but deal primarily with measurable quantities, such as physical parameters, chemical analysis and leaching test results, and help to perform a process of sampling or laboratory analysis consistently. General laboratory practice is important in this context. EN ISO/IEC 17025 is a European Standard that specifies good laboratory practice. This typically is addressed in the performance of standard test procedures for which a well-defined protocol is available. In standardization, as part of issuing the standard a ruggedness evaluation and an inter-laboratory comparison is carried out. This activity provides performance characteristics of the standard in terms of within and between lab variability. These properties can be used to estimate the uncertainty in the analysis performed according to the standard protocol.

6.3 Interpretation and evaluation of geotechnical data

The physical properties of the waste are relevant in the context of slope/dam stability for dry-stacked tailings or waste rock dumps, for the use of waste (waste rock and/or coarse tailings) in dam construction, for the calculation of loadings on dams and for the assessment of consequences in case of a failure. One specific aspect to consider is the potential for liquefaction.

Information on the physical properties of the waste may be required as input to the design of waste facilities. However, data on the waste material only represents one of many categories of information needed for the design work and the safe design of waste facilities falls outside the scope of this guideline document.

In addition, the geochemical properties of the waste may have an impact on stability. During weathering of the waste material the grain size distribution and other physical properties may change, secondary minerals may form (e.g. clay minerals) that can destabilize the waste material. The primary minerals, solution chemistry (pore-water chemistry) and climate (temperature and rainfall) are the key parameters controlling the formation of secondary minerals.

Some key references and standards related the determination of geotechnical properties, the interaction between physical and chemical behaviour of mineral wastes and the design and monitoring of dams and dumps can be found in the Eurocode (2011), in other EN/ISO Standards (as listed in Annex C) and in a series of ICOLD Bulletins on tailings dams.

6.4 Mineralogy, chemistry, leaching and A/NRD evaluation

6.4.1 General

Evaluation and interpretation of mineralogy, chemical composition, leaching tests, acid-base accounting and kinetic tests is a complex task, however, it is important since the results to a large extent will guide the waste handling and the closure design at an operation.

Laboratory studies have clear limitations in predicting medium and long-term behaviour of the waste material. The methods are simulations of nature. However, with knowledge about the limitations of the methods, they can still assist in the making of sound decisions to minimize risks for the environment and human health and safety.

Laboratory testing with long-term prediction through modelling and verification against controlled field experiments (lysimeters) and actual field data from tailings ponds of waste rock dumps form an integrated approach using interrelated test results that together should cover the full range of conditions likely to be encountered in the location and time span considered. This is the underlying basis for EN 12920 which describes a tiered approach to laboratory and field testing.

The following clauses divide properties and tests into: mineralogy; chemistry; leach tests; and A/NRD, with suggestions on how to use the data and the limitations of the data obtained through the related test methods. In many cases, and in particular where sulfide minerals are present and oxidizing, a combination of all four classes of data may be necessary to give sound predictions of the potential impact.

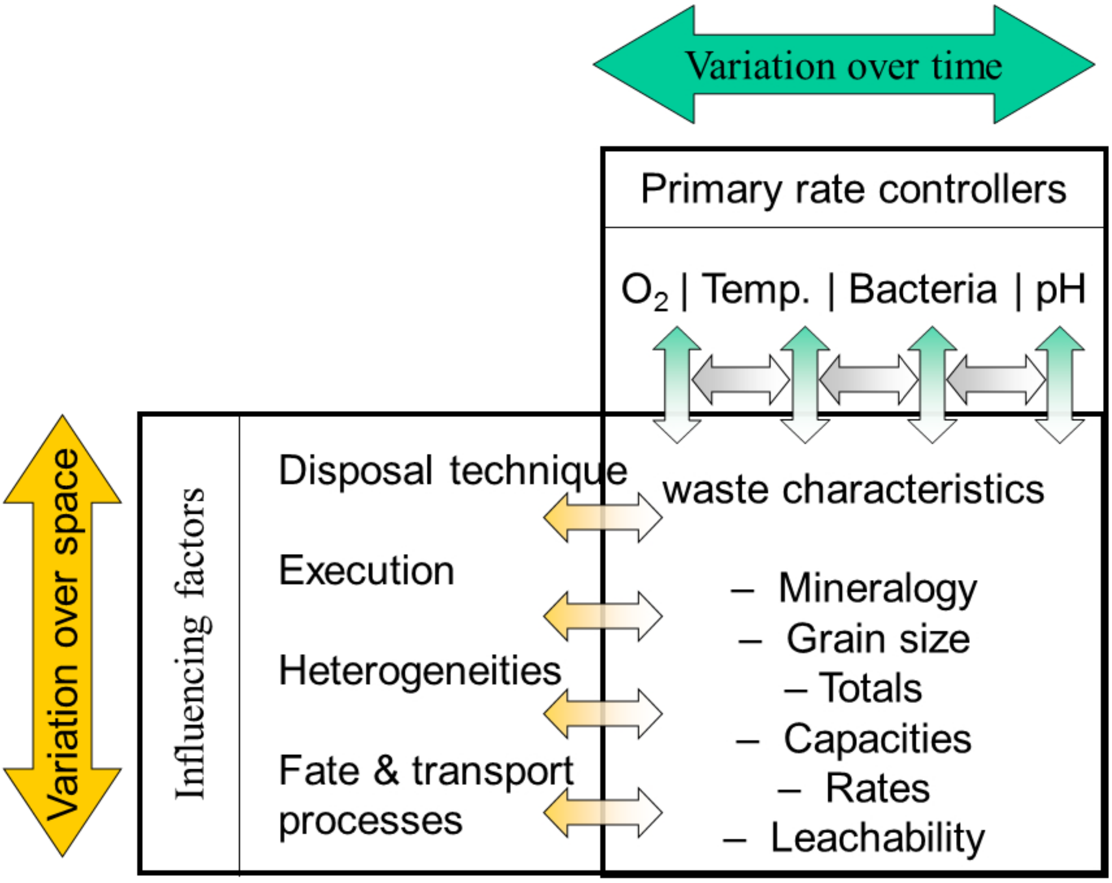

Figure 9 — Illustration of interrelationship between waste properties and external factors

Figure 9 attempts to illustrate the fact that chemical and physical stability of extractive wastes in the short and long term depends not only on the intrinsic waste characteristics, but also on waste management measures and on surrounding conditions. Acid generation potential itself depends on the intrinsic properties of the waste (mineralogy, capacities, etc.), but ultimately, the parameters that control the rate of acid generation are environmental (e.g. oxygen access, temperature, occurrence of specific bacterial populations and pH). Through the choice of different waste management options, the waste characteristics and/or the environmental conditions may be modified (especially the exposure to oxygen). The understanding of these interrelationships is crucial for how individual test data should be interpreted and what, if any, iterative characterization steps are needed to fully meet the characterization objectives.

6.4.2 Mineralogy

Mineralogical analysis is usually the first analysis performed in order to evaluate potential environmental effects from the waste material due to weathering processes. However, detailed mineralogical analysis is also an intricate part of understanding the result of solid and drainage chemistry, and A/NRD processes.

Mineralogical analysis will give the first indications of whether drainage water quality may be an issue. It is the minerals with their variable chemistry that are controlling drainage chemistry. Some minerals have the potential to produce acid while others have the potential to consume acid. In these reactions there are elements released to and removed from solution. Some minerals dissolve rapidly and can quickly control/buffer a solution with respect to elements within certain physico-chemical ranges. For example, as long as pH is above 5, precipitation of iron oxides/hydroxides will in most cases limit the iron concentration in the drainage to less than a few mg/l. Calcite will buffer a solution to pH 6 - 7 as long as it is available.

Many of the silicate minerals consume hydrogen ions (increase pH) during dissolution/weathering. However, the reaction rate is low and this neutralization is effective only when the contact time is long. Silicate minerals may contribute calcium, sodium, potassium, silicon, aluminium, iron and magnesium to the solution.

Sulfide minerals may contribute iron, hydrogen ions, sulfate, and many of the trace metals. Knowing the sulfide mineralogy is therefore essential in evaluating short term and long term A/NRD leaching potential. As discussed in more detail in Annex G, different sulfides have different potential to generate acid. While many sulfides do not generate much or any acid when oxidizing they may still be a major source of elements of concern, such as lead, cadmium, silver, arsenic and selenium. The knowledge of trace element distribution in sulfides is also an important issue because the susceptibility of sulfide to be weathered depends on their distribution.

Metal hydroxides and many clay minerals have the potential to sorb (adsorption, absorption, ion exchange) trace elements. The sorption capacity is depending on the solution chemistry (pH and element concentrations). Anions like selenate or arsenate will adsorb to metal hydroxides in acidic solutions, while cations of copper, zinc, lead e.g. will adsorb in alkaline conditions. Knowing the amount of minerals with sorption capacities, or the potential for these to form, is therefore one of the keys to evaluating natural attenuation and also the potential for release if conditions change.

As described in Clause 2, in specific cases mineralogical analysis in combination with existing information can be sufficient to conclude that leaching is not an issue. More commonly, the mineralogical analysis will be used to interpret the result of further geochemical analysis and it will be an essential part of performing any geochemical modelling. It is also important to be aware of and understand the limitations of mineralogical analysis when used in the evaluations.

The literature gives information about reaction rates and rate controls for many of the minerals that can leach, oxidize, neutralize etc. However, there are commonly several orders of magnitude between the reported mineral reaction rates and what is found in a natural setting with complex mineralogy and non-pure minerals. This is why data from mineralogical analysis in most cases will not give an acceptable prediction of drainage water chemistry. Chemical analysis, leach tests and kinetic testing may therefore provide relevant complementary information when contaminated drainage may be an issue. Mineralogical information can then be used to evaluate and interpret the results from the leaching and A/NRD analysis.

6.4.3 Chemistry

Chemical analysis is in many cases easy to interpret. The bulk chemical analysis are primarily used for

- assessment of potential concerns (increased levels of sulfur or other “substances of concern”,) and thereby reason for further investigation;
- evaluation against national or international criteria (if they exist) and
- verification (primarily during operation) to evaluate consistency of waste material.

The chemical analysis of solids can be performed as total concentrations by using XRF, or by different digestion methods that have different mineral dissolution capabilities. This is discussed in more detail in 5.5. The result will vary depending on the method used. Therefore, the method should always be specified. For the same reason, if limit criteria are set it is important that the corresponding digestion method is specified.

6.4.4 Acid-base accounting

Acid-base accounting (ABA) is a set of methods used to evaluate if material has the potential to generate acid or not. As described in 5.6 the main acid producers in this respect are iron containing sulfides, especially pyrite and pyrrhotite, while the main rapid neutralizers are the carbonate minerals, especially calcite.

Total sulfur from bulk analysis can be used to evaluate the acid potential (AP). If it is assumed that all sulfur analyzed is present as pyrite this will give the worst case scenario. Mineralogical analysis may aid in refining this assessment by subtracting sulfur present in e.g. gypsum and calculate AP using the reduced sulfur content.

If there is a good correlation between total sulfur and AP, bulk sulfur analysis which can be analysed quickly on site, can be used to evaluate waste handling on a daily basis in the operational phase.

The neutralization potential (NP) of the material in question is analyzed directly by measuring how much acid the material can consume within 24 h (EN 15875).

Both AP and NP are commonly expressed in kg $CaCO_3$ eq/tonne of material. AP or NP expressed in carbonate equivalents ($CaCO_3$) in kg/t can be converted to H+ content in mol/kg by multiplying by 0,02.

The result of AP and NP is evaluated in different ways. The net acid neutralizing potential (NNP) is calculated by Formula 2 and the neutralizing potential ratio (NPR) by Formula 1.

The NNP and the NPR is first of all used to evaluate the risk of the waste material going acid, resulting in the production of acid rock drainage (ARD).

Commonly the NPR and NNP are evaluated by the following rule of thumb (Price, 2009):

NPR < 1 ;	Potentially acid generating
1 < NPR < 3 ;	Uncertainty zone
NPR > 3 ;	Non-acid generating

NNP < -20 ton $CaCO_3$(eq)/1000 tones of material ;	Potentially acid generating
20 < NNP < 20 ;	Uncertainty zone
NNP > 20 ;	Non-acid generating

The NPR is commonly plotted in a diagram NP vs. AP (Figure 10).

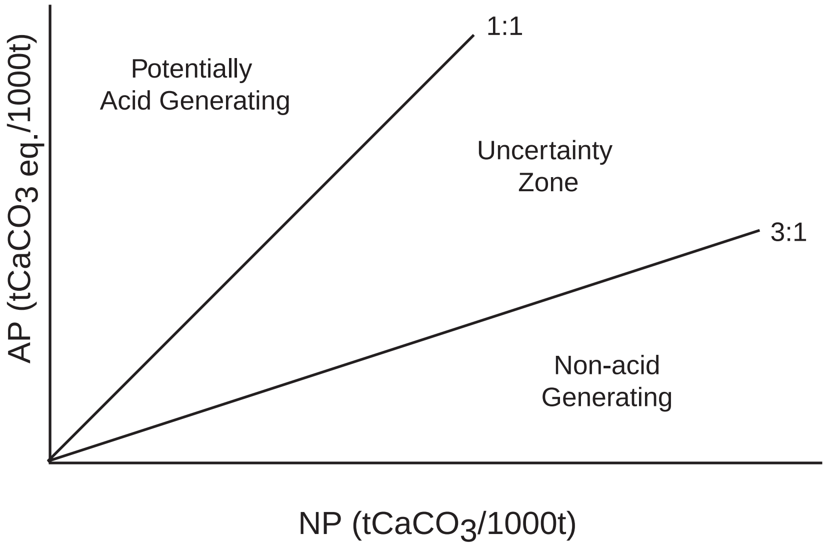

Figure 10 — AP vs NP with the common interpretation

However, it should be noted that these interpretation criteria are simplifications. It is important to understand how and why they may be misleading in some cases:

- If the material is heterogeneous there is potential, even though the criteria indicates that there will be no acid generation, that parts of the waste material in question will go acidic where the neutralizing material is not available (more common in waste rocks than tailings). The result will in this case very much depend upon the sampling procedures.
- There may be neutralizing material available, however, if the sulfides oxidize, hazardous elements mobile in neutral environment may be released and cause problems (neutral rock drainage, NRD).

Some additional factors that might lead to the wrong conclusion are discussed in the following paragraphs

The data being plotted into the figure NP vs AP is based on the data received from the ABA testresults. When interpreting the plot at least the following issues needs to be considered:

- Is the AP based on sulfide sulfur or total sulfur?
- Is there a significant amount of sulfide minerals in addition to pyrite?
- Is there a significant amount of carbonate minerals in addition to calcite?
- Is there a significant amount of siderite?
- Is there a significant amount of acid generating sulfate minerals?
- Is there a significant amount of minerals in addition to carbonate minerals that can under some condition act as neutralizing minerals?

The answers to these questions will lead to the following adjustments:

a) If there is a significant amount of other sulfide minerals than pyrite in the analyzed material, the acid generation potential is overestimated both by using total sulfur and sulfide sulfur, so the AP should be reduced (Walder et al, 2000).

b) If the condition is such that silicate minerals have time enough to react many of them have the potential to neutralize acid. This is especially possible in tailings and in waste rocks in arid and semi arid environment or in cold climates (Walder et al, 2010). Under such conditions, the NP is underestimated using EN 15875.

NOTE However, if the sulfides oxidize, hazardous elements mobile in neutral environment may still be released and cause problems, e.g. neutral rock drainage.

c) Iron sulfate minerals dissolve easily and generate acid. The iron sulfate minerals are common secondary product from iron sulfide oxidation. If they are present in the material in significant amount, the AP is underestimated.

d) Siderite is an iron carbonate and will at low pH act as a neutralizer, however, under pH conditions where iron hydroxide precipitate it will become an acid generating mineral. This will occur above pH 3,5 - 4. If siderite is present in a significant amount the AP is underestimated.

These issues should be included in the evaluation of the ABA result and the effects are illustrated in Figure 11.

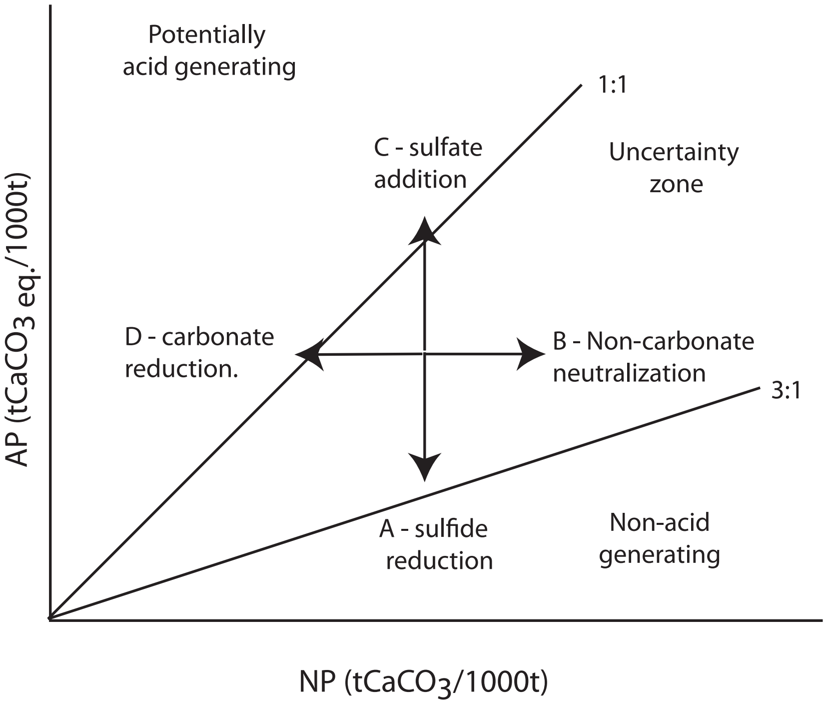

Figure 11 — Illustration showing how mineralogical data can be used to evaluation of the ABA result

The addition of acid based on the total carbonate content according to EN 15875 instead of based on a fizz test as in previous test protocols will in most cases be an improvement of the consistency of the result between the laboratories. The largest uncertainty will be when there are high amounts of carbonate minerals with slow reaction time, especially dolomite and siderite. This will result in an overly high acid addition at the beginning. The early measurement of pH in the test should observe this and a new test can be performed with lower acid addition.

6.4.5 Kinetic testing

The principles for kinetic testing are introduced in 5.6.4 and further discussed in CEN/TR 16363.

Kinetic testing is primarily applied when the ABA test (6.4.4) does not give a clear answer regarding acid generation potential. Kinetic testing may also be carried out e.g. when there is a need for a better understanding of processes in the waste to make the right waste management decisions. Depending on the set-up, the kinetic test results may form the basis for an evaluation of, among other things, A/NRD potential, reaction rates and drainage quality.

Commonly kinetic testing refers specifically to tests run to evaluate acid generation behaviour of sulfidic wastes, i.e. to quantify reaction rates for acid producing and buffering reactions and to evaluate if the material actually will go acidic or not. Also in cases when the acid produced is buffered to a neutral pH kinetic testing may give information on the release of constituents of concern (i.e. Neutral Rock Drainage).

The reaction rates can be calculated by using the optimum concentration when the release rate has stabilized, e.g. towards the end of the evolution curves in Figure 12. CEN/TR 16363 discusses the most common approaches to calculating reaction and leaching rates based on kinetic test data.

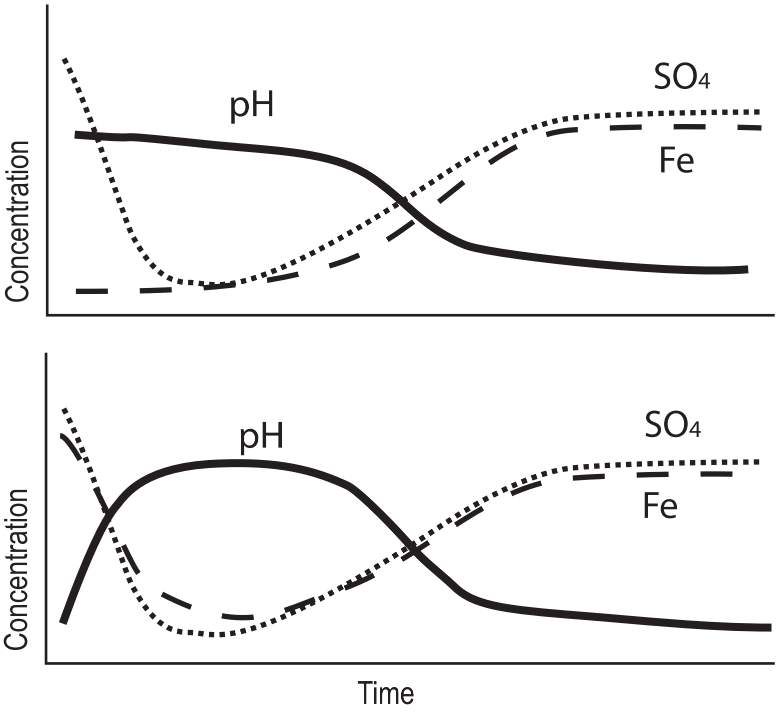

Figure 12 — Illustration of a possible kinetic test result. The top graph illustrates a sample that contains an easily dissolved sulfate mineral (e.g. gypsum) that does not generate acid when dissolving, while the lower graph illustrates a sample containing easily soluble acid generating sulfate (e.g. jarosite). Both samples are with time acid generators

The column tests can be very useful in evaluating different remediation options such as waste mixing, lime treatment, and cover systems. The material in the columns can be evaluated following the tests to determine the processes taking place within the columns. From these evaluations, treatment systems may be improved or designed.

CEN/TR 16363 discusses interpretation and evaluation of kinetic test results in some detail. Some key points are summarized in the following.

It is necessary to clearly define the testing objectives to select and design kinetic tests appropriately to obtain the desired information for interpretation and evaluation of the data.

The application of laboratory data to field conditions should always be done with caution. The implications of the differences between the laboratory set up and field conditions need to be well understood and taken into consideration.

Some of the issues that should be considered when evaluating the potential differences between the natural setting and the test setup are:

- Is the oxygen availability higher in the tests?
  - higher oxidation rate in the tests;
  - oxidation in tailings are commonly oxygen limited.
- Is the air flow drying out the sample more than in the natural setting?
  - to the extent where it leads to a reduction of oxidation rate in the test?
  - reduction of leach rate in the test?
- Is secondary mineral precipitation avoided in the tests, but likely taking place in the natural setting due to higher water: rock ratio in the tests?
  - mineral precipitation can result in encapsulating acid neutralizing minerals, thus reducing their neutralizing capabilities in the field;
  - precipitation can result in sorption and reduced release in the field;
  - lower flow rate in the field can result in more effective neutralization from slowly reacting minerals.

In conclusion, laboratory scale tests may provide useful information on material properties and behaviour but commonly have a relatively high uncertainty when applied to field conditions and used to predict developments in the long term. The uncertainty needs to be evaluated relative to how the results are being used. Scaled up tests may be of great value to verify conclusions and to reduce this uncertainty once the operation is in production.

6.4.6 Leaching

Throughout this document the potential impact on water is a key aspect, be it drainage water, pond water, or percolating water. The concentration of different elements in such waters is controlled by the contact with the waste. The concentration can be equilibrium based (if water flow is low relative to the rate of solid solution exchange), or kinetically controlled by weathering and diffusion. In many cases these processes are biologically mediated. Many factors control the resulting concentrations in water that comes in contact with the waste, such as pH, Eh, EC, L/S, minerals, sorptive phases like iron-, aluminium-, manganese oxide and organic matter.

As described in 5.7, there are a number of different leaching tests available. Some of the tests have been designed for a specific context, but often wrongly used outside the areas of design with the consequence that the data does not give any meaningful information. Only a few leaching tests have been designed specifically for the waste from the extractive industry. Some procedures are still general enough to be applicable for characterizing extractive wastes in scenarios where local equilibrium can be assumed. However, these tests do not capture the effect of mineralogical weathering and weathering rates.

Leaching addresses the release of substances by dissolution from the surface of particles and/or by diffusion from larger particles after dissolution in the pores of a solid matrix. However, leaching is only the first step in an impact assessment. As many environmental impacts are caused by substances being transported in the water phase, only constituents that can dissolve under realistic conditions can play a role. Factors controlling solubility under realistic exposure conditions are: mineral precipitation/dissolution, sorption on iron-oxide phases, binding to clay surfaces, and cations exchange.

All leach tests (except for the kinetic tests that may also be considered leaching tests) are based on short term leaching. For most minerals that may be found in waste from the extractive industry short term tests will only give limited information about long term leachability.

The short term leach tests are most suited for waste that has been exposed to weathering for some time (months to years) and are in a quasi-stable leaching mode. Test results can then be used to evaluate leachability during the operational phase (short to medium term). For example, to assess what will be released to the water phase during the next rainfall.

Results from leaching tests can also be used to evaluate geochemical modelling or as an input to such modelling. Evaluation of geochemical modelling can be done through comparing modelling results with actual leach test results. This comparison is used for optimising or verifying the modelling setup. If leach test results are used as an input to geochemical modelling it will commonly be as an initial solution. In both cases the data from the leaching tests should be adjusted at least for the water: rock ratio and the grain size/surface area used in the modelling.

6.5 Field verification

Most of the analysis and methods described in this document are based on relatively small samples and short term analysis. Even the kinetic tests, that may last for a year or more, are considered short term when taking into account that the analysis/methods are in some cases used to evaluate long term effects over hundreds to thousands of years.

If there is a potential for mineralogical weathering affecting the chemical and geotechnical stability of the waste, long-term onsite field scale tests can be a very useful complement when trying to interpret waste characterization results. These tests can be in place throughout the mine life and provide feedback to update interpretations and closure plans. Most operations will have a monitoring program including drainage water quality and impacts on surface and ground water. This information on water chemistry provides valuable ongoing information on waste behaviour and may in some cases be sufficient to validate or invalidate initial predictions.

Investigations of abandoned waste sites have many similarities with evaluating operating sites. With the important difference that normally there are no initial or existing test results to verify. In those cases, field investigations and laboratory analysis of field samples are the most reliable means of gaining a reasonable indication of the long term chemical and geotechnical stability of the material.

Field investigations of old tailings storage facilities with sulfidic tailings may provide very useful information. If there is information available to support the assumption that the tailings were originally homogeneous with respect to sulfides, i.e. that the oxidized tailings above the oxidation front initially had the same sulfide content as the observed content below the oxidation front, it is possible to calculate the bulk sulfide oxidation rate. The bulk oxidation rate together with the depth to the oxidation front can be used for verification of geochemical modelling of the tailings. Modelling can then be used e.g.:

- to estimate leaching rates of constituents in the future based on expected changes in release controlling conditions;
- as an input to designing appropriate closure.

It should be noted that in many cases the tailings are in fact not homogeneous. This may be due e.g. to changes in the ore mineralogy with depth, high-grading in “bad times” or processing of marginal ore towards the end of mine-life.

6.6 Water quality predictions

When a quantitative assessment of long term releases from extractive waste facilities is needed/motivated, geochemical reaction transport modelling is often the only way forward as testing in the laboratory will not allow a prediction of what will happen under a variety of exposure conditions. This approach needs to be built up in steps, starting from an understanding of the leaching behaviour in controlled laboratory tests and ultimately to drainage chemistry in the field. Taking those steps, verification of modelling output with experimental data may provide the level of understanding needed to move to the next level of complexity.

The waste related data produced by following this guidance can be used as input for geochemical modelling. This clause introduces some basic aspects of modelling. Annex H discusses modelling in more detail and lists available modelling tools. However, guidance on how to carry out a modelling exercise will have to be sought elsewhere.

There are many different geochemical and hydrological modelling programs available. Different programs have different capabilities. Some are only suited for limited tasks while others may handle many different tasks. The most sophisticated programs are reactive transport modelling programs that combine, geochemical and flow modelling. The more sophisticated the programs are, the more input data is needed to get useful information from the output.

The input parameters may be (depending upon the programs used):

- primary mineral phases;
- mineral quantities;
- mineral chemistry;
- secondary mineral phases;
- initial water quality;
- input water quality;
- mineral reaction rates;
- porosity;
- saturated and unsaturated hydraulic conductivity;
- water saturation;
- air permeability.

These parameters may be generated by interrelated observations from the different tests described in Clause 5.

A first step of a geochemical modelling scheme may be to model the output of the leach tests/kinetic tests performed on the material and, if working on an established waste facility, to model the observed/analysed information from the field.

7 Documentation and reporting

Proper documentation and reporting is important in building trust between involved parties (operator, regulators, general public, NGOs) and for the credibility and traceability of test results. The documentation of the waste characterization should cover all the steps from describing the rationale behind performing the waste characterization through to the evaluation and conclusions.

The structure of the documentation of the result and interpretation should follow good reporting practices and any applicable legal requirements. For a mineralogically, geochemically and geotechnically simple setting the report will also be simple. A recommended structure[3], including the documentation of relevant supporting information, is given below. Items marked with * represent a simple case building on existing information with no additional testing:

1) introduction *
2) background information of the (future) operation (type and description of extraction method, nature of the intended product) *;
3) geology of the site (nature of surrounding rocks, their chemistry and mineralogy, mineralization typology, including physical properties, size and geometry of deposit, weathering of the deposit) *;
4) objective(s) and approach(s) to the characterization;
5) sampling procedures;
6) analytical procedures;
7) baseline data for the site of disposal *;
8) presentation of the results of the characterization;
    - field data;
    - analytical data.
9) evaluation and discussion of the result;
    - waste types and its intended handling *;
    - mineralogical and geochemical description of the waste *;
    - volume/mass of different types of waste and when waste is produced *;
    - description of potential geotechnical and geochemical impact *;
10) recommendations for follow up work;
11) references to background documents/existing information *;
12) attachment of:
    - laboratory reports;
    - field reports (field analysis and observations);
    - sampling plan.

Copies of all reports should be stored at the operator’s office for possible future auditing.

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[3] Building on the structure of COM decision 2009/360/EC

Annex A
(informative)

Terms and definitions

For the purposes of this Technical Report, the following terms and definitions apply.

This document adopts the terms and definitions used in the Directive on the management of waste from extractive industries, 2006/21/EC.

acid rock drainage
ARD
sulfide mineral oxidation and acid generation due to air and water exposure of sulphide minerals, and metal release. Due to sulfide mineral oxidation and metal release and acid leaching of other minerals with possible acid consumption. The term is commonly used in the extractive industry, but it is a natural process that is enhanced by excavation, and crushing giving more surface area

bulk sulfide oxidation rate
oxidation rate of sulfide minerals, not specific mineral in a rock with mixed mineralogy

macro flow
flow of water through conduits within the waste rock dumps (preferential flow paths), water that reports to the base shortly (hours to days after a rainfall)

matrix flow
flow of water within finer material of waste rock dumps, water that may report to the base months to years after a rainfall

micro flow
flow of water into and out of particles within waste rocks due to capillary forces and evaporation when the material is somewhat porous

mineral deposit
a naturally occurring enrichment of minerals that potentially can be economically exploited

Neutral Rock Drainage
NRD
sulfide oxidation, and metal release, where the acid generated is neutralized by other minerals

ore deposit
a naturally occurring enrichment of heavy minerals that can be or has been economically exploited

overburden
layer of natural grown soil or massive rock on top of a mineralization. In case of open pit mining operations it has to be removed prior to extraction of the ore

oxidation front
maximum depth were oxidation within particles, waste dumps, or tailings is occurring. Beyond this front there is no oxidation, behind this front oxidation is taking place or has been completed

topsoil
natural huminous layer on top of the mineralization, which has to be stripped prior to start-up of extraction

waste-rock
part of the mineralization, with or without low grades of value minerals, which cannot be sold profitably

waste management plan
plan for handling the waste generated during exploration and extraction of a mineral deposit as described in the EU-Directive for waste from extractive industries

waste deposit
dump, pond, where waste has been discharged

waste disposal
discharge of waste

waste disposal site
location for discharge of waste

Abbreviations

AAS	Atomic Adsorption Spectroscopy
ABA	Acid-Base Accounting
AFM	Atomic Force Microscopy
ANC	Acid Neutralization Capacity
A/NRD	Acid/Neutral Rock Drainage
AP	Acid producing Potential
BET	Brunaur, Emett and Teller method
BSE	Back-Scattered Electrons
CEN	European Committee for Standardization
COM	Commission
CPT	Cone Penetrometer Testing
EDS	Energy Dispersive Spectroscopy
EPMA	Electron Probe Micro-Analyzer
ICP-AAS	Inductively Coupled Plasma - Atomic Absorption Spectrometry
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
NAG	Net Acid Generation
NNP	Net Neutralization Potential
NP	Neutralizing Potential
NPR	Neutralization Potential Ratio
QA/QC	Quality Assurance/Quality Control
SE	Secondary Electrons
SEM	Scanning Electron Microprobe
SOP	Standard Operating Procedures
SPT	Standard Penetrometer Testing
VSI	Vertical Scanning Interferometer

WDS	Wavelength Dispersive Spectroscopy
WRI	Water Rock Interaction
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
XRPD	X-ray Powder Diffraction

Annex B
(informative)

Test methods – Geochemical analysis – Applicability of EN and ISO Standards

B.1 Introduction

CEN/TC 292 decided to evaluate the applicability of existing CEN/TC 292 standards and work items for wastes of the extractive industries. Additionally, suitable methods from other CEN and ISO Technical Committees are also listed. This includes methods from the aggregate sector (CEN/TC 154) and work done on horizontal (applicable across different matrices) methods for content in the context of the Construction Product Directive (CEN/TR 16045) as well as methods listed in the BREF document (Reference document on BAT, 2004) and in the CBM review provided by Euromines (CBM report, 2005) plus methods developed in CEN/TC 400 (formal project HORIZONTAL). The leaching tests included in the review are presented separately in Annex D.

B.2 Evaluation of applicability

B.2.1 General

There are numerous methods to analyse waste from the extractive industries. To have a better overview methods have been put into groups which are:

- sample preparation;
- physical-chemical parameters;
- digestion methods;
- major, minor and trace elements;
- sulfur (total, sulfate and sulfidic);
- organic and inorganic carbon;
- compliance testing;
- leaching methods (see Annex D);
- eluate and leachate analysis.

When several methods are available for the same item, then the multi-matrix and multi-substance methods are preferred tools in view of their broader applicability. When methods developed in different sectors are basically the same, this is indicated and in that situation either one can be selected. The detection limit needed in a specific case should be evaluated and specified since this may differ between alternative methods.

B.2.2 Procedure

Applicability has been checked from the desk using the following multistep approach.

- compilation of standards from CEN/TC 292 “Characterization of waste”, CEN/TC 154 “Aggregates”, CEN/TC 308 “Characterization of sludges”, ISO/TC 190 “Soil quality”, CEN/TC 400 "Horizontal standards in the fields of sludge, treated biowaste and soil" (former project HORIZONTAL), CEN/TC 351 "Construction products: Assessment of release of dangerous substances" and other relevant international Technical Committees;

- addition of standards from the Reference Document on BAT (2004) and the CBM Report (2005);

- addition of other existing standards (if possible) to fill any gaps identified;

- check of the scope of the methods and elimination of those with an inappropriate scope or usage;

- elimination of those with specific limitations (e.g. digestion with acetic acid, inappropriate liquid/solid ratios, insufficient degree of representivity);

- check of full copies of the standards remaining on the list;

- marking all methods that have not been validated with the exception of methods that cannot be validated (e.g. description of sample pre-treatment).

B.2.3 Types of wastes

Applicability of methods has been evaluated for 3 types of waste as addressed in the directive 2006/21/EC:

- tailings;

- waste rock and overburden*;

- topsoil*.

*provided that they constitute waste as defined in Council Directive 75/442/EEC of 15 July 1975 on waste.

B.2.4 Levels of applicability

The following tables give the applicability for tailings, waste rock and overburden, and topsoil, respectively. The applicability has been divided into 3 levels

“yes”	this method is general applicable to wastes from the extractive industries for each type (i.e. tailings, waste rock/overburden, and topsoil);
“(yes)”	this method is applicable to wastes from the extractive industries for each type but several restrictions have to be considered or methods are not yet validated;

NOTE All methods developed by CEN/TC 400 (the project HORIZONTAL) have been rated in this level.

“no”	this method is not applicable to wastes from the extractive industries for each type.

Geotechnical tests are handled in Annex C.

B.3 Methods to analyse waste from the extractive industries

B.3.1 General remark

Methods that are fully applicable are rated “yes” without any remark. The preferred method based on most wide applicability, multi-matrix or multi-substance method is indicated as the preferred method. This does not preclude use of any of the other methods, but specifying a preferred method will help to improve comparability of data within the sector. All other methods are marked and an explanation for the choice of the level of application is given as comments within each table.

This list is not exhaustive; it gives methods that are applicable to most wastes from extractive industries. The use of other methods is possible, particularly when working under special circumstance (type of product and waste, parameters not listed, etc.).

B.3.2 Sample preparation

Parameter/ method	**Number**	**Title**	**Applicable**	**Comment**
General	EN 932-2	Test for general properties of aggregates – Part 2: Methods for reducing laboratory samples	yes	
Ecotoxicity	EN 14735	Characterization of waste – Preparation of waste samples for ecotoxicity tests	yes	
General	EN 15002	Characterization of waste – Preparation of test portions from the laboratory sample	yes	preferred
General	ISO 11464	Soil quality – Pre-treatment of samples for physico-chemical analysis	(yes)	Method is limited to particle size < 2 mm while > 2 mm material is not treated because for soil analysis only material < 2 mm is relevant. If this method should be applied to wastes the fraction > 2 mm has to be crushed and included in the sample.
Freeze-drying	ISO 16720	Soil quality – Pre-treatment of samples by freeze-drying for subsequent analysis	(yes)	Method has limitations as water may not be fully removed, better applicable to very moist samples

B.3.3 Physical-chemical parameters (see also Annex C)

Parameter /method	**Number**	**Title**	**Applicable**	**Comment**
Water content	EN 1097-5	Tests for mechanical and physical properties of aggregates – Part 5: Determination of the water content by drying in a ventilated oven	(yes)	Methods in principal applicable but the result is given to a different basis. For wastes, solid mineral fuels (coal) and sludges the moisture content is calculated by dividing the moisture mass by the sample mass (i.e. dry plus moisture mass), while for soil and aggregates it is calculated by dividing the moisture mass by the dry mass.

	EN 12880	Characterization of sludges – Determination of dry residue and water content	yes	
	EN 14346	Characterization of waste – Calculation of dry matter by determination of dry residue or water content	yes	EN 14346 and prEN 15934 are basically the same. Preferred method
	prEN 15934	Sludge, treated biowaste, soil and waste – Calculation of dry matter by determination of dry residue or water content	yes	EN 14346 and prEN 15934 are basically the same.
	ISO 589	Hard coal – Determination of total moisture	yes	
	ISO 11465	Soil quality – Determination of dry matter and water content on a mass basis - Gravimetric method	(yes)	see EN 1097-5
	CEN ISO/TS 17892-1	Geotechnical investigation and testing. Laboratory testing of soil – Part 1: Determination of water content (ISO/TS 17892-1)	(yes)	see EN 1097-5
pH and conductivity	EN 12176	Characterization of sludge – Determination of pH-value	(yes)	The scope is limited to sludges. However, it includes solid sludges similar to the wastes classified here. Although not tested the method seems to be applicable.
	ISO 10390	Soil quality – Determination of pH	no	Too many options in this standard. Many parameters may influence the result (e.g. influence of stirring, contact time before pH-measurement, the packing of material in the spoon). Methods have not been tested for tailings and waste rock/overburden.
	prEN 15933, prEN 15937	Sludge, treated biowaste and soil - Determination of pH Sludge, treated biowaste and soil - Determination of specific electrical conductivity	yes	Standard to be validated. Preferred method
	ISO 11265	Soil quality – Determination of the specific electrical conductivity	yes	
Loss on ignition	EN 1744-1	Tests for chemical properties of aggregates – Part 1: Chemical analysis Article 17: determination of loss on ignition; T = 975 °C	(yes)	The 4 following standards use different temperatures for ignition (550 °C to 975 °C) which may give different results not validated
	EN	Characterization of waste – Determination of loss on ignition in waste, sludge and	yes	EN 15169 and EN 15935 are

	15169	sediments ; T = 550 °C		basically the same. Preferred method
	EN 15935	Sludge, treated biowaste, soil and waste – Determination of loss on ignition (LOI), T = 550 °C	yes	Standard in enquiry.
	ISO 1171	Solid mineral fuels – Determination of ash, T = 815 °C	(yes)	see EN 1744-1

B.3.4 Digestion and extraction methods

Parameter/ method	Number	Title	Applicable	Comment
Total digestion with mineral acids	EN 13656	Characterization of waste - Microwave assisted digestion with hydrofluoric (HF), nitric (HNO_3) and hydrochloric (HCl) acid mixture for subsequent determination of elements	yes	Preferred method when digestion of silicates is required
	ISO 14869-1	Soil quality – Dissolution for the determination of total element content - Part 1: Dissolution with hydrofluoric and perchloric acids	yes	
Total digestion by alkaline fusion	CEN/TR 15018	Characterization of waste – Digestion of waste samples using alkali- fusion techniques	yes	
	ISO 14869-2	Soil quality – Dissolution for the determination of total element content - Part 2: Dissolution by alkaline fusion	yes	
Aqua regia extraction	EN 13657	Characterization of waste – Digestion for subsequent determination of aqua regia soluble portion of elements	yes	Preferred method EN 13657 is the same as prEN 16174
	prEN 16174	Sludge, treated biowaste and soil – Digestion of aqua regia soluble fractions of elements	(yes)	Multi-matrix method
	ISO 11466	Soil quality – Extraction of trace elements soluble in aqua regia	yes	

B.3.5 Composition: major, minor and trace elements

Parameter/ method	Number	Title	Applicable	Comment
XRF	EN 15309	Characterization of waste and soil – Determination of elemental composition by X-ray fluorescence	(yes)	
ICP-AES	prEN 16170	Sludge, treated biowaste and soil – Determination of trace elements by	yes	Preferred method – Multi-substance method

		inductively coupled plasma optical emission spectrometry (ICP-OES).		
	ISO 22036	Soil quality – Determination of trace elements in extracts of soil by inductively coupled plasma - atomic emission spectrometry (ICP AES)	(yes)	
ICP-MS	prEN 16171	Sludge, treated biowaste and soil – Determination of trace elements using inductively coupled plasma mass spectrometry (ICP-MS)	yes	Preferred method – Multi-substance method
AAS graphite furnace	prEN 16172	Sludge, treated biowaste and soil – Determination of trace elements in aqua regia and nitric acid digests – Graphite furnace atomic absorption spectrometry method (GFAAS)	(yes)	
AAS flame	CEN/TS 16188	Sludge, treated biowaste and soil – Determination of elements in aqua regia and nitric acid digests – Flame atomic absorption spectrometry method (FAAS)	(yes)	
	ISO 11047	Soil quality - Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc - Flame and electrothermal atomic absorption spectrometric methods	yes	
Mercury	prEN 16175-1	Sludge, treated biowaste and soil – Determination of mercury in aqua regia and nitric acid digests – Part 1: Cold vapour atomic absorption spectrometry (CVAAS)	(yes)	
	prEN 16175-2	Sludge, treated biowaste and soil – Determination of mercury in aqua regia and nitric acid digests – Part 2: Cold vapour atomic fluorescence spectrometry (CVAFS)		

	ISO 16772	Soil quality – Determination of mercury in aqua regia soil extracts with cold-vapour atomic spectrometry or cold-vapour atomic fluorescence spectrometry	yes	
Thallium	ISO 20279	Soil quality – Extraction of thallium and determination by electro thermal atomic absorption spectrometry	yes	
Arsenic, Antimony, Selenium	ISO 20280	Soil quality – Determination of arsenic, antimony and selenium in aqua regia soil extracts with electro thermal or hydride-generation atomic absorption spectrometry	(yes)	
Chromium (VI)	CEN/TR 14589	Characterization of waste – State of the art document – Chromium VI specification in solid matrices	yes	
	EN 15192	Characterization of waste and soil – Determination of Chromium(VI) in solid material by alkaline digestion and ion chromatography with spectrophotometric detection	yes	

B.3.6 Organic and inorganic carbon

Parameter/ method	Number	Title	Applicable	Comment
TOC	EN 13137	Characterization of waste – Determination of total organic carbon (TOC) in waste, sludges and sediments	yes	Preferred method
	prEN 15936	Sludge, treated biowaste, soil and waste – Determination of total organic carbon (TOC) by dry combustion	yes	EN 13137 and prEN 15936 are basically the same
Elemental C analysis	ISO 10694	Soil quality – Determination of organic and total carbon after dry combustion (elementary analysis)	yes	
	ISO 29541	Solid mineral fuels – Determination of total carbon, hydrogen and nitrogen content – Instrumental method	no	Method is only applicable to coal. It gives both organic and inorganic (carbonate) carbon
Carbonate	EN 196-2	Methods of testing cement – Part 2: Chemical analysis of cement;	yes	

determination		section 15 – Determination of the carbonate content		
	ISO 10693	Soil quality – Determination of carbonate content – Volumetric method	yes	
	prEN 15936	Sludge, treated biowaste, soil and waste – Determination of total organic carbon (TOC) by dry combustion	yes	TIC = carbonate is part of the method. Preferred method
	ISO 925	Solid mineral fuels – Determination of carbonate carbon content – Gravimetric method	yes	

B.3.7 Sulfur (total, sulfate and sulfidic) (see also Annex F for sulfide analysis)

Parameter/ method	Number	Title	Applicable	Comment
Bomb combustion	EN 14582	Characterization of waste – Halogen and sulfur content - Oxygen combustion in closed systems and determination methods Method A: bomb combustion	yes	Preferred method
Schöniger apparatus	EN 14582	Characterization of waste – Halogen and sulfur content - Oxygen combustion in closed systems and determination methods Method B: Schöniger apparatus	no	Method not sensitive enough
High temp. combust.	ISO 351	Solid mineral fuels – Determination of total sulfur – High temperature combustion method	yes	Combustion temperature shall be high enough to ensure that all sulfur components including temperature stable inorganic sulfates (e.g. calcium sulfates) are detected
	ISO 15178	Soil quality – Determination of total sulfur by dry combustion	yes	
iodine method	EN 1744-1	Article 11: determination of total sulfur content	no	Method not recommended because of the use of very toxic substances
sulfates and sulfides	EN 1744-1	Article 12: determination of acid soluble sulfates	(yes)	Method is in principle applicable but very labour and time consuming
	ISO 157	Coal – Determination of forms of sulfur	no	Method is only applicable to coal. The principle of distinction between sulfate and pyritic sulfur used in this standard (dissolution in hydrochloric acid followed by nitric acid) might be applicable but has not been tested. However, the method is very labour and time consuming
	ISO 11048	Soil quality – Determination of water-soluble and acid-soluble sulfate	no	Method is not applicable because the use of hydrochloric acid at a concentration of $c(\mathrm{HCl}) = 6$ mol/l will not only dissolve sulphates but also sulfides. Additionally, method is limited to particle size < 2 mm while > 2 mm material is not treated

B.3.8 Eluate and leachate analysis

Parameter/ method	Number	Title	Applicable	Comment
Elements	EN 12506	Characterization of waste – Analysis of eluates – Determination of pH, As, Ba, Cd, Cl, Co, Cr, Cr VI, Cu, Mo, Ni, NO_2, Pb, total S, SO_4^{2-}, V and Zn	yes	
Group parameters	EN 13370	Characterization of waste – Analysis of eluates – Determination of Ammonium, AOX, conductivity, Hg, phenol index, TOC, Cyanide (easily liberatable), F^-	yes	Preferred method
Dissolved solids	EN 15216	Characterization of waste – Determination of total dissolved solids (TDS) in water and eluates	(yes)	
ICP-OES	prEN 16170	Sludge, treated biowaste and soil – Determination of trace elements by inductively coupled plasma optical emission spectrometry (ICP-OES)	yes	Preferred method – Multi-substance method

B.3.9 Eluates: analytical methods referenced in EN 12506 and EN 13370

The European Standards EN 12506 and EN 13370 do not describe methods used for the characterization of eluates but give reference to existing methods used for water analysis. All methods listed in B.3.9 and B.3.10 are applicable to eluates derived from any waste from the extractive industries.

EN 12506			
Parameter	**Number**	**Title**	**applicable**
pH	ISO 10523	Water quality – Determination of pH	yes
As	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
	EN ISO 11969	Water quality – Determination of arsenic - Atomic absorption spectrometric method (hydride technique) (ISO 11969)	yes
Ba	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Cd	ISO 8288	Water quality – Determination of cobalt, nickel, copper, zinc, cadmium and lead – Flame atomic absorption spectrometric methods	yes
	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes

Chloride	ISO 9297	Water quality – Determination of chloride – Silver nitrate titration with chromate indicator (Mohr's method)	yes
	EN ISO 10304-1	Water quality – Determination of dissolved anions by liquid chromatography of ions – Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1)	yes
	EN ISO 10304-2	Water quality – Determination of dissolved anions by liquid chromatography of ions – Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water (ISO 10304-2)	yes
Co	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Cr	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Cr (VI)	ISO 11083	Water quality – Physical, chemical and biochemical methods - Determination of chromium (VI) – Spectrometric method using 1, 5-diphenylcarbazide	yes
Cu	ISO 8288	Water quality – Physical, chemical and biochemical methods - Determination of cobalt, nickel, copper, zinc, cadmium and lead: flame atomic absorption spectrometric methods	yes
	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Mo	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Ni	ISO 8288	Water quality – Physical, chemical and biochemical methods - Determination of cobalt, nickel, copper, zinc, cadmium and lead: flame atomic absorption spectrometric methods	yes
	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Nitrite	EN 26777	Water quality – Physical, chemical and biochemical methods - Determination of nitrite: molecular absorption spectrometric method (ISO 6777)	yes
	EN ISO 10304-1	Water quality – Determination of dissolved anions by liquid chromatography of ions - Determination of fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions - Method for water with low contamination (ISO 10304-1)	yes
	EN ISO 10304-2	Water quality – Determination of dissolved anions by liquid chromatography of ions – Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water	yes

		(ISO 10304-2)	
	EN ISO 13395	Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric detection (ISO 13395)	yes
Pb	ISO 8288	Water quality – Physical, chemical and biochemical methods – Determination of cobalt, nickel, copper, zinc, cadmium and lead: flame atomic absorption spectrometric methods	yes
	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Total sulfur	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Sulfate	EN ISO 10304-1	Water quality – Determination of dissolved anions by liquid chromatography of ions – Determination of fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions - Method for water with low contamination (ISO 10304-1)	yes
	EN ISO 10304-2	Water quality – Determination of dissolved anions by liquid chromatography of ions – Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water (ISO 10304-2)	yes
V	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes
Multi substance method	prEN 16170	Sludge, treated biowaste, and soil – Determination of trace elements by inductively coupled plasma optical emission spectrometry (ICP-OES)	Yes: preferred method
	prEN 16171	Sludge, treated biowaste and soil – Determination of trace elements using inductively coupled plasma mass spectrometry (ICP-MS)	yes
Zn	ISO 8288	Water quality – Physical, chemical and biochemical methods – Determination of cobalt, nickel, copper, zinc, cadmium and lead: flame atomic absorption spectrometric methods	yes
	EN ISO 11885	Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885)	yes

EN 13370			
Parameter	**Number**	**Title**	**applicable**
Ammonium	EN ISO 11732	Water quality – Determination of ammonium nitrogen - Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732	yes
	ISO 7150-1	Water quality – Determination of ammonium - Part 1: Manual spectrometric method	yes
AOX	EN 1485	Water quality – Determination of adsorbable organically bound halogen (AOX	yes
Electrical conductivity	EN 27888	Water quality – Method for the determination of electrical conductivity (ISO 7888)	yes
Hg	EN 1483	Water quality – Determination of mercury	yes
Phenol index	ISO 6439	Water quality – Determination of phenol index - 4-Aminoantipyrine spectrometric methods after distillation	yes
	EN ISO 14402	Water quality– Determination of phenol index by flow analysis (FIA and CFA) (ISO 14402)	yes
TOC	EN 1484	Water analysis - Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)	yes
Cyanide (easily liberatable)	ISO 6703-2	Water quality – Determination of cyanide – Part 2: Determination of easily liberatable cyanide	yes
	EN ISO 14403	Water quality – Determination of total cyanide and free cyanide by continuous flow analysis (ISO 14403)	yes
Fluoride, chloride, bromide, nitrate, sulfate	EN ISO 10304-1	Water quality – Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions, using liquid chromatography of ions – Part 1: Method for water with low contamination (ISO 10304-1)	Yes, preferred method - Multi-substance method
Fluoride	ISO 10359-1	Water quality – Determination of fluoride – Part 1: Electrochemical probe method for potable and lightly polluted water	yes

B.3.10 Eluates: further analytical methods

Parameter	Number	Title	applicable
ammonium nitrogen	EN ISO 11732	Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732)	yes
Cd	EN ISO 5961	Water quality – Determination of cadmium by atomic absorption spectrometry (ISO 5961)	yes
Cr	EN 1233	Water quality – Determination of chromium - Atomic absorption spectrometric methods	yes
Hg	ISO 5666, EN 1483*	Water quality – Determination of mercury	yes
Hg	ISO 17852, EN 13506*	Water quality – Determination of mercury – Method using atomic fluorescence spectrometry	yes
Elements	ISO 17294-1	Water quality – Application of inductively coupled plasma mass spectrometry (ICP-MS) – Part 1: General guidelines	yes
Elements	ISO 15586	Water quality – Determination of trace elements using atomic absorption spectrometry with graphite furnace	yes
Nitrogen	ISO 11905-1	Water quality – Determination of nitrogen – Part 1: Method using oxidative digestion with peroxodisulfate	yes
Nitrite, nitrogen and nitrate nitrogen	EN ISO 13395	Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric (ISO 13395)	yes
Phosphorus	EN ISO 6878	Water quality – Determination of phosphorus - Ammonium molybdate spectrometric method (ISO 6878)	yes
Phosphorus	EN 1189	Water quality – Determination of phosphorus – Ammonium molybdat spectrometric method	yes
* the ISO and CEN methods are technically identical			

Annex C
(informative)

Physical and hydraulic properties

C.1 Introduction

The determination of physical and hydraulic properties of extractive wastes may be done for various purposes, e.g. to analyse input parameters for design of geotechnical structures such as piles and embankments. This annex is presenting the most common methods to obtain parameters that may be used for geotechnical and hydraulic evaluations. The tables summarize the applicable standards, focusing primarily on international and European Standards, or other standards issued in English. The tables are followed by a short description of the discussed physical properties of the material.

For several properties a number of standardized methods are available that refers to different characterized volume, test type (in situ, ex situ, laboratory, etc.) and/or different test speed. In some cases indirect determination methods are also applied in practice (often due to their simplicity, time- or cost efficiency).

C.2 Existing standards

A. Characterization of physical properties			
Classification of soils			
	EN ISO 14688-1:2002	Geotechnical investigation and testing – Identification and classification of soil – Identification and description	Recommended
	EN ISO 14688-2:2004	Geotechnical investigation and testing – - Identification and classification of soil - Principles for a classification.	Recommended
	ASTM D2487	Classification of soils for engineering purposes (unified soil classification system)	
	ASTM D2488	Standard practice for description and identification of soils (visual - manual procedure)	
Granulometry			
Grain size distribution: Sieving and hydrometry	CEN-ISO/TS 17892-4:2004	Geotechnical investigation and testing –Laboratory testing of soils – Particle size distribution	Recommended
	EN 933-1:1997	Tests for geometrical properties of aggregates - Sieving method	
	ASTM D422-63	Standard test method for particle-size analysis of	
	ASTM D6913	Particle size distribution (gradation) of soils using sieve analysis	
	BS 1377-2	Methods of test for soils for civil engineering purposes – Classification tests	
	BS 1337	Structural bearings	

	AASHTO T88	Standard method of test particle size analysis of soils	
(sieves)	ISO 3310-1:2000	Test sieves – Technical requirements and testing - Test sieves of metal wire cloth.	Recommended
	EN933-2:1995	Determination of particle size distribution. Tests sieves, normal size of apertures	Recommended
	ASTM E11	Standard specification for woven wire test sieve cloth and test sieves	
(hydrometers)	BS 812-103.2	Testing aggregates – Method for determination of particle size distribution - Sedimentation test	
	ASTM C88	Standard test method for soundness of aggregates by use of sodium sulfate or magnesium sulfate	
	AASTHO T104	Standard method of test for soundness of aggregate by use of sodium sulfate or magnesium sulfate	
Moisture content, plastic soil behaviour and soil index properties			
Moisture content, water content	CEN-ISO/TS 17892-1:2004	Geotechnical investigation and testing – Laboratory testing of soil – Determination of water content	Recommended
	EN 1097-5:2008	Tests for mechanical and physical properties of aggregates – Determination of the water content by drying in a ventilated oven.	Recommended
	ASTM D2216	Laboratory determination of water (moisture) content of soil and rock by mass.	
	EN 12880:2000	Characterization of sludges – Determination of dry residue and water content.	Recommended
	BS 812-109	Testing aggregates – Methods for determination of moisture content	
	BS 1377-2:1990	Methods of test for soils for civil engineering purposes – Classification tests	
	BS 1377-4	Methods of test for soils for civil engineering purposes – Compaction-related tests tests	moisture condition value
Moisture content, indirect, rapid method	ASTM D4944	Standard test method for field determination of water (moisture) content of soil by the calcium carbide gas pressure tester method	
	AASHTO T217	Determination of moisture in soils by means of calcium carbide gas pressure moisture tester	
Plasticity tests	CEN-ISO/TS 17892-12:2004	Geotechnical investigation and testing – Laboratory testing of soil – Determination of Atterberg limits	Recommended
	CEN ISO/TS 17892-6:2004; CEN ISO/TS	Geotechnical investigation and testing – Laboratory testing of soil – Fall cone test	Recommended

	ASTM D4318	Standard test methods for liquid limit, plastic limit, and plasticity index of soils	
	ASTM D427	Standard test method for shrinkage factors of soils by the mercury method	Not recommended (use of Hg)
	ASTM D4943	Shrinkage factors of soils by the wax method	
	BS 1377-2	Methods of test for soils for civil engineering purposes – Classification tests	
	AASHTO T90	Standard method of test for determining the plastic limit and plasticity index of soils	
	AASHTO T92	Standard method of test for determining the shrinkage factors of soils	
Soil density, void ratio			
Particle density	CEN-ISO/TS 17892 - 2:2004	Geotechnical investigation and testing – Laboratory testing of soil – Determination of density of fine-grained soil	Recommended
	CEN-ISO/TS 17892 - 3:2004	Tests for mechanical and physical properties of aggregates – Determination of particle density. Pycnometer method	Recommended
	ASTM D854	Standard test methods for specific gravity of soil solids by water pycnometer	
	BS 1377-2	Methods of test for soils for civil engineering purposes – Classification tests	
	AASHTO T100	Standard method of test for specific gravity of soils	
Bulk density, void ratio	EN 1097-6:2000; EN 1097-6:2000/A1:2005	Tests for mechanical and physical properties of aggregates – Determination of particle density and water absorption	Recommended
	EN 1097 - 3	Tests for mechanical and physical properties of aggregates – Determination of loose bulk density and voids	Recommended
	EN 1097-7	Tests for mechanical and physical properties of aggregates – Determination of the particle density of filler - Pycnometer method	Recommended for soils d < 0,063 mm
	BS 1377-2	Methods of test for soils for civil engineering purposes – Classification tests	
	ASTM C127-88	Standard test method for density, relative density (specific gravity), and absorption of coarse aggregate	for coarse aggregates
Maximum, minimum density	ASTM D1556	Standard test method for density and unit weight of soil in place by sand-cone method	Recommended for maximum density
	ASTM D4254	Minimum index density and unit weight of soils and calculation of relative density	
	ASTM D4253	Maximum index density and unit weight of soils using a vibratory table	Vibratory table not common, for alternative inhouse method see [Germaine, 2009]
	BS 1377-4	Methods of test for soils for civil engineering purposes – Compaction-related tests tests	maximum / minimum density

Bulk density field determination	ASTM D2167-08	Standard test method for density and unit weight of soil in place by the rubber balloon method balloon density apparatus	Balloon density apparatus, Not recommended for sharp-edge particles, and soft, easily deforming soils
	ASTM D2216	Standard test methods for laboratory determination of water (moisture) content of soil and rock by mass	
	BS 1377-9	Methods of test for soils for civil engineering purposes: in-situ tests	in situ density tests - sand replacement method
	AASHTO T191	Standard method of test for density of soil in-place by the sand-cone method	
	SS 27110	Geotechnical tests – Compaction properties - Field determination of density	
Porosity / void ratio	SS 27117	Geotechnical tests – Void ratio and porosity	
B.) Characterization of mechanical properties			
Compactibility / Proctor	EN 13286-2:2010	Unbound and hydraulically bound mixtures – Test methods for the determination of the laboratory reference density and water content. Proctor compaction	Recommended (up to 25 % > 63 mm)
	ASTM D698	Standard test methods for laboratory compaction characteristics of soil using standard effort (12 400 ft-lbf/ft3 (600 kN-m/m^3))	
	ASTM D1557	Standard test methods for laboratory compaction characteristics of soil using modified effort (56,000 ft-lbf/ft3 (2,700 kN-m/m^3))	
	BS 1377-4	Methods of test for soils for civil engineering purposes – Compaction-related tests	dry density / moisture content relationship
	ASTM D4718	Correction of unit weight and water content for soils containing oversize particles	
	AASHTO T99	Standard method of test for moisture-density relations of soils using a 2,5 kg (5.5-lb) rammer and a 305 mm (12-in.) Drop	
	SS 27108	Geotechnical tests – Compaction properties	
Shear strength and bearing capacity			
Direct shear tests	CEN/ISO TS 17892-10:2004; CEN/ISO TS 17892-10:2004/AC:2005	Geotechnical investigation and testing – Laboratory testing of soil – Direct shear tests	Recommended
	BS 1377-7	Methods of test for soils for civil engineering purposes – Shear strength tests (total stress)	
	BS 1377-8	Methods of test for soils for civil engineering purposes – Shear strength tests (effective stress)	

	ASTM D3080	Standard test method for direct shear test of soils under consolidated drained conditions	
Unconfined compressive strength	CEN/ISO TS 17892-7:2004	Geotechnical investigation and testing – Laboratory testing of soil – Unconfined compression test on fine-grained soils	Recommended
	ASTM D2166	Unconfined compressive strength of cohesive soil	
Triaxial test of unconsolidated undrained conditions l	CEN ISO/TS 17892-8:2004	Geotechnical investigation and testing – Laboratory testing of soil - Unconsolidated undrained triaxial	Recommended
	ASTM D2850	Standard test method for unconsolidated-undrained triaxial compression test on cohesive soils	
	AASHTO T296	Standard method of test for unconsolidated, undrained compressive strength of cohesive soils in triaxial compression	
Triaxial test, consolidated undrained or drained conditions	CEN ISO/TS 17892-9:2004	Geotechnical investigation and testing – Laboratory testing of soil – Consolidated triaxial compression test on water saturated soils	Recommended
	ASTM D4767	Standard test method for consolidated undrained triaxial compression test for cohesive soils	
	EN 13286-7	Unbound and hydraulically bound mixtures – Cyclic load triaxial test for unbound mixtures	Recommended e.g. for seismicity test
	ASTM D3999	Standard test methods for the determination of the modulus and damping properties of soils using the cyclic triaxial apparatus	e.g. for seismicity test
	AASHTO T297	Standard method of test for consolidated, undrained triaxial compression test on cohesive soils	
Oedometer	CEN ISO/TS 17892-5:2004	Geotechnical investigation and testing – Laboratory testing of soil. Incrementing loading oedometer test	Recommended
	BS 1377-5	Methods of test for soils for civil engineering purposes – Compressibility, permeability and durability tests	one-dimension consolidation properties, oedometer
	AASHTO T216	Standard method of test for one-dimensional Consolidation Properties of soils	
	ASTM D2435	Standard test methods for one-dimensional consolidation properties of soils using incremental loading	
	ASTM D4546	Standard test methods for one-dimensional swell or collapse of cohesive soils	Smectite-bearing soils
	BS 1377-5	Methods of test for soils for civil engineering purposes. Compressibility, permeability and	swelling and collapse characteristics
	SS 27126	Geotechnical tests - Compression properties - Oedometer test, CRS-test - Cohesive soil	
Bearing capacity tests			
CBR (California Bearing Ratio) test	EN 13286-47	Unbound and hydraulically bound mixtures – Test method for the determination of California bearing ratio, immediate bearing index and linear swelling	Recommended
	BS 1377-4	Methods of test for soils for civil engineering purposes – Compaction-related tests tests	laboratory CBR
	ASTM D1883	Standard test method for CBR (California bearing ratio) of laboratory-compacted soils	

	AASHTO T193	Standard method of test for the California bearing ratio	
Field test methods			
Plate bearing tests	DIN 18134	Determining the deformation and strength characteristics of soil by the plate loading test	
	BS 1377-9	Methods of test for soils for civil engineering purposes: in-situ tests	in situ vertical deformation and strength by plate loading test
	ASTM D1194	Standard test method for bearing capacity of soil for	
	ASTM D1195	Standard test method for repetitive static plate load tests of soils and flexible pavement components, for use in evaluation and design of airport and highway pavements	Designed for pavements.
	ASTM D1196	Standard test method for non-repetitive static plate load tests of soils and flexible pavement components, for use in evaluation and design of airport and highway pavements	Designed for pavements.
Dynamic plate test	TP BF-StB B 8.3 version 2003	Dynamic plate loading test with the aid of the light falling weight deflectometer	Important method without applicable standard, only technical specifications are available. This method is with quickly increasing importance.
	ZTV E-StB 09,	Additional technical contract terms and guideline for digging on road constructions	
	T-StB 95	Additional technical contract terms and guideline for base layers during digging earthworks	
	T-StB 9,	Additional technical contract terms and guideline for excavation on road traffic areas	
	RVS 08.03.04 March 2008	Compaction tests by means of the dynamic plate test	
	RIL 836, Deutsche Bahn AG	Guideline for the use of the light falling weight deflectometer during railway works	
Field vane shear test	CEN-ISO/TS 22476-9	Geotechnical investigation and testing – Field testing – Field vane test	Recommended
	BS 1377-9	Methods of test for soils for civil engineering purposes: in-situ tests	in situ vane shear test
Dynamic soundings, SPT (Standard penetrometer testing)	EN ISO 22476-3:2005	Geotechnical investigation and testing – Field testing – Standard penetration test	Recommended
	EN ISO 22476-2:2005	Geotechnical investigation and testing – Field testing – Dynamic probing	Recommended
	ASTM D1586-08	Standard test method for standard penetration test (SPT) and split-barrel sampling of soils	
	BS 1377-9	Methods of test for soils for civil engineering purposes: in-situ tests	dynamic probing
	BS 1377-9	Methods of test for soils for civil engineering purposes: in-situ tests	standard penetration test

Static soundings, CPT (Cone penetrometer testing)	EN ISO 22476-12:2009	Geotechnical investigation and testing – Field testing – Mechanical cone penetration test (CPTM)	Recommended
	CEN-ISO/TS 22476-10	Geotechnical investigation and testing – Field testing – Weight sounding test	Recommended
	ASTM D6067 - 10	Standard guide for using the electronic cone penetrometer for environmental site characterization	
	BS 1377-9	Methods of test for soils for civil engineering purposes: in-situ tests	cone penetration test
in situ CBR (California Bearing Ratio)	BS 1377-9	Methods of test for soils for civil engineering purposes: in-situ tests	in situ CBR
Hydraulic conductivity and permeability			
Hydraulic conductivity and permeability	CEN ISO/TS 17892-11:2004; CEN ISO/TS 17892-11:2004/AC:2005	Geotechnical investigation and testing – Laboratory testing of soil – Determination of permeability by constant and falling head	recommended
	ASTM D5084	Standard test methods for measurement of hydraulic conductivity of saturated porous materials using a flexible wall permeameter	
	ASTM D2434	Standard test method for permeability of granular soils (constant head);	
	BS 1377-5	Methods of test for soils for civil engineering purposes - Compressibility, permeability and	OK permeability by the constant head method
	BS 1377-6	Methods of test for soils for civil engineering purposes - Consolidation and permeability tests in hydraulic cells and with pore pressure measurement	OK determination of permeability in a hydraulic consolidation cell
	AASHTO T215	Standard method of test for permeability of granular soils (Constant Head)	
	DIN 18130	Laboratory tests for determining the coefficient of permeability of soil	
subsurface flow parameters	ASTM D5126	Standard guide for comparison of field methods for determining hydraulic conductivity in vadose zone	

The following sub-clauses give a brief explanation of the listed methods/properties.

C.2.1 Physical properties

C.2.1.1 Granulometry

The aim is to determine the grain-size (*d*) distribution curve of the tested material. This is usually done by sieving and/or sedimentation tests. Based on the curve, the partitions of gravel, sand, silt and clay can be determined, on which the soils geotechnical classification and mechanical nomenclature are based. Moreover, based on the shape of the curve and the relation between given points on the curve the behaviour of the material can be characterized. Based on the grain size distribution the following coefficients may be calculated:

The uniformity coefficient ($C_u = d_{60}/d_{10}$), and the curvature coefficient $C_c = (d_{30})^2/(d_{60} * d_{10})$ give information on the heterogeneity of the soil in terms of grain size, where d_x is the grain size indicated on the grain size distribution curve at *x*-percent passing level. The more heterogeneous the material is ("well-graded", $C_u >$ 6, $C_c >$ 3) the better it can be compacted and the higher strength in the drained state can be achieved. In case the material is "low-graded" (high homogeneity) the compatibility is low and in sands and silts with high moisture content (i.e. low degree of compaction) liquefaction is possible.

The grain size distribution curve can be obtained by sieving to a minimum grain size of 0,063 mm and by means of sedimentation procedure for particle sizes smaller than 0,063 mm.

C.2.1.2 Moisture content, plastic soil behaviour and soil index properties

Moisture content (w) in geotechnics is defined as the ratio of the mass of pore water related to mass of the solid soil skeleton. It is determined by weighing a sample before and after drying. $w = (m_0 - m_s)/ m_s$, where m_0 and m_s is the weight of the sample in wet and dry conditions respectively. Its value therefore may be above 100 %. It should be noted that in the context of environmental tests and analyses it is customary to express both the moisture content and the dry content as a percentage or fraction of the total (wet) mass of the sample (i.e. $w = (m_0 - m_S)/m_0$). This definition is for instance used in all the leaching standards produced by CEN/TC 292 and in EN 15875 (the static acid/base potential accounting test).

For a classification of fine-grained cohesive materials and to describe their behaviour, the Atterberg limits are generally used. Since the remoulded material behaviour (rigid, plastic or liquid) is moisture content dependent, the soil properties are defined by limits between these states (shrinkage (w_S), plastic (w_P) and liquid (w_L) limits) and these limits together with the moisture content of the soil serve as a base for soil classification.

From the limit values different index values can be calculated: The plasticity index (I_p) is a measure of the sensitivity of the soil properties for a change in moisture content:

$$I_p = w_L - w_P \qquad \text{(C.1)}$$

A high I_p value indicates a clay, low values characterize silts, cohesionless soils like sands have no plasticity index.

The consistency index, (I_C) and liquidity index (I_L):

$$I_c = (w_L - w)/(w_L - w_P) \qquad \text{(C.2)}$$

$$I_L = (w - w_P)/(w_L - w_P) \qquad \text{(C.3)}$$

show the current state of the soil. The consistency index is used for relatively stiff soils with low water contents whereas the liquidity index is used for loose soils with high water contents.

The plastic limit is determined by rolling the material into a thin thread by hand. The definitive method of determination of the liquid limit is the cone penetration test in which the sample is tested for a range of moisture contents.

C.2.1.3 Soil density and compatibility

The particle density and specific gravity (ρ_s) of the soil can be determined using a pycnometer. The bulk density of cohesive soils can be measured in laboratory using different methods, and also in situ. The bulk density of less cohesive soils has to be determined in situ. For in situ measurements, sand replacement tools or a balloon density apparatus can be used. In all cases both the wet, ρ, and dry, ρ_d, densities should be calculated from the weight of the extracted sample. The parameters are calculated from the measured volume and the actual moisture content using the following formula:

$$\rho_0 = \rho_n/(1+w_n) \qquad \text{(C.4)}$$

The compatibility of the sediments and the tailings is moisture dependent. Very wet and very dry materials are hardly compactable, but between the two extreme situations there exists an optimum moisture content where the material is most compactable using a given standardized energy. The compatibility is measured by the densities of the soil sample compacted at different moisture contents. To evaluate the optimum water content and the maximum dry density, the density values are plotted vs. water content (Proctor-chart).

The applied methods differ in compaction energy (height and weight of falling compactor, number of impacts) and in the size of the compaction mould.

C.2.1.4 Shear strength and bearing capacity

The aims of the below listed tests are to characterize the strength of the tested materials, to determine internal friction, cohesion, and different elastic and plastic parameters of the soils, and to characterize the deformations at different conditions. All the tests aim to describe the material parameters used in the recognized constitutive laws as Mohr-Coulomb, Drucker-Prager, Cam-Clay, etc.

Knowing the mentioned parameters the stability of slopes, the resistance against failures in different directions and the subsidence of surfaces can be analyzed and calculated.

In a triaxial test a cylindrical sample (core or artificially produced sample of loose material is mounted in a cell where a supporting side pressure from radial direction and an axial force is applied. During the test both axial and radial deformations are possible and they are measured together with volume change. As a result, the void ratio changes at pre-defined stress paths and the triaxial strength at different radial pressures can be determined. From the results, e.g. the internal friction, cohesion and plastic and elastic cam-clay parameters can be calculated. In case only the air pressure acts in the cell, unconfined tests to measure the uniaxial unconfined strength of the sample can also be performed. Triaxial tests can be performed as “quick” or “consolidated” tests: the difference being if the sample is allowed to consolidate for the supporting side pressure and initially applied axial force. The consolidated tests can further be performed as drained or undrained tests, the difference being if the sample is allowed to successively consolidate with the increasing axial stress during the test or if the volume is kept constant during this stage. In the latter case the change in pore water pressure is measured during the test

In an oedometer test the sample is inserted into a rigid cylindrical ring and compressed axially. The test can be performed incrementally, whereby the load is applied in steps and the axial deformation is measured at predefined time intervals after each load application, or as automatic tests in which the sample is compressed continuously and the load, deformation and the pore pressure in the sample are measured at regular time intervals. The oedometer test gives information about the consolidation behaviour of the sample, and also some of the elastic and plastic parameters in different constitutive laws can be derived from the results.

The CBR (California bearing ratio) test was developed for testing highway basements where a compacted or natural surface is loaded with a cylindrical plate and the forces and deformations are simultaneously recorded. In the laboratory a CBR mould is used with no lateral deformations, in situ the lateral deformations are unlimited during the test. The test results should be modified to correlate to each other.

At in situ plate bearing tests different diameter rigid steel plates are used and pressed onto the ground surface with different loads causing sinking of the plate and ultimate failure in the ground. From test results the internal friction, the cohesion and the Young-modulus can be calculated. These test methods are used to estimate the bearing capacity of a soil under field loading conditions for a specific loading plate and depth of embedment (ASTM D1194).

A dynamic plate test can be performed using a light drop-weight tester. The equipment enables a fast and simple determination of the soil bearing capacity and compaction quality of non-cohesive soils and basements. It can also be used for soil improvement applications. During measurement a dropped weight causes transient stress increases in the ground. The mean settlements and the dynamic values (dynamic modulus of deformation), E_{vd}, of soils in the range E_{vd} = 15 MN/m^2 to 80 MN/m^2 can also be determined.

The shearing resistance of both stiff and loose materials can be determined using direct and simple shear tests. At direct shear, a prescribed surface to be sheared is applied, whereas at simple shear the weakest surface will be sheared. Since the technical realization of simple shear is more difficult to achive, the direct shear approach is more widely used. In this device a normal load is applied to a sample confined in a cell and shear perpendicular to the load is induced by fixing half of the sample and moving the other half. The shear force is measured and peak and residual shear stresses are calculated knowing the sheared area. From the results, cohesion and internal friction values can be determined. The size of the sample may vary within wide ranges, normally from 0,01 m^2 to 1 m^2 square shear surfaces: for coarse and/or inhomogeneous materials large sample sizes are recommended.

To determine the shear strength of cohesive materials in situ, the field vane shear test is widely used. The equipment has a special cross shaped measuring head (vane) which is stuck into the soil/tailing and then rotated. The peak torque needed to rotate the vane head is proportional to the undisturbed shear resistance, and from the static torque needed for continuous rotation after a number of turns the remoulded (or residual) strength can be estimated.

Dynamic (SPT, standard penetrometer testing) and static (CPT, cone penetrometer testing) soundings are also suitable to perform comparative investigations of waste disposal sites.

At dynamic SPT tests, a given weight falls from a standard height onto a rod attached to a core sampler, and the advance of the rod and sampler is measured. The different natural and artificial formations provide different resistance against the penetration of the sampler, therefore the method is suitable to determine layering and stiffness of the soil. The method also provides samples of the soil, which can be used for further classification in the laboratory.

Cone penetrometer testing (CPT) is a method of providing continuous data with depth for use in characterizing the subsurface and evaluation of soil properties. It consists of a steel penetrometer with a cone tip that is pushed into the ground at a constant rate of penetration. Sensors in the penetrometer continuously measure and collect data during the penetration. Standard cone penetrometers collect information to classify soil type by using sensors that measure cone-tip resistance, side friction and pore water pressure at the tip. Cone penetrometers are nowadays used also to take pore fluid samples and the CPT equipment and technique has also become a platform for collecting information about a variety of contaminants (petroleum hydrocarbons, organic compounds, metals, radioactive components, soil moisture, etc.).

C.2.2 Hydraulic properties

C.2.2.1 Porosity, void ratio, saturation

Porosity is the portion of pore volume, V_p, in the total volume, V_t, of the samples ($n = V_p/V_t$). The void ratio can be calculated by dividing the pore volume, V_p, by the volume of the solid particles in the soil skeleton, V_s, ($e = V_p/V_s$). The two values are interrelated $n = e/(1+e)$ and $e = n/(1-n)$. The porosity and the void ratio give information on the degree of compaction (the smaller they are the better the soil is compacted) and also on the soils resistance against water migration (i.e. the hydraulic conductivity). The degree of saturation, S, describes the portion of pore volume that is filled with pore fluids. When S=1 the sample is fully saturated. Effective porosity (n_0) describes the pore volume portion of the full sample volume where gravity driven flow can occur (open pores).

C.2.2.2 Hydraulic conductivity and permeability

Intrinsic permeability $k[L^2]$ and hydraulic conductivity $K[L/T]$ describes the ability of water migration in the soil or tailings. The hydraulic conductivity, K, depends on the soil grain size, the structure of the soil matrix, the type of soil fluid, and the saturation. The important properties relevant to the matrix of the soil include pore size distribution, pore shape, tortuosity, specific surface, and porosity. In relation to the soil fluid, the important properties include fluid density, ρ_f, and fluid dynamic viscosity, μ. For a saturated sample, the hydraulic conductivity permeability relation is $K = k\rho g/\mu$, the less saturated the samples are, the lower is the hydraulic conductivity. At a given degree of saturation no more flow occurs, $K = 0$.

The measurement of hydraulic conductivity and permeability can be performed using constant head permeameter (coarse material: gravels, sands) or falling head or flexible wall permeameters (fine materials.) In situ testing of permeability can be done using infiltrometers and pumping or infiltration tests in boreholes or wells.

C.2.2.3 Subsurface flow parameters

Subsurface flow is directed by the flow potential, which is directly related to the h piezometric level in the subsurface waters. To determine the flow direction and speed, monitoring wells should be established in which the piezometric levels should be measured using water level indicators to create hydrographs in relation to a given reference level. The first derivative (the slope) of the subsurface piezometric potential field called hydraulic gradient, i, shows the direction of the flow. The speed of flow can be calculated using the Darcy law: $v = K \cdot i/n_0$, where n_0 is the effective porosity.

The amount of water which can be stored in a unit of aquifer volume or in a unit area of the aquifer is described by specific storage coefficient, S_s, and storage coefficient, S. $S = S_s \cdot m$, where m is the thickness of the aquifer. Specific yield is the volume portion of the total volume that can be released at a lowering of the subsurface water level. The storage coefficient and the specific yield are key parameters for time dependent water storage both in mining dumps and tailings.

Annex D
(informative)

Leach tests

D.1 Leach tests

This annex presents an evaluation of the applicability to extractive waste of existing standard (CEN and ISO) leaching tests. The procedure for this evaluation is presented in more detail in Annex B. The table on applicability is followed by brief descriptions of leaching test procedures. For detail about the methods, the reader is referred to original documents found as references for each of the method.

D.2 Applicability of leaching tests

Parameter / Method	Number	Title	Applicable	Comment
Upflow percolation	CEN/TS 14405	Characterization of waste – Leaching behaviour tests – Upflow percolation test	yes	Provide the basis for impact assessment by percolation. Very low liquid to solid ratio (L/S) matches porewater composition. No kinetic information.
Influence of pH	CEN/TS 14429	Characterization of waste – Leaching behaviour tests – Influence of pH on leaching with initial acid/base addition	yes	pH dependence test results in combination with speciation modelling provide an understanding of the release controlling phases (mineral or sorptive phases). Identification and quantification of potential acid neutralizing minerals. No kinetic information.
continuous pH control	CEN/TS 14997	Characterization of waste – Leaching behaviour tests – pH dependence test with continuous pH control	yes	Same as CEN/TS 14429, except for computer controlled acid/base addition
Compacted granular leach test	WI 003510 09	Compacted granular leach test for granular materials with release behaviour dominated by diffusion	yes	Relevant in special cases with very fine grained tailings
Acid and base neutralization capacity	CEN/TS 15364	Characterization of waste – Leaching behaviour testes – Acid and base neutralization capacity test	yes	
One stage:	EN 12457-1 *	Characterization of waste – Leaching – Compliance test for leaching of granular waste	(yes)	The four parts of EN 12457 are also applicable to wastes from the

liquid/ solid ratio 2 l/kg < 4 mm		materials and sludges – Part 1: One stage batch test at a liquid to solid ratio of 2 l/kg with particle size below 4 mm (without or with size reduction)		extractive industries.
one stage: liquid/ solid ratio 10 l/kg < 4 mm	EN 12457-2 *	Characterization of waste – Leaching – Compliance test for leaching of granular waste materials and sludges – Part 2: One stage batch test at a liquid to solid ratio of 10 l/kg with particle size below 4 mm (without or with size reduction)	(yes)	See EN 12457-1
Two stages: liquid/ solid ratio 2 l/kg and 8 l/kg < 4 mm	EN 12457-3 *	Characterization of waste – Leaching; Compliance test for leaching of granular waste materials and sludges – Part 3: Two stage batch test at a liquid to solid ratio of 2 l/kg and 8 l/kg for materials with high solid content with particle size below 4 mm (without or with size reduction)	(yes)	See EN 12457-1
One stage: liquid/ solid ratio 10 l/kg < 10 mm	EN 12457-4 *	Characterization of waste – Leaching – Compliance test for leaching of granular waste materials and sludges – Part 4: One stage batch test at a liquid to solid ratio of 10 l/kg with particle size below 10 mm (without or with limited size reduction)	(yes)	see part 12457-1
One stage. basket < 32 mm	EN 1744-3	Test for chemicals properties of aggregates – part 3: preparation of eluate by leaching of aggregates	no	This method uses particles 0 mm to 32 mm with a different technique (stirring the sample stored in a basket instead of shaking). Results of this test will be different to the ones from other methods listed in this clause. In CEN/TC 351 alternative approach to coarse granular material in preparation. Relevant for assessment of waste rock.

*Methods used for quick verification against quality objectives. An alternative is to use one step from the more elaborate tests to demonstrate compliance or conformity with prior information. A single number without context can be very misleading. Placing verification or QC data in context with more detailed information enhances the conclusions that can be drawn from s quick test.

D.3 Leaching test procedures

D.3.1 pH-dependence leaching test

The pH dependence leaching tests (CEN/TS 14429, CEN/TS 14997) provide information on the pH sensitivity of leaching behaviour of the material. The test consists of a number of parallel extractions of a material at a liquid/solid ratio (L/S) of 10 (l/kg) for 48 h at a series of pre-set pH values. Since pH is one of the main leaching controlling parameters, the information can be used to evaluate the repeatability in testing (resulting from measurement at steep concentration - pH slopes) and to provide information on the sensitivity to pH in specific field scenarios. The changes in mineral solubility and sorption resulting from acidification are captured by this method but it does not give information on the weathering process leading to changes in pH and solubility. For this type of information, repeated testing is needed on the same material exposed to weathering for different duration. The acid neutralization capacity (ANC) that can be derived from the test is a useful property in this respect. In combination with geochemical speciation modelling (van der Sloot and van Zomeren, 2010) it provides insight in minerals controlling acid neutralization behaviour other than carbonates. For material characterization this has been proven to be a very useful method [van der Sloot, 1997; van der Sloot et al, 2008; Dijkstra et al, 2006; Carter et al, 2008, van der Sloot et al, 2007]. The method is standardized in two experimental modes by CEN/TC 292/WG 6 (CEN/TS 14429 and CEN/TS 14997). It has been standardized for soil and soil related materials (CEN-ISO/TS 21268-4). US-EPA has adopted the method as EPA method 1313 for inclusion in SW 846. (During 2011 the method is validated by intercomparison studies with different participating laboratories to obtain performance characteristics of the method; in parallel the European methods are tested, which allows conclusions on the comparability of the methods by the end of 2011).

D.3.2 Column leaching test

Column leaching test CEN/TS 14405 percolation test has been developed at European level for waste. It is a column test in which seven eluate fractions are collected within the range of L/S = 0,1 l/kg to 10 l/kg. The total test duration is approximately 21 days. The leachant is demineralized water (DMW). The test material is applied as received (Ø 1 cm) and up-flow (14 ml/h) is applied through a column waste height of 28 cm and a diameter of 10 cm. The low L/S condition gives an indication of pore water concentrations, which are relevant in field conditions with relatively low infiltration. A very similar method has been developed for soil and soil related materials (CEN-ISO/TS 21268-3). US-EPA has adopted the method as EPA method 1314 for inclusion in SW 846. (This method is also being validated and in parallel the European method has been carried out, which by the end of 2011 will allow a conclusion on the comparability).

D.3.3 Dynamic monolith leaching test

Dynamic monolith leach test CEN/TS 15863 specimen is subjected to leaching in a closed tank. The leachant is renewed after 8 h and 1; 2,25; 4; 9; 16; 36 days at a leachant to product volume ratio (L/V) of approximately 5. The results are expressed in mg/m^2. This test is a procedure to evaluate the release from monolithic material by predominantly diffusion control (e.g. exposure of structures to external influences). The distinction is necessary, as the transport limitations set by a solid form result in a significantly lower environmental impact than derived from crushed material. This condition is valid as long as the product maintains its integrity. US-EPA has adopted the method as EPA method 1315 for inclusion in SW 846. (This monolith leach test is also being validated and in parallel the European method CEN/TS 15863 will be carried out, which by early 2012 will allow a conclusion on the comparability of the US-EPA and European methods).

D.3.4 Compacted granular leaching test

The compacted granular leaching test NEN 7347 resembles the tank leach test for monolithic materials in the way it is carried out and the data are handled. The method is designed for granular materials that behave as a monolith in the scenario under investigation (e.g. clay lens in a sandy soil). US-EPA has adopted this method as part of EPA method 1315 for inclusion in SW 846. (the procedure is being validated in the US).

D.3.5 Sequential extraction

Sequential extraction methods were established several decades ago in order to evaluate trace element availability in soils both for exploration purposes and agriculture uses (Chao and Zhou, 1983; Tessier et al, 1979; Sondag, 1981). It has recently been adapted to mine waste investigations (Dold, 2003, 2009; Moritz et al. 2009; Walder et al, 2004) but is still not very commonly used.

Sequential extraction is an operationally defined method to evaluate element leachability relative to their location within the material, whether the elements are sorbed to other minerals or occur as separate minerals. The result can be used to evaluate secondary mineralogy as a product of sulphide oxidation, and sorption of hazardous elements in mine material, based on the chemical composition of gradually stronger leach solutions starting with de-ionized water ending with a mix of four acids for total digestion (Table D.1). A seven step procedure has been defined (Ribet et al, 1995; Dold, 1999, 2003). These seven steps are as follows:

Table D.1 — Sequential extraction steps based on B. Dold Ph.D. Thesis, 1999. A detailed description of the procedure can be found in Dold (1999 and 2001) and the reference included therein

Step	Purpose	Rinse solution
1	Water soluble fraction	Rinse in deionized water
2	Exchangeable fraction	Ammonia acetate rinse
3	Ferric (Fe(III)) oxy-hydroxides	Ammonia oxylate rinse (darkness)
4	Fe(III) oxides	Heated Ammonia oxylate rinse
5	Organics and secondary Cu sulfates	Heated hydrogen peroxide rinse
6	Primary sulphides	$KClO_3$+ HCl and then HNO_3 rinse
7	Residual	$KClO_3$ + HCl +HF + HNO_3 rinse

The result will give a good indication of what type of conditions that are needed to leach different constituents. For example: if the test result indicates that copper is only released in step 6, the material has to oxidize to be able to release copper. On the other hand if it is released in step 3, a change in pH may be enough to release the copper. The method is however, not useful if the purpose is to calculate metal loading or runoff water quality.

The sequential extraction methods still needs more adjustments to be used widely for characterization of mine waste material. The segregation of mineral dissolution in different types of leach solution is not well enough tested for a variety of mining material. It may still give an indication of the availability of elements under different conditions, but is not a stand-alone test.

D.3.6 Field tests

In-field reaction tests may be the most appropriate test method to use for prediction of acid generation and metal leaching. This group of tests includes simulated rainfall leach tests performed in the field on waste rock or rock surfaces, pilot scale tests set up in field conditions in a controlled environment, lysimeter studies and field scale investigations.

The simulated rainfall test can be performed by adding a measured amount of water spread on a defined area (e.g. on a waste rock dump or pitwall), thereby, simulating a rainfall (Walder et al, 1997). The water is collected at the base of the designated surface area and analysed for the constituents of interest, as a minimum pH, TDS, sulfate, iron, and alkalinity.
Continuous monitoring of runoff water from rock surfaces may be difficult, but in some cases it may be feasible to install a more permanent water collection system. Sampling should then be performed after each rainfall event.

Lysimeter studies and field scale investigations may be the type of testing that is closest to practice, as they are carried out under natural exposure conditions. This type of tests need careful planning and would normally be run over long periods of time (years) to allow for natural processes in the waste to develop.

There are many influencing factors at this larger scale of testing. Combining test results at different scales of testing (laboratory, lysimeter and field scale) may be a way to combine the benefit of the repeatability in the laboratory test and the higher relevance of full scale performance at the larger scale of testing (van der Sloot et al, 2008; van Zomeren et al, 2005; van Zomeren et al, 2011).

Field tests are also discussed in CEN/TR 16363 on kinetic testing of sulfidic waste.

D.3.7 Short term tests

Short tests or compliance test, as they are called in the waste sector, are mostly single step tests, that by themselves give a very limited answer to the question of release from waste. In a tiered approach (Kosson et al, 2002; EN 12920) consisting of a hierarchy of tests, these type of tests are very functional as they provide in conjunction with prior characterization testing a cost effective and comprehensive data management system

EN 12457 - compliance test for granular materials EN 12457 has been validated (van der Sloot et al, 2001). EN 12457-1 and -2 are single step procedures. Part 3 consists of two extraction steps at L/S = 2 and subsequently at cumulative fraction L/S = 10. Outside Europe, TCLP [US EPA 1311, 1986, 1990], has been used for assessing impact. The TCLP has been designed for co-disposal in municipal solid waste landfills and is therefore not applicable for mine waste material.

Leach test for aggregates EN 1744-3 as developed in CEN/TC 154 Aggregates. This method consists of a leaching of the intact aggregate at a liquid to solid ratio of 10 l/kg for 24 h. For this purpose the aggregate is placed on a grid above a magnetic stirrer. No particle size requirements are provided to limit the contribution of fines. The data cannot be linked to impact, which is a serious limitation for its application.

Alternative compliance procedures are single steps derived from the full characterization tests (e.g. first fraction of percolation test). The advantage of such approach is that when the full method is validated, the compliance procedure is implicitly validated. In addition, there will be no debate on the comparability of the methods, as they are essentially the same.

Annex E
(informative)

Mineralogical analysis

In Annex E, the main analytical methods for assessing mineralogy / mineral-chemistry and acid-base accounting methods are presented.

E.1 Mineralogical analytical methods

E.1.1 General

Mineralogy should form an integral part of the waste characterization work. It is usually conducted by transmitted and reflected light microscopy, and by various X-ray techniques (XRD, XRF). Although these techniques are commonly used, other techniques like electron probe microanalysis (EPMA), scanning electron microscopy (SEM) and other more specialized methods may also be employed to complement the mineral characterization or element speciation (Table E.1).

It is usually possible to determine the frequency of occurrence of individual minerals within a sample by the examination of a number of fields of view. Quantitative mineralogical analysis by this method is termed modal analysis. Analyses can also be done by computer to determine mineralogical composition from whole rock elemental oxide analysis and knowledge of the chemical composition of mineral species present in a sample. The result of this analysis is called normative composition.

These different analytical techniques should be used step by step, each step entailing time and costs (Figure E.1).

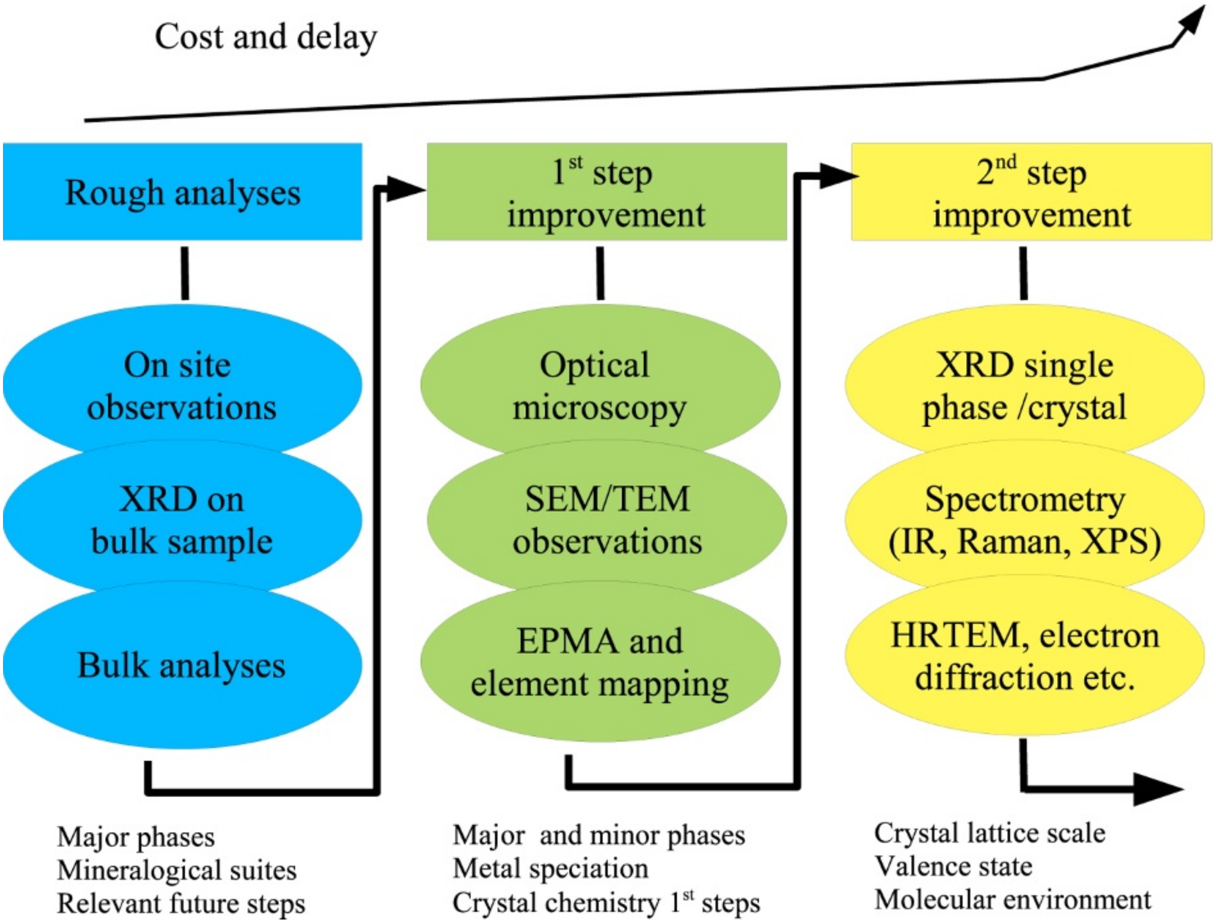

Figure E.1 — Schematic flow chart of major applicable mineralogical analyses for wastes from the extractive industry. The three steps represent increasing level of accuracy, particularity and cost

Table E.1 — List and major characteristics of analytical methods applicable for mineralogical characterization of wastes from the extractive industry in order of complexity and availability

Methods	Support	Obtained data	Specialization	Availability
Transmitted light microscopy	Thin section, covered or polished	Precise determination of translucent minerals, mineral habits, rock texture	University of geology	Easy
Reflected light microscopy	Polished thin section or inclusion	Precise determination of opaque minerals, mineral habits, texture	University of geology with a specialist of metallography	Medium
X-ray diffraction (XRPD)	Powder	Determination of all minerals more than ≈ 5 %, estimation of percentage of minerals in samples by Rietveld method	University, private laboratory	Easy
Scanning electron microscopy (SEM) and Electron probe microanalysis EPMA	Polished thin section, polished section, or grains	Determination of all minerals, mineral habits, texture. Possibility to make chemical micro-mapping, and spot semi-quantitative to quantitative analyses (EDS to WDS spectrometry) and electron diffraction	University of geology	Easy to medium
Thermal analysis differential scanning calorimetry (DSC) and Thermogravimetric analysis (TGA) may be coupled with analyzer of flue gas	Powder	Determination of minerals, water content, sulfate, sulfur, carbonates, carbonate, sulphate, pyrite, organic material in minerals, soil and rocks	University of geology, chemistry	Easy to medium
X-ray diffraction (XRD)	Monocrystal or very weak quantity	Precise determination of a mineral, mineral structure, atom distribution in the network	University of mineralogy, to need Precession and Weissenberg chamber or Debye-Scherrer or Gandolfi cameras or a high sensibility linear detector	Rare
High-resolution transmission electron microscopy (HRTEM)	Powder or microtome sections	Imaging of the crystallographic structure of a sample at an atomic scale	University of geology	Medium
X-ray photoelectron spectroscopy (XPS) or electron spectroscopy for chemical analysis (ESCA)	Polished thin section, or solid surface	Elemental composition of the surface (top 1 nm to 10 nm usually), empirical formula of pure materials, chemical or electronic state of each element in the surface. Alteration products.	University of geology, chemistry	Medium
Optical spectroscopy,	Powder and mineral	$Ti^{(+3)}$, $V^{(+3-+4)}$, $Cr^{(+3-+5)}$, $Mn^{(+2-+3)}$, Fe	University of geology, chemistry, physicist	Easy to medium

visible	surface	$^{(+2-+3)}$, Co, Ni, Cu$^{(0- +2)}$,Al-OH, OH.	or specialist	
Near infrared to infrared (FT-IR method) and Raman spectroscopy	Powder and think sections. But also field assays with portable Infra-Red Mineral Analyser	$AsO_4^{(-3)}$, $PO_4^{(-2)}$, $SO_4^{(-2)}$, $OH^{(-1)}$, H_2O, $CO_3^{(-2)}$, clays, sulphates, crystallinity, polymorphs. Possibility to make mineralogical micro-mapping, and spot species characterization.	University of geology, chemistry, physicist or specialist	Easy to medium
Vertical Scanning Interferometry (VSI)	Polished surface	High resolution image of surface, surface roughness characterization	University of mineralogy, chemistry, physicist or specialist	Rare
Electron paramagnetic resonance	Powder	Site occupancy, distortion, oxidation state, glass structure, radiation defects.	University of geology, chemistry, physicist or specialist	Medium to rare
Nuclear magnetic resonance	Powder	Si, Al, P, $_1H$, $_2H$, Pb, Hg, Zn, Cs, O, Structure, glass, ion exchange, incipient alteration effects.	University of geology, chemistry, physicist or specialist	Medium to rare
Mössbauer spectrometry	Powder	Fe, Sn, Dy, Tm, Eu, Sb, Au. Valence state, site occupancy, local order, magnetic structure.	University of geology, chemistry, physicist or specialist	Medium to rare
EXAFS/XANES (Extended x-ray absorption fine structure /x-ray absorption near-edge Structure)	Powder, liquids	Except light elements. Local structure, oxidation state, interatomic distance, dilutes species, bonding data. Possibility to make chemical valence micro-mapping, and spot species characterization.	Synchrotron, physicist or specialist	Rare

In the following the three most important analytical methods – X-ray powder diffractometry, optical microscopy and electron microscopy techniques – are discussed in some detail, focusing on advantages and limitations for investigations of wastes from the extractive industry. In addition, X-ray photoelectron spectroscopy (XPS), a specific technique which is very effective in investigation of surface alterations, is briefly described.

E.1.2 X-ray powder diffractometer

Powder diffraction methods (XRPD) are used routinely for tasks of mineral phase identification, structure refinement, analysis of crystal strain. In the last 20 years, with computerization XRPD has been brought to a high level of sophistication with analysis, profile fitting, and modelling capabilities (Lüttge and Arvidson, 2008).

One of the most powerful and widespread methods of profile fitting and modelling is the Rietveld refinement. The benefit of this method for extractive waste mineralogy is the best appraisal of the bulk mineralogical composition. This is especially important for identification of small portions of phases (under 5 %). Good quality Rietveld refinement requires cross-checking with additional information on bulk chemistry (atomic absorption or mass spectroscopy) and on chemical composition of individual phases (optical microscopy and/or electron microprobe).

This sophisticated computer-based analytical procedure uses the full information content of the powder pattern, applying least-squares refinements until the best fit is obtained between the entire observed powder diffraction pattern and the entire calculated pattern based on the simultaneously refined models for the crystal structure(s) and other parameters (diffraction optics effects, instrumental factors).

E.1.3 Optical microscopy

Transmitted-light microscopy is used to obtain a petrographic description, and reflected-light microscopy defines the "ore" mineralogy. A primary focus of the microscopy is to confirm and refine the identification of minerals with the XRPD method. The two methods should yield correlative results, therefore diffractometry without microscopy, and vice versa, is not recommended (Jambor and Blowes, 2000).

When the presence of a specific mineral is too low or poorly crystalline to be determinable by the XRPD, or resolvable X-ray lines from several minerals in the sample are overlapping, microscopy is a reliable complementary analytical method. On the other hand, identification of different carbonate mineral phases by optical microscopy is rather ambiguous without justification by XRPD.

Optical microscopy is the basic method for characterization of textural properties of the material, i.e. grain size, the interrelationship between grains and the arrangement of weathering products within individual mineral grains.

E.1.4 Electron microscopy techniques

Scanning electron microscopy (SEM) is a robust micro-analytical tool. It can provide images of the sample surface with 3 to 4 orders of magnitude higher resolution than a conventional light optical microscope. In addition, SEM in combination with EPMA can also provide valuable chemical information of the surface of individual mineral grains.

The interaction of electrons with the surface of the sample yields emission of back-scattered electrons (BSE), secondary electrons (SE) and characteristic X-rays. Secondary electron images – having high resolution depth – can be used to characterize the grain shape and surface roughness.

For geochemical interpretation, the BSE images have distinct importance, since these images are sensitive to the average atomic number of the observed features. BSE images may for example be used to investigate oxidation rims, or to detect element zonation within a mineral grain.

Characteristic X-rays, emitted by electron beam bombardment can be analysed either based on their energy (EDS, energy dispersive spectroscopy) or wavelength (WDS, wavelength dispersive spectroscopy). Altogether these methods are named electron microprobe analysis (EPMA). EDS systems provide a fast elemental analysis of the sample surface. The accuracy of EDS is at the level of 1 % to 2 %, while greater accuracy (down to tenths or hundredths of %) can be achieved using WDS.

Computer-controlled modern microprobes are powerful tools for element-mapping: The method can visualize the element distribution and element substitution within individual mineral grains. Combined observations of BSE images with EDS detection provide detailed and useful information about alteration processes of waste materials.

EM techniques are commonly used to supplement the information obtained by optical microscopy and XRPD. Their application is essential to obtain compositional data for solid-solution minerals (e.g. carbonates, feldspars, pyroxenes etc.), to obtain information on fine-scale features such as alteration rims, and to verify the identification of grains too fine-grained to be unambiguously identified by optical microscopy. For species identification of tiny grains or materials of reaction rims, corroborative data on chemical composition and crystal structure is necessary. In this case electron probe micro-analyser (EPMA) is a powerful tool for identification of chemical components, and monocrystal XRD techniques are available for crystal structure determination of small volume samples.

It should be emphasized that SEM and EPMA alone are not sufficient to characterize bulk chemistry or to identify mineral species.

E.1.5 X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (XPS) is a method for investigations of mineral surface chemistry. XPS studies can provide fine-scale detail about the reactions that occur at the surface and near-surface of sulphide-S, and can help explain the mechanisms of alteration. The method has been reviewed in several monographs (see e.g., Briggs and Seah, 1983; Hochella, 1988). XPS measures the kinetic (binding) energy of photoelectrons produced during interaction with an X-ray source. In addition to elemental composition of the surface and near-surface layers, XPS is also sensitive to the chemical environment of these elements, including oxidation and coordination state. Angle-resolved XPS (AR-XPS) provides a non-destructive means of analysing compositional change with depth. The main advantage of this technique is the capability of obtaining quantitative chemical determination of the species distribution with respect to depth by a non-destructive technique (Lüttge and Arvidson, 2008).

An example can be the XPS investigation of pyrrhotite alteration through studies of monoclinic pyrrhotite (Pratt et al. 1994; Mycroft et al. 1995). In the initial stages of alteration in nature, pyrrhotite is replaced by marcasite that is heterogeneous and commonly somewhat fibrous in appearance. With more advanced alteration, the marcasite is replaced by Fe oxy-hydroxide, commonly in the form of rims of goethite (much less commonly lepidocrocite), and subsequently by pseudomorphs of goethite (Jambor and Blowes, 2000).

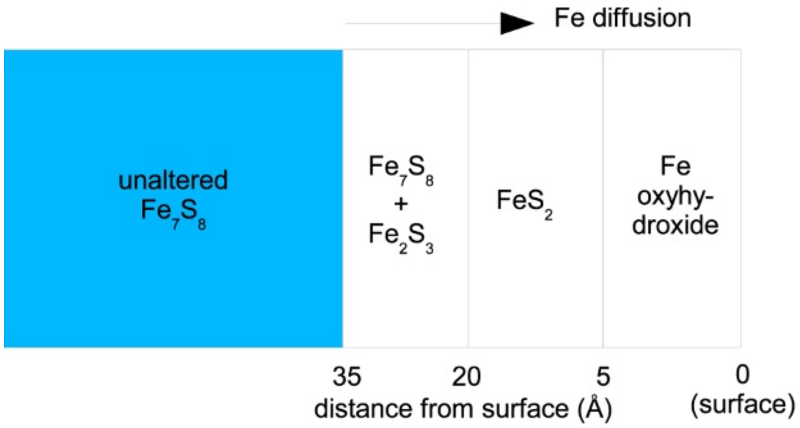

Figure E.2 — Schematic representation of the chemical changes after oxidizing pyrrhotite in air. The zone adjacent to unaltered pyrrhotite is S-rich and Fe-poor as a result of Fe diffusion (but not of S) to the surface layer. Adapted from Mycroft et al. (1995)

Annex F
(informative)

Sulfur speciation in waste from extractive industries

F.1 Total sulphur

International (ISO and CEN) standards are available for the determination of total sulfur. As large numbers of samples are to be analysed automated analysers have been developed that provide accuracy with high throughput at moderate costs.

Total sulfur content is determined either by bomb combustion according to EN 14582 or by high temperature combustion according to ISO 351 or ISO 15178.

F.2 Sulfur species

For the analysis of sulfur species no international standards are available. Speciation of sulfur compounds is a difficult task and is depending on the material to be analysed. A number of different methods have been developed and are used in the mining industry worldwide.

Main purpose of all species analyses is the determination of sulfides: monosulfides (e.g. ZnS) and – most important – disulfides, the most important one is pyrite. This can be done either by direct determination of pyrite or by difference of total and sulfate sulfur (assuming no other sulfur species like elemental sulfur is present).

F.3 Analysis program for sulfur species

The selection of samples was made to cover a broad range of different mining sectors. Samples came from coal and metal mining in Europe. Samples chosen are listed in Table 1:

Sulfur analyses were performed by different laboratories using different methods. Laboratories were chosen from different areas, i.e. having a coal or metal mining background.

Table F.3 — Selected waste samples for the sulfur study

Sample number	**Type**	**Country**	**Information (operation, minerals etc.)**	**Active mine**	**indicative total S %**
1	**8** Tailings	Hungary	Sulfide minerals (Au, Cu)	No	0,25
2	**9** Tailings	Austria	Tungsten	Yes	0,27
3	**10** Tailings	Germany	Hard coal	Yes	0,59
4	**11** Tailings	Sweden	Zn, Pb	Yes	0,69
5	**12** Tailings	Poland	Hard coal	Yes	1,16
6	**13** Tailings	Hungary	Sulfide minerals	No	1,48
7	**14** Waste rock	Finland	Au (arsenopyrite and pyrite)	Yes	1,53
8	**15** Tailings	Finland	Nickel	Yes	1,77
9	**16** Waste rock	Sweden	Zn, Cu, Au, Ag	Yes	3,3

F.4 Analytical procedures for sulfides

F.4.1 Sulfides (pyrite) determination by combustion at different temperatures

F.4.1.1 Direct determination

Sulfur species have different thermal stabilities when being combusted. (Mono- and Di-) Sulfides and elemental sulfur need lower combustion temperatures than sulfates (see also Annex B). This fact is used for determination of species. Sulfides (and elemental sulfur) are measured at a temperature of 750 °C (LTIR1) or 810 °C (LTIR2) in an automated sulfur analyser.

F.4.1.2 Indirect determination

Alternatively the sulfide sulfur is determined by roasting (pyrolysis) the sample in an oven at a temperature of 550 °C (PYR1) or 650 °C (PYR2). After roasting the sulfur content of the pyrolysis residue is determined. Sulfide sulfur is calculated by subtracting sulfur in the roasted sample from sulfur in the untreated sample (= total sulfur).

F.4.2 Sulfides (pyrite) determination (direct) by reaction with HCl

Another procedure (H_2S) is used in the German coal standard DIN 51724-2 which is almost identical to ISO 157. It gives two procedures: one for the determination of monosulfides and a second one for mono- plus disulfides. 1 g of finely ground sample is placed in a round bottom flask and 2 ml ethanol are added. For the determination of disulfides 20 g granulated zinc and 1 g Cr(III)chloride are added, nothing for monosulfides. 50 ml of ethanolic HCl are then added and the released hydrogen sulfide (H_2S) is trapped in a washing bottle with a zinc acetate solution. Sulfur is determined by adding iodine and HCl, shaking and backtitration of unreacted iodine with sodium thiosulfate solution using starch as an indicator.

F.4.3 Sulfides (pyrite) determination (direct) by reaction with $NaCO_3$

Aqueous carbonate solutions are a good solvent for sulfates. 0,25 g of finely ground sample is weighed into a 150 ml beaker. 25 ml of 10 % sodium carbonate solution is added and the solution is brought to gentle boil for 30 min. After filtration through a glass fiber filter the residue is washed two times with 20 ml deionized water each. Filter and residue are dried and then analysed for sulfur. Assuming that all sulfates are removed the result gives sulfide sulfur (plus elemental sulfur, if present) (two laboratories: CARB1, CARB2).

In case of a suspected presence of elemental sulfur an additional step of leaching with carbon disulfide (CS_2) is introduced between filtration and washing with deionized water (CARBCS). Carbon disulfide is a good solvent for elemental sulfur, the result gives sulfide sulfur.

F.4.4 Sulfides (pyrite) determination (indirect) by leaching with HNO_3

Nitric acid (HNO_3) is a good solvent for mono- and disulfides. When samples are treated with HNO_3 it is assumed that only sulfides are leached and all other sulfur species remain in the sample.

The so called “Sobek method” (EPA 600/2-78-054:1978) uses 0,5 g of finely ground sample which is put into an Erlenmeyer flask. 50 ml HNO_3 (HNO_3:water = 1:7 which is 12,5 %) is added and left overnight at room temperature. Afterwards the slurry is filtrated and washed with 100 ml deionized water to remove all chlorides. All leachates are discarded. After that the sample is dried and analyzed for sulfur

No laboratory followed exactly this procedure. Two laboratories (NITR1, NITR2) used HNO_3 at higher temperature (about 80 °C to 85 °C) for approximately 7 hours; this is usually called “modified Sobek”.

Sulfide sulfur is calculated by subtracting sulfur in the HNO_3 leached residue from sulfur in the HCl leached residue.

F.4.5 Sulfides (pyrite) by calculation

The sulfide content can also be calculated (CSUL) assuming that sulfur appears as sulfidic, sulfatic, and elemental only. Sulfide sulfur (plus elemental – if present) sulfur is then calculated as the difference between total and sulfate sulfur if both measured directly.

F.5 Results for sulfide (pyrite) sulfur

F.5.1 Pyrite-rich waste samples

In 4 of the 9 samples most sulfur occurs as pyrite (70 % to 90 % of total sulfur), these are:

Table F.4 — Pyrite rich waste samples

Sample number	Type	Country	Mining type
3	Tailings	Germany	Hard coal
5	Tailings	Poland	Hard coal
6	Tailings	Hungary	Sulfide minerals
9	Waste rock	Sweden	Zn, Cu, Au, Ag

Results are shown in Figure F.1.

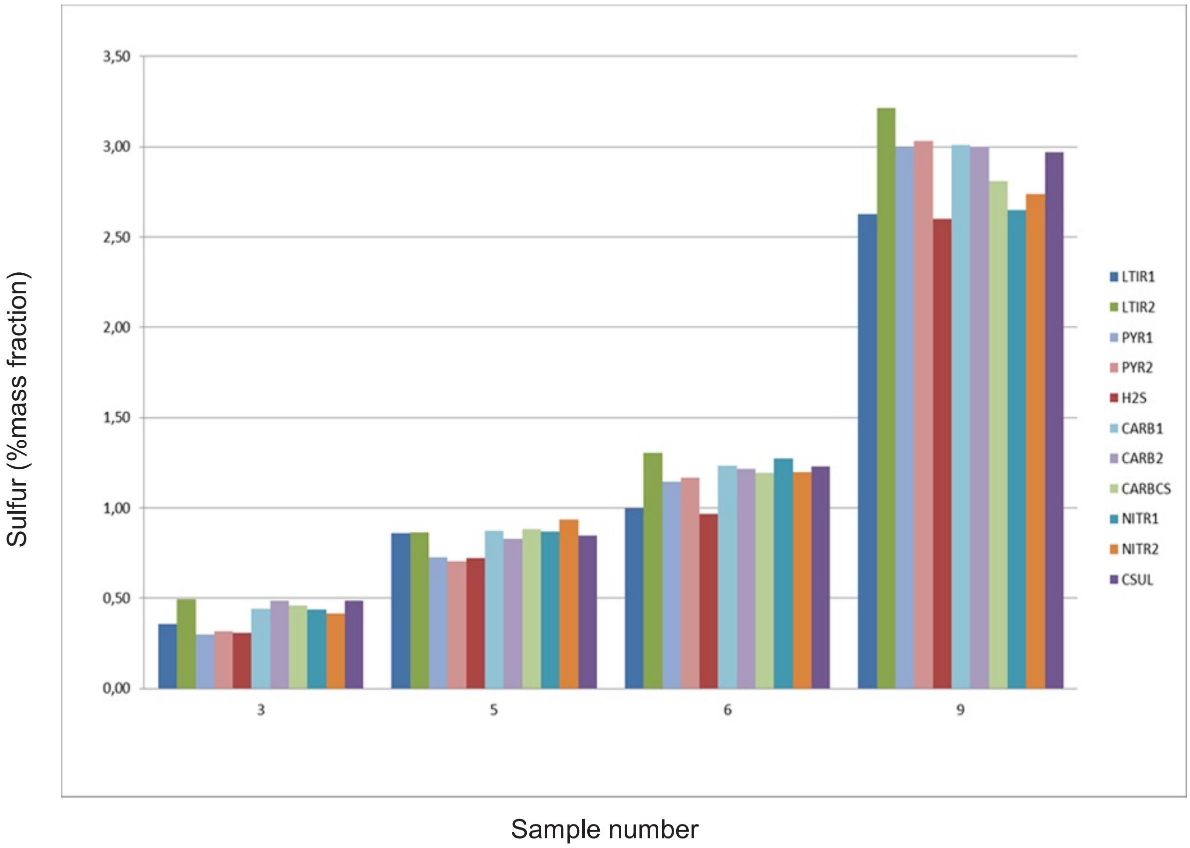

Figure F.1 — Sulfide sulfur (% mass fraction) of 4 pyrite rich waste samples (explanation for abbrevations of methods see text of F.3.1 – F.3.5)

The results are comparable for all different methods applied. Reproducibility is about 10 % relative at concentrations near or above 1 %, below that higher. Generally, these are acceptable accuracy data.

F.5.2 Other waste samples

No averages have been calculated for five out of 9 waste samples as the results obtained by different methods differ too much. They are shown as graphs in Figure F.2.

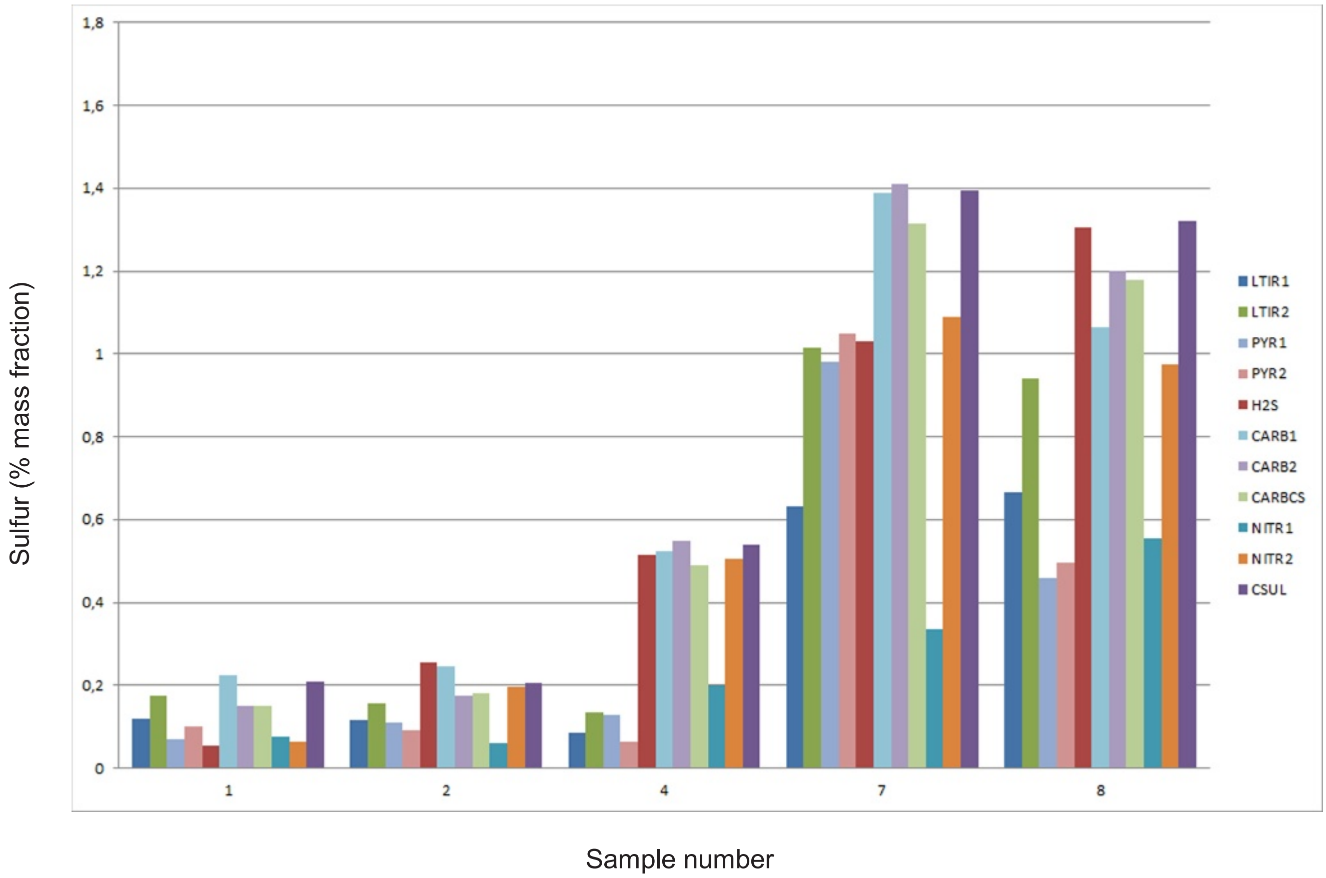

Figure F.2 — Sulfide sulfur (% mass fraction) of 5 waste samples (explanation for abbrevations of methods in the text of F.3.1 – F.3.5)

Sample number 4 (tailings from a Swedish zinc and lead mine) contains sphalerite (ZnS) and pyrrhotite, also some galena (PbS). The results show that methods doing a pyrolysis at lower temperature (LTIR and PYR) give lower results than the others. The method with HCl/$CrCl_3$ (H_2S) and all methods that leach sulfates and give sulfide sulfur as a difference to total sulfur give almost the same result. This is valid also for the modified Sobek procedure (NITR), at least for NITR2.

Sample number 2 (tailings from an Austrian tungsten mine) also contains rather sulfides than disulfides (pyrite). The results show that methods doing a pyrolysis at lower temperature (LTIR and PYR) give lower results than the others. The method with HCl/$CrCl_3$ (H_2S) and all methods that leach sulfates and give sulfide sulfur as a difference to total sulfur give almost the same result. This is valid also for the modified Sobek procedure (NITR), at least for NITR2. The appearance is similar to the one of sample 4 but the differences between pyrolysis and other methods are not that strong

Sample number 1 (weathered tailings from a closed Hungarian gold and copper mine) contained copper arsenic sulfides, other sulfides and pyrite. Due to weathering some sulfides have reacted to sulfates. The results by different methods are not easy to interpret. Highest values are found when sulfates are leached with carbonate and sulfide is calculated by difference to total sulfur. The direct method with HCl/$CrCl_3$ (H_2S) gives the lowest value.

Sample number 7 (waste rock from a Finnish gold mine) contains arsenopyrite and arsenic-rich pyrite. The results show that methods doing a pyrolysis at lower temperature (LTIR and PYR) give comparable results to the method with HCl/$CrCl_3$ (H_2S). Carbonate leaching (CARB and CARBCS) and direct determination of sulfates (CSUL) give similar but higher results. Modified Sobek procedure (NITR) gives a result in between, at least for NITR2.

Sample number 8 (tailings from a Finnish nickel mine) contains pyrite, chalcopyrite and some pyrrhotite. The results show that methods doing a pyrolysis at lower temperature (LTIR and PYR) give lower results than the others, but direct method (LTIR) is higher than indirect (PYR). The method with HCl/$CrCl_3$ (H_2S), carbonate leaching (CARB and CARBCS) and direct determination of sulfates (CSUL) give similar but higher results. The results from the modified Sobek procedure (NITR) are somewhat in between.

F.6 Summary and recommendations

When the main constituent of sulfidic minerals is pyrite, all different methods give similar results and may be used. When a major part of sulfidic minerals is non-pyritic the results differ much more. Higher thermal stabilities are found for minerals that contain other metals as iron, so for Pentlandite ($(Fe,Ni)_9S$) and even more for mono-sulfides like Galena (PbS) and Spalerite (ZnS). Therefore all methods that use low temperature combustion (LTIR, PYR) show systematic lower values. If these minerals are present it could be advisable to use carbonate leaching (CARB, CARBCS or CSUL) or a direct method (H_2S). Also the modified Sobek procedure (NITR) can be applied.

Annex G
(informative)

A/NRD processes and mineralogy

G.1 Introduction

The formation of acid/neutralising rock drainage (A/NRD), a key issue for the management of many wastes from the extractive industry, is strongly related to the mineral assemblage and the mineral chemistry. The main text of this overall guidance document presents mineralogy and A/NRD processes briefly. This annex expands on those issues. However, for detailed information the readers are suggested to read text books on mineralogy and a variety of publications on A/NRD (e g - Price, 2009; GARDGuide, 2009; MAC Short Courses vol. 27 and 31).

There are three processes in the area of A/NRD that are elaborated on in this annex:

- natural supergene A/NRD processes (G.2);
- mineral oxidation / dissolution of the main mineral groups that play a key role in A/NRD (G.3);
- some basic concepts of environmental mineralogy such as the mineral reaction rates concepts (G.4.2) as well as issues related to the quantification of the mineral surface area and characterization of mineral texture (G.4.3 and G.4.4).

G.2 Natural supergene A/NRD processes

The A/NRD processes are naturally occurring processes that have been known to economic geologists for a long time and labelled, in some settings, as *supergene* processes (Figure G.1). They are mineralogically and geochemically the same processes as taking place in tailings and waste rock. Evidence of ARD can commonly be seen in sulfide ore bodies where oxidation of the primary ore has taken place and the sulfide minerals have been replaced by oxide minerals. This natural *in situ* sulfide oxidation and acid generation can sometimes be mistaken for mining related ARD (Walder et al, 2000).

The supergene processes - natural weathering processes - occur due to sulfide mineral oxidation, metal transport and formation of secondary minerals. Below the supergene zone is the hypogene zone containing primary minerals. In the supergene zone above the water table the metal and anion components are mobilized, primarily downward, where these will precipitate in the zone of accumulation/enrichment. At the surface, traces of A/NRD processes may be seen as red/brown iron oxide/hydroxide coloured minerals either as staining or massive (Gossan in Figure G.1) or low pH ochre coloured surface drainage (Glader et al, 1996). This is the leftover iron and upward migration of iron from the iron sulfide oxidation.

ARD processes are essential in the formation of several types of ore deposits, especially in the supergene enrichment of porphyry copper deposits, where oxidation of chalcopyrite (and pyrite) forms an acidic environment allowing for copper transport downward or outward (Robb, 2005; Chavez, 2000). The zonation that develops is due to the mobility difference of the metals, primarily in response to changing temperature and pH. Understanding these processes aids in predicting what will likely happen in tailings and waste rocks produced from these rocks.

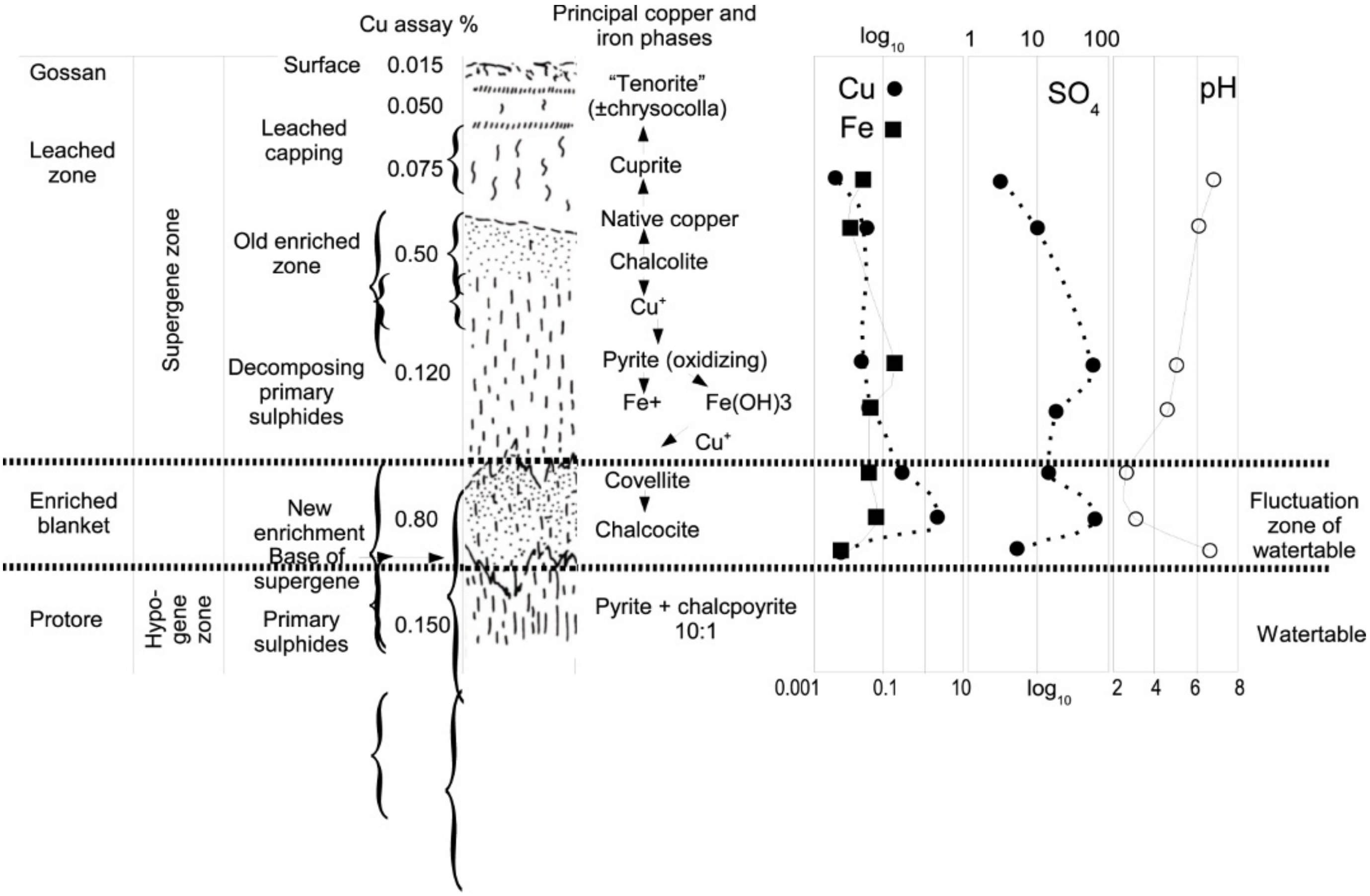

Figure G.1 — Supergene enrichment process in plesyumi porphyry copper (Nouvelle Guinée and Titley, 1978)

G.3 Mineral oxidation / dissolution

G.3.1 General

Pyrite oxidation processes have been studied intensively over the last 15 years to 20 years, both as pure mineral reactions and in rocks from mineral deposits due to the environmental significance of A/NRD. The results of these studies and the knowledge of ore genesis processes give background for evaluating other sulfide minerals that have not gotten the same attention. In the following paragraphs the main aspects of mineral oxidation and dissolution for sulfide minerals, iron sulfate minerals, and neutralizing minerals are discussed.

G.3.2 Sulfide

There are many factors that affect the oxidation rate of sulfides, and hence, control acid generation and metal release. They may be grouped as:

- mineral-related: e.g. exposed surface area, trace element concentrations;
- physico-chemical: e.g. pH, temperature and chemical activation energy, oxygen concentration (in water and gas phase), ferric iron concentration;
- other micro-environmental factors;
- bacteriological activity; and

- removal of reaction products.

As described before, sulfide oxidation and A/NRD are also natural processes. However, when material is mined, crushed, and/or milled, more surface area becomes available for reactions and thereby the bulk oxidation will increase.

Oxygen availability is the most essential limiting factor for sulfide mineral oxidation. Ferric iron can also act as a sulfide oxidizing agent and, if available, is more effective than oxygen. In a reducing environment where oxygen (and thereby ferric iron) is highly limited, oxidation will also be limited or even reverted to a reduction of sulfate to sulfide. When oxygen is available in an acidic environment, the reaction rate is more controlled by bacteriological activity (Schippers et al, 2010; Hallberg, 2010).

The bacteriological activity is affected by pH and nutrient availability. The oxidation rate for sulfides can be one or two orders of magnitude higher when the bacteria have good supply of nutrients, under the same physio-chemical conditions.

Pyrite is the most common sulfide mineral. However, in addition to pyrite, other common sulfide minerals in sulfidic ores are chalcopyrite, arsenopyrite, bornite, galena, sphalerite, marcasite, thetraedrite, pyrrhotite and pentlandite.
Besides pyrite, several of the other, but not all, sulfide minerals have the potential to produce sulfuric acid due to oxidation. The amount of acid produced depends on the sulfide metal/sulfur ratio, type of oxidant, and precipitating minerals (Plumlee, 2003).Pyrite oxidation by atmospheric oxygen with presence of water – as typical in tailings and waste rock piles – produces from one mole of pyrite one mole of Fe^{2+}, two moles of SO_4^{2-} and two moles of H^+ (Nordstrom, 1982):

$$FeS_2 + 7/2\ O_2 + H_2O \rightarrow Fe^{2+} + 2SO_4^{2-} + 2H^+$$
(G.1)

Depending on pH and Eh conditions of the environment, the dissolved Fe^{2+} may (a) remain in solution, or (b) precipitate as ferrous sulfate and ferrous hydroxide minerals, or (c) further oxidize and precipitate as ferric oxy-hydroxides (Petruk, 2000). Thus (b) and (c) lead to the formation of secondary minerals precipitating as coatings on pyrite and other minerals, or forming individual grains.

The potential subsequent reaction where the ferrous iron (Fe^{2+}) is oxidized to ferric iron (Fe^{3+}), that precipitates as ferrihydrite ($Fe(OH)_3$), producing an additional two moles of H^+ may be described by the following two reactions:

$$Fe^{2+} + 1/4\ O_2 + H^+ \leftrightarrow Fe^{3+} + 1/2H_2O$$
(G.2)

$$Fe^{3+} + 3H_2O \leftrightarrow Fe(OH)_3 + 3H^+$$
(G.3)

Thus, the resulting overall reaction yields:

$$FeS_2 + 15/4\ O_2 + 7/2H_2O \rightarrow Fe(OH)_3 + 2SO_4^{2-} + 4H^+$$
(G.4)

On the other hand, there are many sulfide minerals that do not produce sulfuric acid when oxidizing. In fact, these minerals may consume acid during their dissolution/oxidation and give a net of zero hydrogen ions. Sphalerite and galena are two such minerals that may oxidize as shown in the following reactions:

$$ZnS + 2O_2 = Zn^{2+} + SO_4^{2-} \quad (G.5)$$

$$PbS + 2O_2 = Pb^{2+} + SO_4^{2-} \quad (G.6)$$

However, the oxidation of these sulfide minerals may be enhanced by ferric iron as an oxidizing agent instead of oxygen, as in the following reactions:

$$PbS + 8Fe^{3+} + 4\ H_2O = Pb^{2+} + 8H^{+} + SO_4^{2-} + 8Fe^{2+} \tag{G.7}$$

$$ZnS + 8Fe^{3+} + 4H_2O = Zn^{2+} + 8H^{+} + SO_4^{2-} + 8Fe^{2+} \tag{G.8}$$

In this setting (Formula G.7 and G.8), the oxidation of the sulphide minerals produce acidity due to iron reduction. In sphalerite, iron may substitute for the zinc, up to 50 mole %. If iron is substituted for zinc, sphalerite will be an acid generator in a similar way as pyrrhotite.

There are a number of investigations of relative reactivity of sulfide minerals reported in literature. Plumlee (2003, and references therein) suggested the following relative oxidation resistance, starting with the less resistant phases: Pyrrothite, troilite ($Fe_{1-x}S$); Galena (PbS); Sphalerite (ZnS); Pyrite (FeS_2); Arsenopyrite (FeAsS); Tennantite ($Cu_{12}As_4S_{13}$); Energite (Cu_3AsS_4); Marcasite (FeS_2); Chalcopyrite ($CuFeS_2$); Molybdenite (MoS_2).

Using the overall formulas for sulphide minerals oxidation (e.g. Formula. G.4), moles of hydrogen ions produced per mole of mineral oxidized or per weight can be calculated (Table G.1).

Table G.1 — Relative reaction rates are from *Rimsted et al. (1994) given at pH 2 with ferric ion as the oxidizer and **Nicholson and Sharer (1994) given at pH 2 with oxygen as the oxidizer. Pyrite reaction rate is defined as 1

Mineral	Mole H^+/mineral	Relative wt -%	Relative reaction rate
Pyrite, marcasite	4	0,03	1 *
Arsenopyrite	3	0,018	1 *
Chalcopyrite	2	0,011	0,1 *
Pyrrhotite, Troilite	2	0,022	100 **
Enargite	1	0,002	
Galena	0	0	1 *
Sphalerite (pure form)	0	0	0,1 *

The contribution of a sulfide mineral to the ARD processes depends not only on relative acid production but also on relative reaction rate. The reaction rates are different between different sulfide minerals (Table G.1.) and depend on the physico-chemical and biological conditions. Beside crystal chemistry, reaction rates are mainly a function of the surface area per mass of the particular mineral (Wiersma and Rimstidt, 1984). Therefore reaction rates depend on crystal habit and form of occurrence (fine-grained or colloform diagenetic versus coarse-grained hydrothermal) as well as on the deformation state. The latter can be a result of grinding, leading to increased oxidation rate of pyrite in tailings (Sasaki 1994).

Fine grain size or an uneven surface, framboidal texture, will enhance the oxidation rate relative to coarse grain size or euhedral crystal shape. A low content of trace elements in crystal lattice will promote the resistance to oxidation compared to higher trace element content. As the solubility of sulfide minerals is increased in acidic environments, trace element content can be mobilized resulting in elevated concentrations in the drainage. Galena, for example, is reported to contain silver, arsenic, and antimony. Sphalerite is reported to contain cadmium and mercury (Mason and Berry, 1968).

In addition, the order of resistance of sulfide minerals can vary depending on field conditions (oxidative vs. non-oxidative environment, grain size, texture, opportunity of formation of secondary mineral coatings), Jambor and Blowes (1998) urges that "laboratory data for sulfide-mineral reactivities, including reactivities determined from the interaction of reagents with polished surfaces of sulfides, should not be freely extrapolated to field scenarios. The field observations should have precedence in applications to long-term ARD prediction".

Since so many different factors control the oxidation rates of the different sulfide minerals, a list of reaction rates has not been established. The reader is referred to the references within this document for establishing an appropriate rate for each site or case. Nicholson (1994) has summarized several studies on pyrite oxidation rates.

G.3.3 Secondary minerals

Secondary minerals precipitate throughout the vadose zone in tailings storage facilities and waste rock dumps, forming crystals in interstitial pores, or coatings on mineral grains (see section G.1). The secondary minerals have various degrees of solubility. Therefore, for some minerals the precipitation and re-dissolution can vary with seasonal changes, while other secondary minerals are practically insoluble and do not re-dissolve (Petruk, 2000). In arid or semi-arid environments, precipitation of secondary sulfates in dry periods and dissolution in wet periods cause dramatic seasonal variations in metal concentrations (Alpers et al, 1994).

The most abundant and widespread secondary minerals in most ARD scenarios are goethite, gypsum, and jarosite-group minerals (typically solid solutions of jarosite, natrojarosite and hydronium jarosite). The presence of gypsum in carbonate-bearing wastes indicates the dissolution of carbonate minerals that serve as a source of Ca^{2+}. Jarosite-type minerals indicate the weathering of silicates (primarily feldspars) to provide a source of K^+ and Na^+. (Jambor and Blowes, 1998).

Iron sulfate minerals also generate acid. Moreover, they are not dependent on oxygen availability to generate an acidic environment. They are commonly highly soluble and are normally found as reaction products of iron sulfide oxidation in dry environment or in low pH environments with high iron and sulfate concentrations (evaporating environments, e.g. Iron Mountain (Nordstrom et al. 1982).

As an example, step-wise dissolution of melanterite is shown by the following three formulas. The net result of these reactions is a net production of two moles of acid for each mole of melanterite dissolved (Lapakko, 2002).

$$FeSO_4 \cdot 7H_2O(s) = Fe^{2+}(aq) + SO_4^{2-}(aq) + 7H_2O(aq) \tag{G.9}$$

$$Fe^{2+}(aq) + (1/4)O_2(g) + H^+(aq) = Fe^{3+}(aq) + (½)H_2O(aq) \tag{G.10}$$

$$Fe^{3+}(aq) + 3H_2O(aq) = Fe(OH)_3(s) + 3H^+(aq) \tag{G.11}$$

Conversely, rims of insoluble secondary minerals formed on primary sulfides, e.g. anglesite rims on galena, increase the resistance to dissolution, reducing the direct contact with the oxidizing agents. On the other hand, secondary zinc sulfates are highly soluble. Therefore galena can be more resistant in field conditions than sphalerite, even though opposite reactivity is indicated by laboratory experiments (Jambor and Blowes, 1998).

G.3.4 Carbonate minerals

Many ore deposits are hosted by or associated with carbonates. The majority of these carbonate bodies are composed of trigonal carbonates (calcite and dolomite structure) such as calcite [$CaCO_3$], siderite [$FeCO_3$], ankerite [$Ca(Fe,Mg)(CO_3)_2$] and dolomite [$CaMg(CO_3)_2$]. Substitution among Ca^{2+}, Mg^{2+}, Fe^{2+}, Mn^{2+} cations is common for these minerals. The composition of calcite is typically close to pure CaCO3, while Fe^{2+} in siderite is often substituted by Mg^{2+} and Mn^{2+}, generally with Mg <10 mol% and Mg >> Mn (Jambor and Blowes, 1998). Routine optical or X-ray methods are not eligible to distinguish between dolomite and ankerite, therefore these are grouped together in practice.

Carbonate minerals, especially calcite are very efficient neutralizers and have dissolution rates 2-3 orders of magnitude faster than the pyrite oxidation rate. Under neutral conditions, calcite is relatively stable. Dolomite ($CaMg(CO_3)_2$) is also an acid neutralizer, however it’s reaction rate is 1-2 orders of magnitude slower than calcite.

Ca-Mg-Fe-Mn substitutions in trigonal carbonates as well as presence of trace elements can influence considerably their dissolution rates (Terjesen et al. 1961). Laboratory data and field observations indicate that calcite is by far the most soluble of the common gangue minerals, and resistance to dissolution increases in the order: calcite - dolomite - ankerite – siderite (Jambor and Blowes, 1998).

The high rate of calcite dissolution can maintain near-equilibrium conditions and keep pore water pH in the range of 6,5 to 7,5 (Petruk, 2000). Laboratory column experiments and field observations show that the calcite dissolution rate varies strongly with grain size. Experiments conducted for 7,5 years, reported by Paktunc and Dave (1999) showed that pyritic uranium tailings with high acid producing potential, amended with sufficient fine-grained limestone did not produce ARD, whilst the same tailings material amended with the same weight proportion of coarse-grained limestone produced some acidic drainage. Calcite dissolution experiments also show that internal crystal faults – dislocations, twinning planes and vacancies – contribute to increased dissolution rate (Mádai, 2005).

As mentioned earlier, siderite in acid generating wastes needs special attention. At low pH it will act as a neutralizer: However, above pH 3,5 to 4, where iron hydroxide precipitates, it will become an acid generating mineral (Formulas G.12, G.13).

$$Fe^{2+}(aq) + (1/4)O_2(g) + H^+(aq) = Fe^{3+}(aq) + (1/2)H_2O(aq) \qquad (G.12)$$

$$Fe^{3+}(aq) + 3H_2O(aq) = Fe(OH)_3(s) + 3H^+(aq) \qquad (G.13)$$

When the Fe^{2+} may react with the released HCO^{3+}, secondary siderite can precipitate as that was detected by several authors (Boin and Cherry 1986; Blowes et al. 1991; Blowes and Ptacek 1994; Ptacek and Blowes 1994).

G.3.5 Silicate minerals

Many silicate minerals also have the capability to neutralize acid. However, the reaction rate is much slower than for calcite; and the capacity is much less than that of the carbonate minerals. Silicate minerals can neutralize in areas where an acid plume is moving relatively slowly, e.g. groundwater flow, and water flow through tailings.

Because of the predominating abundance of silicates in the common crystalline rocks, weathering reaction rates of these minerals have been studied in numerous laboratory experiments. The alteration kinetics of extractive wastes in most aspects is similar to natural weathering, however it is frequently controlled by specific conditions: acidic, or highly acidic environment, increased specific surface because the rocks and minerals have been crushed, providing more available surface for reactions.

In acidic environments, the dissolution rate for the common rock-forming silicate group minerals and quartz decreases in the order: forsterite (olivines) - pyroxenes - biotite - muscovite – quartz. Although this is too broad for ARD prediction, it shows that most of the non-carbonate minerals, which are typically associated with metalliferous deposits, other than those in skarns (mineral deposits formed from due to alteration of host rocks by intrusive bodies) and ultramafic rocks (magmatic rocks abundant in olivines and pyroxenes), react extremely slowly (Jambor et al, 2000).

Feldspar dissolution in weathering has extreme importance because minerals of this group (plagioclases and K-feldspars) constitute 70 % to 80 % of the upper continental crust (Nesbitt and Markovics 1997). This is the most common rock forming mineral group also in most waste rocks. Despite their slow dissolution rates, some feldspars - highly calcic plagioclases under very low pH conditions, when all carbonates and simple hydroxides are depleted - may be an important source of neutralization by long-term weathering. Feldspar dissolution can be indicated by an increase in the Si and Al contents in the pore water (Jambor and Blowes, 1998).

For petrographic characterization the main rock forming silicate minerals are frequently mentioned with mineral group names e.g. plagioclase, olivine, pyroxene etc. However, there are important differences in chemical composition of the different end-members or intermediers of the group. These differences influence also the weathering stability, thus the dissolution rate of these minerals.

The relative dissolution rates of common silicate minerals are summarized in table G.2 based on laboratory experiments at pH 5 and far from saturation (Jambor et al, 2000). Rates are expressed relative to calcite based on data from Drever and Clow (1995) and from Nagy (1995), using in the latter the rates relative to muscovite.

Table G.2 — Relative dissolution rates of common silicate minerals at pH 5 and far from saturation, based on laboratory experiments (Jambor et al, 2000). Calcite and dolomite are mentioned as reference minerals

Mineral	**(Rate/ Rate for calcite)x10^5**	**Mineral**	**(Rate/ Rate for calcite)x10^5**
Calcite Dolomite	100 000 6 000	plagioclase An76	0,25
Forsterite	4	plagioclase An46	0,12
Diopside	1,4	plagioclase An13	0,02
Enstatite	0,93	plagioclase An0	0,02
Talc Chrysotile	0,06 0,06	sanidine	0,03
Biotite	0,01 – 0,03	microcline	0,01
Chlorite	0,02		
Kaolinite	0,006 – 0,02		
Muscovite	0,006		
Montmorillonite	0,002		
Quartz	0,0005		

The importance of the specific chemical composition and crystal structure is reflected in significant difference in dissolution rate of basic (An76) and acidic (An13, An0) plagioclases. Another example is chlorite, which is a common mineral associated with sulfide mineral deposits, mentioned with a low dissolution rate in Table G.2. However, as it was found by Nagy (1995), the rate will increase by one to three orders of magnitude when the iron content of the mineral is increased.

Calculated dissolution rates for main rock forming minerals were contributed by Paktunc (1999) - At pH 5, in decreasing order the dissolution rates were determined as: brucite - calcite - olivine (Fo0) - dolomite - plagioclase (An10) - wollastonite - nepheline - olivine (Fo91) - jadeite - olivine (Fo100) - diopside - enstatite - hornblende - augite - plagioclase (An80) - serpentine (antigorite) - plagioclase (An40) - serpentine (chrysotile) -gibbsite - biotite - microcline - plagioclase (An0) - plagioclase (An20) -sanidine - talc - K-feldspar - phlogopite - muscovite - chlorite - epidote - kaolinite - quartz -montmormillonite – anthophyllite.

According to his study, the order of decreasing dissolution rates at pH 2 is: calcite - dolomite -plagioclase (An100) - olivine (Fo0) - wollastonite - olivine (Fo91) - olivine (Fo100) - magnetite - diopside - hornblende -biotite - plagioclase (An80) - augite - enstatite - plagioclase (An40) - plagioclase (An20) - plagioclase (An60) - K-feldspar - muscovite - plagioclase (An20) - kaolinite –anthophyllite.

Most of the time, the silicate minerals are not effective neutralizers in the acid base accounting analysis. However, if the pH stays at the low end for a while silicate minerals may contribute to the neutralization. For example actinolite has been shown to be a very efficient neutralizer even in moderate acidic solutions (pH 4 - 5). Olivine is known to be a relatively rapid neutralizer and biotite is somewhat slower, however it can be a considerable acid consumer especially in low pH environment.

G.4 Mineralogy issues

G.4.1 General

The mineral reactions are controlled by mineral surfaces, texture and and physio-chemical conditions. Physio-chemical issues are included in the main text under leaching and kinetic tests. The following sections briefly explain the surface conditions of minerals and their implications for mineral reactions.

G.4.2 Mineral reaction rate concepts

Most mineral reaction rates are determined based on laboratory studies of pure minerals with high water to rock ratio. Such studies are often performed in batch or flow through reactors. These experiments indicate the reaction rate parameters on the molecular scale at the reaction surface. The high water to rock ratio are used to avoid build-up of secondary reaction products and to maintain the solution chemistry near constant. This experimental setup makes it possible to control the effect of mineral properties (crystal structure, chemical bonds) and environmental conditions (temperature, pH etc.) on reaction rates. However, in a natural system the rate of dissolution/oxidation should be investigated on a larger scale (micro – macro environment), because the reaction rates are controlled by three processes:

- transport of constituents to the surface of the mineral (where the reaction is taking place);
- chemical reaction, exchange, release of species at the surface;
- transport of secondary products away from the reaction surface.

The rate of reaction at the mineral surface depends also on grain texture, which defines the amount of available reaction sites, on trace element content (Plumlee and Logsdon 1999) as well as on availability of nutrients. The characterization of grain surface and texture is briefly discussed in the next sections (G.4.3, G.4.4), whilst mineral reaction rates of different mineral groups that are important in ARD processes are introduced in the above sections (G.3).

The oxidation of iron sulfide minerals generate acidic conditions, where iron and sulfur oxidizing bacteria can effectively oxidize sulfide minerals. The Fe-sulfide oxidation is exothermic, which will result in increased temperature. Increased temperature will increase the reaction rate and further improve the conditions for the iron and sulfur oxidizing bacteria. The low pH can result in increased availability of dissolved ferric iron and thereby, further enhance the sulfide oxidation rate.

On the micro-scale, the grain structure (texture) of the rock will affect the transport of reaction products. Tight grains (low porosity and low permeability) will not easily allow the fast transport of constituents needed for a reaction or the flush out of weathering products. In a low flow system, a build-up of reaction products may result in precipitation of secondary products, which are cementing the material, and thereby, increasing the physical stability of the weathering material, lowering the reaction rate or stopping it. In addition, when clay minerals are formed, these clay minerals may be held together by the structure of the rocks, blocking the transport of reaction products. This will result in a reduced reaction rate.

Water quantities will strongly affect the weathering of some minerals. For sulfide minerals, for example, the rate may be lower during heavy flushing because there is not enough hydrogen ion build-up for the bacteria to germinate - Heavy flushing through silicate minerals on the other hand, will result in faster weathering, e.g. tropical climates have commonly more extensive weathering than dry climates.

On the macro- and larger scale, the reaction rates can change over time.

Acid forming waste rock or tailings may not show their acid forming capacity directly. Usually there is a lag time before the actual acid formation starts. This may be due to lack of nutrients, the presence of a readily available (depleting) neutralizing mineral or due to inhibition of bacterial growth due to the presence of toxic concentrations of contaminants. Until a life sustaining level of nutrients is reached, the neutralizing mineral phase is depleted or the inhibitors have been washed out, acid formation cannot actually start.

In environments where there is a high rainfall, the element transport out of the waste rocks may be higher with less or no precipitation of silica. If in addition, there is a considerable content of plagioclase, the cohesiveness of the rocks will likely be reduced during weathering and, thereby, the physical stability of the waste rock piles may be reduced. In order to predict these reactions, it is necessary to perform geochemical modelling. However, the link between the geochemical modelling result and the physical stability of rock fragment and waste-rock piles is difficult to evaluate.

G.4.3 Quantification of the mineral surface

While many reactions are a function of the available surface area, the quantification of the surface represents a complex problem, which greatly depends on the scale of investigation. New, sophisticated analytical tools (e.g. atomic force microscopy - AFM, vertical scanning interferometer – VSI) provide the opportunity to observe these processes at the nano-scale.

To measure dissolution rates, and thus release rates – especially when the mineral reaction is surface-controlled, i.e. the reaction rate depends on the available surface – a quantitative determination of the surface is necessary. Theoretically, there are three different possibilities to characterize the surface area: (1) geometric, (2) total or Brunaur, Emett and Teller method (BET) surface area, and (3) the so-called reactive surface area. Practically, however, only BET surface area is a robust measurement (Lüttge and Arvidson, 2008).

The geometric surface area is an estimation that can be calculated from the crystal/grain dimensions, therefore it is always a simplified model of the grain. It can be obtained from microscopic or SEM images, or estimated from grain size distribution and grain shape values. Geometric surface area is often used in dissolution rate estimations in field systems, nevertheless it is always an underestimation of the real surface area, since it does not consider the surface roughness of the grain (Brantley, 2003).

Surface roughness and porosity is accounted for in the total surface area, or as often used in literature the “BET” surface area. A common way of measuring total surface area is by gas adsorption (BET surface area). The surface roughness is defined as the ratio of the BET to the geometric surface area (Helgerson et al, 1984).

At the crystal surface formation of etch pits is connected to the highly reactive parts of the surface, while the surface between the etch pits is significantly less reactive. Therefore, there is usually agreement that reactive surface area is often significantly smaller than the total surface area (Lüttge and Arvidson, 2008).

The reactive surface area is estimated from the dissolution rate of solid powder (Helgerson et al, 1984), where the highly reactive parts – kink sites, corners, steps – have been increased by pulverization relative to a normal crystal surface.

A practical consequence of this concept for modelling of kinetics of ARD processes can be that models based on laboratory tests and calculations would overestimate the acid producing effect of the observed waste material. BET surface area can be an inappropriate parameter for extrapolation of surface controlled reaction kinetics from laboratory tests to field conditions (Brantley, 2003).

G.4.4 Mineral texture

Mineralogical characterization cannot be limited to identification of phases and their chemical composition but mineral grain properties and interrelations should also be described. Most of these textural properties are difficult to quantify, usually only descriptive terms can be used. However, as it was mentioned above, grain size, forms of mineral aggregations, liberation degree, secondary mineral coatings and other textural properties can strongly modify reaction kinetics of extractive wastes under natural weathering conditions.

Each rock mass can be characterized using the concept of texture components, where the individual constituents are separated by their textural properties such as the grain size, shape, and arrangement. For extractive wastes texture components are divided into primary textures (texture formed before or during emplacement) and secondary textures (textures formed after emplacement). The texture components may be as follows:

- Primary:
    - liberated mineral grains (ore and gangue);

- detritus;
    - clay particles and/or other sheet silicates;
    - tiny grains of other minerals.
- small-size rock fragments of the ore body;
- large-size rock fragments / boulders of the ore body;
- small-size rock fragments of the site rocks;
- large-size rock fragments / boulders of the site rocks.

- secondary:
    - cementing phases;
    - oxidation products / gossan.

Primary and secondary textures are illustrated in Figure G.2 - Pore space, open voids between the grains, may change as a response to the mineral dissolution alteration and precipitation processes. This again will affect the conductivity of water and air within the waste material.

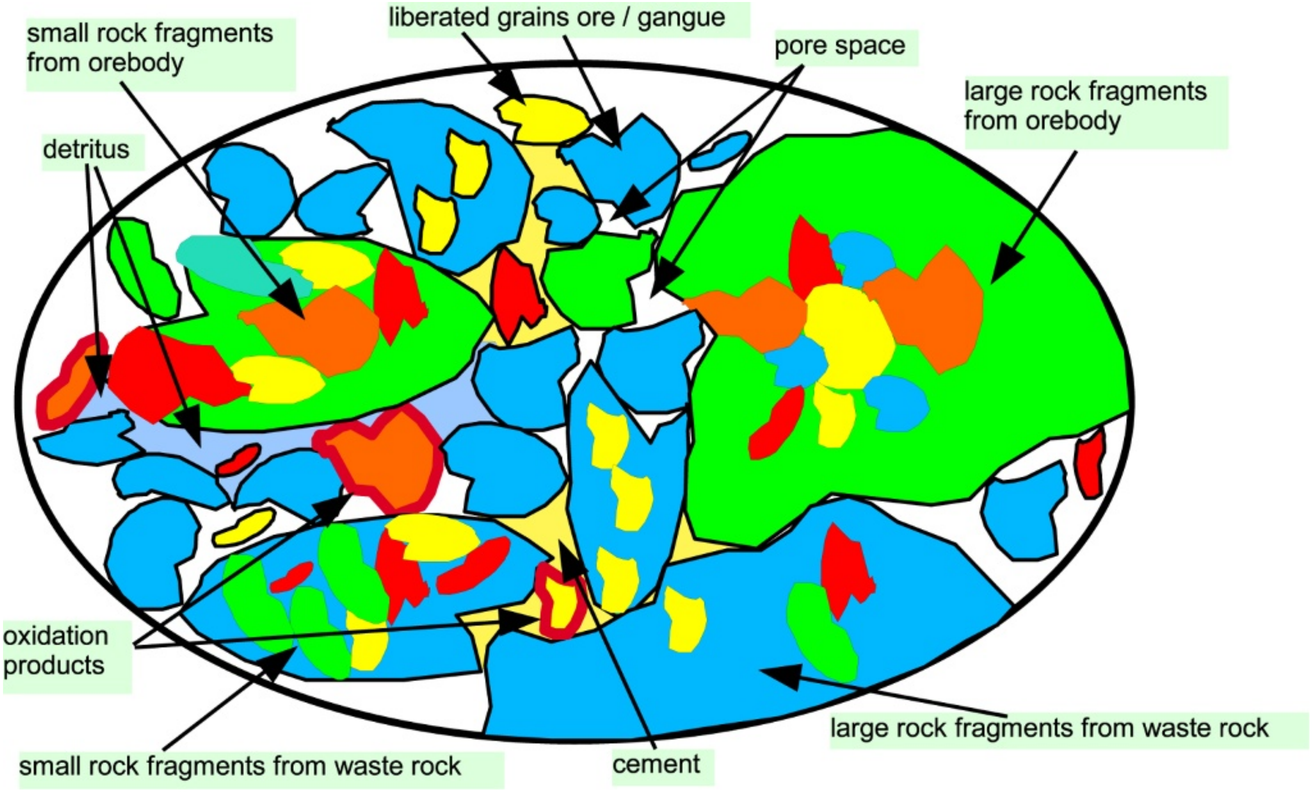

Figure G.2 — Schematic representation of different texture components

Annex H
(informative)

Hydrological and geochemical modelling

H.1 Importance of modelling long-term release

Leaching tests are limited in their capability to assess release of substances in the long term, as conditions in the field cannot be mimicked in the lab. Even field test are too short (years) to assess release at long term. For assessment of long term release from wastes from the extractive industries geochemical reaction transport modelling based on observations in laboratory test, mineralogical characterizations, and actual field observations is the only way forward to allow a prediction of what the water quality seeping from tailings and waste rocks will be under a variety of exposure conditions at the long term.

The mechanistic descriptions resulting from this type of modelling reach further than correlation based descriptions and fixed partitioning approaches (Kd). Obviously, the demand for proper thermodynamic data will increase, when this type of modelling is applied. On the other hand the mechanistic modelling will also help to identify the main gaps in the knowledge (e.g. proper thermodynamic data for oxyanions like Mo, Se, As, Sb).

The mechanistic modelling approach needs to be build up in steps starting from an understanding of the leaching behaviour in controlled laboratory test to release in kinetic testing, lysimeter studies and ultimately to the field. It is crucial that along the way verification of modelling output with experimental data provides the level of understanding of the relevant processes needed to move to the next level of complexity. The use of analogy with existing natural systems or old waste dump in the same area, or submitted to a similar weathering conditions, could be helpful to put constraints on the computer model.

H.2 Uses of modelling

Hydrological and geochemical modelling is used for a variety of questions. Sometimes hydrological aspects and geochemistry are evaluated separately; they can be evaluated successively or be coupled within the same code.

- saturation indices to determine likelihood of precipitation/dissolution;
- predominance diagrams identifying important phases;
- prediction of drainage water quality under a variety of conditions;
- acidification potential of a waste;
- uncertainties in prediction;
- chemical reaction / transport:
    - in the waste body;
    - near field;
    - far field.

Modelling only a very limited selection of substances simultaneously will not very likely lead to realistic predictions. Modelling release without taking major and minor element behaviour into account is unlikely to lead to realistic predictions of key controlling parameters such as pH, redox state, salt level, etc, which determine the ultimate behaviour of trace constituents.

H.3 Commonly used codes

Maest et al. (2005) give an extensive overview for hydrological and geochemical codes used most frequently in the mine waste field (Tables H.1 and H.2). The tables as provided by Maest et al (2005) have been modified to include only near field hydrological models, vadose zone - and groundwater transport codes. The geochemical models list has been expanded with ORCHESTRA (2003).

Table H.1 — Description of selected hydrogeologic codes used for predicting water quality at hardrock mine sites (after Maest et al, 2005; modified to cover near surface, vadose zone and groundwater transport)

Category of code	Subcategory	Available codes	Inputs required	Modelled processes/output
Near surface process hydrologic codes	Water balance (infiltration, runoff, evapotranspiration)	HELP (Schroeder et al, 1 994a, b); SOILCOVER (MEND 1994) CASC2D; CUHP; CUHP/SWMM; DR3M; HEC-HMS (US ACOE 2000); PRMS; PSRM; SWMM; TR20	Precipitation, temperature, wind speed, incident solar radiation, vegetative cover (for evapotranspiration) (climate data can be estimated using CLIGEN or WGEN; USDA ARS); hydraulicconductivity/permeability of soil/geologic material; soil moisture storage and transmission requirements.	Partitioning of precipitation into runoff, evapotranspiration, infiltration; estimation of runoff, infiltration, evaporation rates through/from mine facilities and covers; estimation of amount of precipitation entering pit lake.
	Water balance (infiltration, runoff, evapotranspiration) + contaminant transport	SESOIL (Bonazountas and Wagner, 1981, 1984); PRZM 3 (Version 3, Carsel et al, 1984; US EPA, 2003a); HSPF (Bicknell et al, 1997); LEACHM (Wagenet and Hudson, 1987)	Same as above plus source concentrations/loads, initial soil concentrations, contaminant fate/transport parameters (e.g., adsorption, precipitation).	Quantity and quality of infiltration and runoff from/to mine facilities.

Vadose Zone Codes	Vadose zone percolation	1D codes: SESOIL; HELP; CHEMFLO-2000 (US EPA, 2003b); Hydrus-1D (U.S. Salinity Lab; Simonek et al, 1998); SWACROP (IGWMC); SWIM HEAPCOV (Sulphide Solutions); Unsat-1 (IGWMC); Unsat-H (Pacific Northwest Laboratory); 2D codes: Hydrus-2D (U.S. Salinity Lab); FEFLOW (Waterloo Hydrogeologic); SEEP/W (Geo-slope Intl., 1994); SUTRA (USGS); VS2D	Infiltration rates; any layering or heterogeneity in geologic materials; hydraulic properties of soils/geologic units such as moisture retention properties (measured or modelled).	Seepage through unsaturated portions of mine facilities (e.g., waste dumps) and underlying vadose zone.
	Vadose zone percolation and contaminant transport	SUTRA (USGS); VS2D/T (USGS, Lappala et al, 1987; Healy, 1990);	Same as above plus quality of water entering the vadose zone and initial concentrations of constituents in vadose zone; parameters describing partitioning between soil/rock and water.	As above, but with contaminant transport.

Category of code	Subcategory	Available codes	Inputs required	Modelled processes/output
Groundwater codes	Groundwater flow	MODFLOW (McDonald and Harbaugh, 1988; Harbaugh and McDonald, 1996; MODFLOW 2000); FEFLOW (Waterloo Hydrogeologic)	Hydraulic conductivity, porosity, storage characteristics, thickness of geologic units, areal recharge, surface water recharge, pumping or reinjection of water from wells, discharge to surface water; model boundaries (streams, flow barriers, etc.). For fracture flow/transport: also need fracture spacing, orientation, aperture.	Simulate mine dewatering and reflooding; flow and transport in saturated tailings.
	Groundwater flow + contaminant transport	MODFLOW with MT3D; MODFLOW-SURFACT; SUTRA (USGS); FEFLOW (Waterloo Hydrogeologic); FEMWATER (US EPA). Groundwater flow and solute transport in fractured rock: FRAC3DVS and FRACTRAN (Waterloo Hydrogeologic); TRAFRAP-WT (IGWMC)	Same as above plus contaminant input concentrations; dispersion properties of aquifer, retardation characteristics of contaminant. For fracture flow/transport: also need fracture spacing, orientation, aperture.	Contaminant transport and loading from a mine facility to groundwater or surface water.
Integrated hydrologic/ watershed codes		MIKE SHE (British Institute of Hydrology, Danish Hydraulic Institute); PRMS/MMS (Leavesley et al, 1981; 1983; USGS); HSPF (Bicknell et al, 1997; US EPA)	Same as near-surface process and groundwater codes.	Simulate all components of hydrologic flow regime (snowmelt, overland, channelized, unsaturated/saturated zone flow) and interaction between components.

Table H.2 — Description of selected geochemical codes used for predicting water quality at hardrock mine sites (after Maest et al, 2005; expanded with orchestra)

Category of code	Available codes	Special characteristics	Inputs required	Modelled processes/ outputs
Geochemical speciation and reaction path codes	WATEQ4F v.2 (Ball and Nordstrom, 1991 and database updates)	Most complete mineral database for acid drainage, redox species; database updates for uranium, chromium, and arsenic redox species.	Variable, can include: concentrations in inflows and other waters of interest (filtered), pH, temperature, redox species concentrations and/or Eh, mass and surface area, identity of minerals, infiltration rates/volume, reactive surface area; bacteria, rate constants.	Estimate concentrations of species in solution, amount of minerals precipitating from solution/dissolving from rock, pH, Eh, amount adsorbed to/desorbed from solids.
	MINEQL (Schecher and McAvoy, 1991); MINEQL+ v. 4.5 (Environmental Research Software, 2005)	Basis for MINTEQ (along with WATEQ); v. 4.5 is windows MINTEQA2 with a user interface for relational databases; T = 0 °C to 50 °C, ionic strength <0,5 mol/l.		
	MINTEQ (Allison et al, 1991)	Most complete ion exchange and sorption, supported/approved by EPA, with PHREEQE, most often applied to acid drainage problems.		
	HYDRAQL (Papelis et al, 1988)	Speciation, adsorption, organic ligands.		
	Geochemist's Workbench (Bethke, 1994; 1996 - REACT is mass transfer module)	Can include bacteria, Pitzer formulation, evaporation, mass transfer, isotopic calculations, temperature dependence for 0 °C to 300 °C, sorption, complex kinetics and decoupled redox reactions.		
	PHREEQE/PHRQPITZ (Parkhurst, 1995; Plummer and Parkhurst, 1990); PHREEQC v. 2 (Parkhurst and Appelo, 1999)	With MINTEQ, most often applied to acid drainage problems, includes Pitzer formulation, can define kinetic reactions, mass transfer, reaction path, ion exchange fluid mixing, sorption, solid-solution equilibria, 1D transport, inverse modeling (NETPATH; Plummer et al, 1991; Parkhurst, 1997), carbon		
	SOLMINEQ.88 (Kharaka et al, 1988); SOLMINEQ.GW (explained in Hitchon et al, 1996)	Most user-friendly, Pitzer, organic ligands, covers T = 0 °C to 350 °C and 1 bar to 1 000 bar pressure, mass transfer options (fluid mixing, mineral precipitation/dissolution, ion exchange, sorption).		
	GEOCHEM (Parker et al, 1995)	Speciation and mass transfer, adsorption, soil-water interactions.		

Category of code	**Available codes**	**Special characteristics**	**Inputs required**	**Modelled processes/ outputs**
	EQ3/6 (Wolery and Daveler, 1992)	Path-finding, Pitzer, evaporation, solid solution, best documented mass transfer program, kinetics, organic species	Linked with leaching test results for verification of model prediction.	Partitioning in solid and solution, extended graphing, statistical and reporting functions in LeachXS
	SOLVEQ-CHILLER (Spycher and Reed, 1990)	Reaction of fluids with solid phases, mixing of fluids, gases, evaporation, boiling, requires user to define rates and step size for reactant addition.		
	PATHARC (Alberta Research Council; Bill Gunter and Ernie Perkins)	Most user-friendly reaction path program, dissolution/ precipitation kinetics and equilibrium reactions, gases, evaporation; isothermal, does not include solid solution.		
	ORCHESTRA (Meeussen, 2003)	Object oriented, Java based, operating on multi-processor machines, user friendly selection of reactions. Dissolution/ precipitation kinetics, sorption (oxides and dissolved and particulate matter – Nicca DOnnan), solid solutions, gas interaction, Linked with LeachXS (materials and scenario database; van der Sloot et al, 2008)		
Pyrite oxidation codes	PYROX implementation of the Davis/Ritchie shrinking core model (Wunderly et al, 1995)	Simulates diffusion-limited pyrite oxidation only.	Geometry/structure of waste rock dump, pyrite content, particle size distribution, water content of rock matrix, estimates of diffusion rates of oxygen in bulk and rock matrix.	Simulate generation of acid and sulfate from oxidation of sulfides in mine units; results used with kinetic test results to estimate release of metals from oxidation; effects of barometric pumping not incorporated into the models.
	Davis/Ritchie approach (Davis and Ritchie, 1986; Davis et al, 1986; Davis and Ritchie, 1987; Ritchie, 2003)	Simulates oxygen diffusion as only mechanism for pyrite oxidation using analytical solutions.		
	FIDHELM (Kuo and Ritchie, 1999; Pantelis, 1993; Pantelis and Ritchie, 1991)	Simulates oxygen diffusion and convection as mechanisms of pyrite oxidation; output also tracks temperature.		

	TOUGH AMD (Lefebvre et al, 2002; Lefebvre and Gelinas, 1995)	Simulates unsaturated water flow, oxygen diffusion and convection, heat generation and transfer, and solute transport.		
Coupled reaction path/flow codes	PHREEQM (Appelo and Postma, 1993)	1D, uses PHREEQE, no kinetics, mixing cell, simple.	Variable, can include: infiltration rates, concentrations in inflows (e.g. kinetic test results and background groundwater), moisture contents, reactive surface area, porosity, hydraulic conductivity, soil hydraulic function parameters, diffusion coefficients, dispersivities, bacteria (if used in model),	Fate and transport of constituents in and downgradient of mine waste units, mineralogy, porosity, fluid composition.
	REACTRAN (Ortoleva et al, 1987)	1D, user-defined reaction rates, temperature gradients.		
	MPATH (Lichtner, 1985)	1D, concentration varies only with distance along flow path.		
	MINTRAN (Walter et al, 1994)	2D, uses MINTEQA2 but more rigorous calculation of flow/transport than PHREEQM, for transport in groundwater, assumes total equilibrium between fluid and rock, like PHREEQM, includes shrinking core model and 1D gas oxygen diffusion, kinetics.		
	CIRF.A (Potdevin et al, 1992; University of Illinois)	2D, T and P corrections for thermodynamic properties, multiple rate laws; output = mineralogy, porosity, fluid composition, etc.	equilibrium constants, mineralogy of downgradient aquifer and mine unit, secondary mineral-phase formation (from reaction of mine seepage with aquifer minerals), rate constants, sorption/cation-exchange capacity.	
	FMT (Novak, 1993 and 1994)	2D, finite difference, can simulate flow through fractures, Pitzer and Extended Debye-Huckel activity coefficient corrections.		
	TOUGHREACT and TOUGH2-CHEM (Xu et al, 2001)	Can simulate acid generation and buffer reactions in unsaturated media, kinetics.		
	TOUGH-AMD (Lefebvre et al 2001)	Designed specifically forwaste rock and heap leach systems, includes heat generation by acid production and oxygen convection, no attenuation mechanisms.		
	KGEOFLOW (Sevougian et al, 1992)	1D, similar to 1DReact, uses simple kinetic formulas, uses EQ3/6.		
	RETRASO (Saaltink et al, 2002)	Kinetics, sulfide mineral oxidation, transient flow, secondary mineral		

	OTIS-OTEC (Runkel et al, 1996, 1999)	1D in-stream solute transport and stream-bank storage combined with MINTEQA2, can simulate redox chemistry and sorption.		
	RT3D (Clement, 1997)	3D, multi-species, reactive transport in groundwater.		
	SULFIDOX (based on Ritchie, 1994; see Appendix 1)	Release and attenuation of acid drainage in waste rock and heap leach pads.		
	MINTOX (Gerke et al, 1998)	Tailings, 2D, sulfide oxidation and transport, diffusive gas transport.		
	MIN3P (Mayer et al, 2002)	Update of MINTOX; Finite element, steady-state and transient, variably saturated, user-set rate laws, diffusive gas transport in unsaturated zone, kinetics, sulfur redox, pH buffering, can define rate expressions.		

Category of code	Available codes	Special characteristics	Inputs required	Modelled processes/ outputs
	MULTIFLO (Lichtner, 1996)	Comprehensive general-purpose code of reactive transport, kinetic dissolution of aluminosilicate minerals.		
	PHAST (USGS)	3D transport; combines solute-transport code HST3D (Kipp, 1998) and iterates at every time step with PHREEQC.		
	CRUNCH (Steefel, 2000; see Appendix 1)	Unsaturated-zone processes, can simulate release and attenuation of acid drainage.		
	ORCHESTRA (Meeussen, 2003)	Integrated chemical reaction transport, object oriented, High level of flexibility in selection of elements, node structure and reactions. Predefined scenarios embedded in LeachXS (database/expert system)	Linked with leaching test results or field data from a database (LeachXS) for verification of model prediction.	Multi-element partitioning in solid and solution, extended graphing and reporting functions in LeachXS
Biogeochemical and reactive transport codes	BIOKEMOD (Salvage and Yeh, 1998) coupled to HYDROGEOCHEM (Yeh and Tripathi, 1989)	Simulation of reactive transport modelling with biogeochemical transformation of pollutants in groundwater.		Complexation, adsorption, ion-exchange, precipitation/ dissolution, biomass growth, degradation of chemicals by metabolism of substrates, metabolism of nutrients, and redox,

In the mining waste area, the emphasis in modelling has been strongly focused on field observations (modelling of observed contaminant profiles), prediction of drainage water quality and kinetic testing (Maest et al, 2005) and much less on a close integration of laboratory testing and mechanistic modelling at different scales of testing. Several codes cannot cope easily with new developments in element interactions (e.g organic matter interaction, gas phase reactions, unsaturated chemical reaction/transport with a full mechanistic description) and are therefore limited in their further development (e.g. PhreeqC, Geochemist workbench). A more recently developed code, like Orchestra (Meeussen, 2003) is object oriented, which implies that all chemistry and physical parameters descriptions are outside the calculation kernel. This results in greater speed and creates more flexibility in modifying any part of the chemistry, the physical aspects of a scenario or the node configuration.

H.4 Challenges

The main challenges to be dealt with in modelling are:

- to apply a full mechanistic modelling approach (including mineral precipitation/dissolution, sorption on reactive surfaces – hydrated iron, aluminium and manganese oxides, solid solutions, particulate and dissolved organic matter) to describe release of substances from mining waste;
- to integrate abiotic (oxygen ingress under saturated and partially saturated conditions) and biologically mediated sulphate oxidation with full multi element mechanistic modelling;
- to describe long term release taking relevant chemical (acidification) and physical processes (preferential flow, infiltration) in specific scenarios into account.

Annex I
(informative)

Field investigations of tailings management facilities and waste rock dumps

I.1 Introduction

When evaluating existing waste facilities it is difficult to draw the line between aspects related to the waste material and those related to the facility and its design. Discussion in this annex therefore goes beyond what is usually considered to be waste characterization.

I.2 Tailings

In most cases it is recommended to set up a conceptual model of the system with major grain size variations, likely flow patterns, groundwater table, seep locations etc. (Figure I.1). Based on this conceptual model and the purpose of the study, a field characterization scheme can be planned.

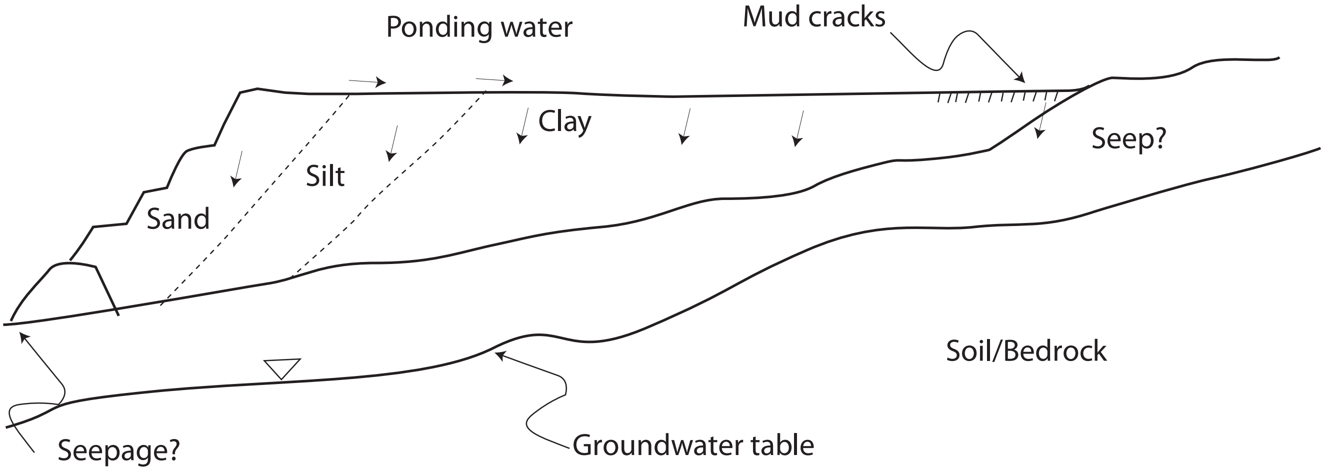

Figure I.1 — Example of conceptual model of a tailings storage facility with an upstream design. Except for the starter dike, the dam is constructed using tailings material (Walder, 2000)

Field investigations can give information about:

- air and water flow systems within the tailings;
- stage of oxidation of the tailings;
- geochemical reactions that occur which increase or reduce the metal transport through the tailings;
- mass balance and drainage chemistry.

The oxidation stage and oxidation front in deposited tailings may be estimated based on field observations in combination with laboratory analysis of oxidized and un-oxidized tailings. This would be done in order to evaluate reaction rates in the tailings impoundment. This is most commonly performed on old/closed tailings facilities but can also be performed on active tailings, primarily close to the embankment where there is enough stability to hold the equipment. Borings in fine grained tailings can commonly be performed using hand auger down to 2 m to 3 m depth. The observations that may be collected from hand auger borings are:

- colour;
- sulfide and calcite content;
- water saturation;
- grain size.

Besides hand auger boring it may be necessary to drill deeper and install wells. Deeper drilling may allow for sampling of solids and water. Using a hollow stem auger with a split spoon will give the possibility to log grain-size layers, colours, and saturation with depth. The drilling may also give indications on good locations to install wells. It is preferable to install nested wells with a bentonite seal in between (see standards for well installation). In these nested wells it is also possible to install lines for gas analysis at different depths.

Potential measurements in wells include:

- water table changes with time;
- water chemistry changes with time from extracted water;
- gas chemistry changes with time;
- infiltration rate.

Analysis of the solid samples may include tests as described earlier; however, more information can be obtained in the field:

- mineralogy (in the field with a hand lens and laboratory analysis);
- chemical composition (laboratory analysis);
- grain size (using simple field methods for the bulk grain sizes and laboratory analysis);
- hydraulic conductivity (infiltration tests in the field and laboratory tests);
- moisture content (saturation index) (field observations and laboratory analysis);
- stability (field and laboratory analysis);
- paste pH and TDS, totally dissolved solids (measurements in the field and laboratory analysis);
- ABA (laboratory analysis); and
- drainage chemistry (rainfall simulation tests or similar in the field and laboratory analysis).

Sampling of seepage water or groundwater below the tailings pile could provide information on the geochemical processes taking place within the pile and in that way work as a full scale column test. However, interpretation of the data can be difficult due to the potential influence of many competing geochemical and geophysical processes.

Drilling in tailings impoundments and dams, and to some extent in waste rock dumps, opens conduits for surface water infiltration and air permeability which may increase weathering and leaching. If drilling is performed through the material to underlying soil, this can result in contamination of groundwater. Drill-holes, therefore, may need to be plugged properly to avoid negative impacts on the surrounding environment (see also 5.4.4).

I.3 Waste rocks

Depending on the context, i.e. potential concerns and existing information, an investigation of an existing waste rock dump may take on very different forms and levels of detail. In any case, as for tailings, a recommended starting point is to develop a conceptual model of the waste rock dump to be investigated.

Waste rock dumps are commonly constructed by dumping of the material either via conveyer belts or dump trucks (Figure I.2). The material is commonly from boulder size down to very fine grained. The grain size distribution depends upon the blasting system and the strength of the rocks making up the waste material.

When the material is placed on the waste dumps it results in a grain size segregation where the coarser material will go the furthest down the outslope while finer particles tend to stay near the top. Each dump may also represent a structure parallel to the outslope. If the waste dump is built in several lifts using dump trucks there will be a compacted layer on top of each lift (see Figure I.2).

Field investigations of waste rocks dumps are commonly more difficult than for tailings. The out slopes are usually unstable (deposited at angle of repose) and the particle sizes are very variable (clay size to boulders) especially for newer and open pit operations. It is therefore often necessary to use larger equipment for sampling (backhoe/track hoe) for near surface sampling and reverse-circulation drilling for solid sampling. Besides obtaining solid samples from the drilling it is possible to set instruments to measure gas composition and temperature within the waste dumps using the drill-holes.

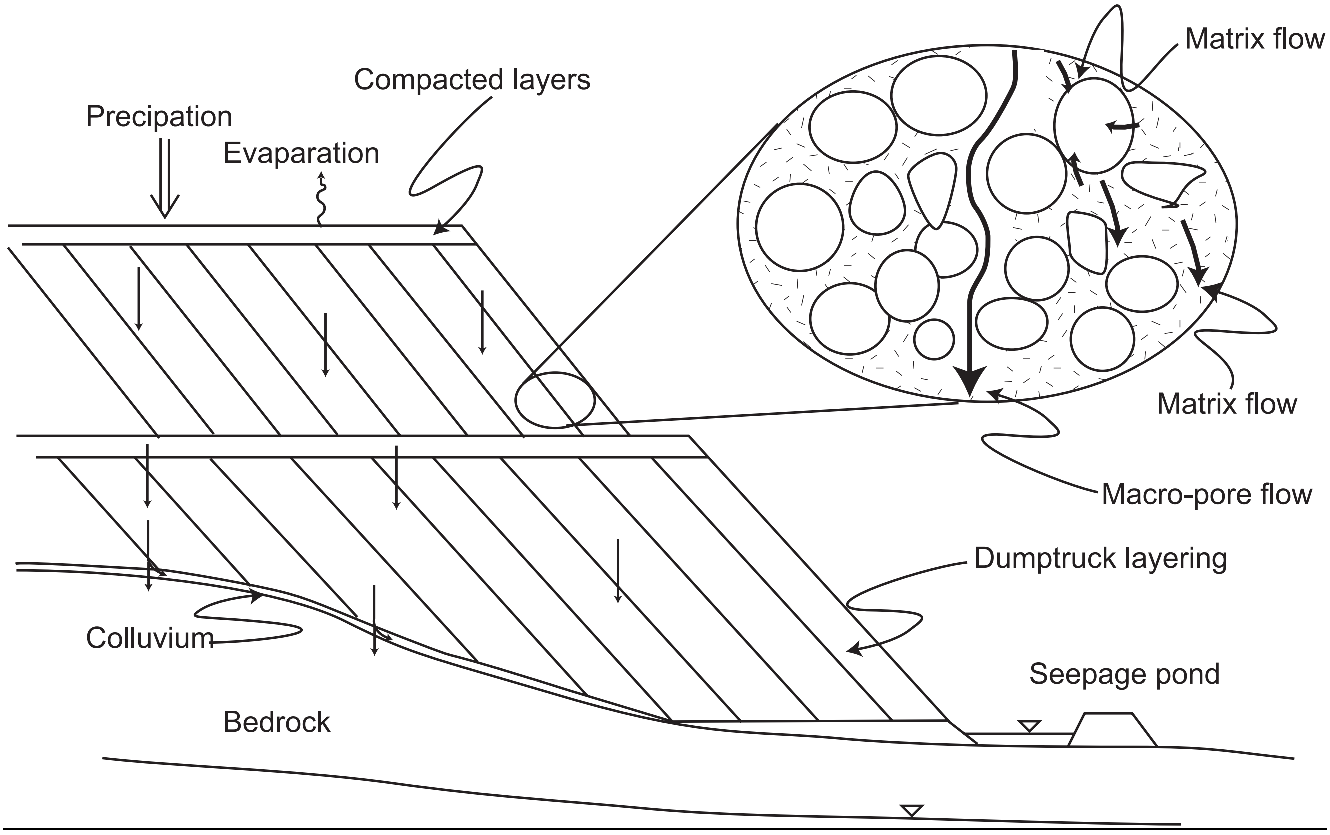

Figure I.2 — Conceptual waste rock model, showing horizontal compacted layers due to driving of heavy dump trucks, weak angled layering due to the dumping and grain segregation (from Walder and Stormont, 2004)

The possible analyses for the solids are the same as for the tailings listed in the above.

The hydraulic conductivity of waste rock piles is commonly high due to the coarseness of the material. Waste rock piles are therefore commonly unsaturated. Smith et al (1996) described a two flow system in waste rocks, macro flow and matrix flow. The macro flow is defined as the channelling flow and the water will report at the base of the pile hours to a few days after infiltrating the waste pile. The matrix flow is water that is flowing within the find grained material in the waste rocks and on the surfaces of the larger particles (Smith et al, 1996). In semi arid and arid environments, the matrix flow may be the dominant flow system (by volume) and average residence times of 50 years have been calculated for such water (Walder et al, 2006).

The chemistry of these two flow regimes can be very different due to the difference in residence time. Easily leachable minerals will primarily control the water quality of the macro-pore flow while the water quality of the matrix flow may be controlled by kinetic controlled reactions. The chemistry of the water may, however, be controlled by a third water component defined by Walder and Stormont (2004) as micro flow, which is in effect when the boulders are somewhat porous. During wetting periods, water soaks into the particles and during drying cycles elements are transported to the surface possibly forming mineral salts. The drying cycle allows air to be transported into the particles to a possible reaction front. The metal salts precipitated on the surface may be available for macro flow leaching.

The data needed to be able to estimate the matrix volume and macro volume portions of the flow system is rainfall, evaporation, infiltration rate, discharge to underlying bedrock, and continuous flow monitoring at the base of the waste pile. The continuous monitoring at the base allows for estimating the base flow as a response to rainfall, and the long-term flow system. By using the kinematic wave theory, macro and matrix flow volumes can be estimated (German and Beven, 1986) together with estimations of residence time. These estimations can be essential for calculating/modelling the long-term drainage chemistry in the waste rocks.

Seepage monitoring and groundwater monitoring near or under the waste rock pile can be very useful for evaluating the geochemical processes possibly taking place within the waste material. A continuous water flow monitoring of the seep, together with a weather station would facilitate an evaluation of the matrix-macro flow system. The continuous water flow monitoring can be complemented with e.g. pH and conductivity probes (together with water sampling) that will aid in understanding the chemical relation between matrix and macro pore flow

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