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Karakterizacija odpadkov - Kinetični preskusi za ocenjevanje celotne kislinske kapacitete odpadkov iz industrije bogatenja mineralnih surovin, ki vsebujejo sulfid

Characterization of waste - Kinetic testing for assessing acid generation potential of sulfidic waste from extractive industries

Charakterisierung von Abfällen - Kinetische Prüfung zur Bewertung des Säurebildungsverhalten sulfidischer Abfälle der mineralgewinnenden Industrie

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Characterization of waste - Kinetic testing for assessing acid generation potential of sulfidic waste from extractive industries

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Foreword

This document (FprCEN/TR 16363:2011) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NEN.

This document is currently submitted to the Technical Committee Approval.

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.

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Introduction

A specific feature of sulfide containing waste is the risk for acid/neutral drainage generation (A/NRD). Acid drainage occurs if the acid generation from sulfide oxidation exceeds the acid buffering from minerals in the waste while, in this context, neutral drainage occurs when neutralisation generation exceeds the acid generation.

Test methods for the determination of acid generation behaviour can be divided into static and kinetic tests. A static test is used for screening purposes. It is usually relatively fast to perform, but gives only indicative information based on total content of sulfur (or sulfides) and of readily available buffering minerals in the waste material. Kinetic tests give more detailed information on behaviour based on the determination of mineral reaction rates under specified conditions. A European standard, EN 15875, has been established for the static testing, while this technical report gives guidance on how the kinetic testing may be performed and interpreted.

Kinetic testing has been required as part of permit processes for many new and operating mine sites. Many different test methods have been used over the last 20 to 30 years. These tests are commonly designed to avoid that the oxidation rate is limited due to the lack of oxygen or build-up of secondary minerals. This way of accelerating the reactions is often seen as of way to simulate long-term acid generation. Kinetic tests based current standards and laboratory-scale standard practise (ASTM D5744 - 96:2001 on and ASTM D5744 - 07:2007; Morin and Hutt, 1997; Lapakko, 2003) are not designed to evaluate short- and longterm drainage water quality. However, adjustments to the standard protocols can be done to produce indicative information about short-term drainage water quality. Together with modelling, this information can be used to predict/estimate long-term drainage water quality.

This technical report is a guidance document that discusses the main kinetic test methods that are used within the mining sector internationally, the applicability of the different tests and how to evaluate the results. Kinetic test results may provide valuable information, but it is important to understand their limitations. Sulfide oxidation in the field is controlled by many different factors that may be difficult to simulate within the laboratory. Some of these factors may in fact be unknown at the time of testing. The complexity of applying test results to field conditions may to some extent be balanced by long experience in evaluating such data.

The objective of this technical report is to support the management of waste from extractive industries by giving guidance on how to characterize the kinetically controlled process of acid drainage generation.

The target audience of the document includes all stakeholders concerned with the management of extractive waste including the extractive industry, authorities, regulators, consultants, and testing laboratories.

Document structure

This technical report is organised to provide the answers to the two main questions below.

What type of data will kinetic testing provide and what methods are available?	After introducing the concepts of kinetic testing for assessing acid generation potential of sulfidic waste, this clause (Clause 2) describes what type of information these tests provide. This clause also reviews the different tests methods and the ability to meet the objectives set out for the different kinetic tests. Methods to evaluate both acid generating reactions and neutralizing
Clause 2 Methods	reactions are described.
How can the data be interpreted?	This clause (Clause 3) gives guidance on how results from kinetic tests can be applied. Included in this clause is guidance on how results from the tests may be used to calculate the bulk oxidation rate for the material; to evaluate the leaching
Clause 3 Interpretation	rates for elements within the test system; and based on the results, to evaluate mineral reactions in the system. Kinetic test relevance for describing field scale

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and evaluation

processes is discussed.

What method to select?

The clause ends with recommendations on the selection of kinetic test design depending on objective(s).

Clause 4 Recommendations

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1 Scope

This Technical Report describes the performance and evaluation of kinetic tests for sulfidic waste material that, according to previous testing (primarily acid base accounting), is likely to go acidic or when the result of such testing is inconclusive. This Technical Report also covers the issue of drainage from sulfidic material that is likely to be well buffered but that will produce a neutral drainage potentially affected by sulfide mineral oxidation.

This Technical Report will not include aspects of sampling and testing that are already covered in the overall guidance document for characterisation of extractive waste (WI 00292066) or in the guidance document on sampling of wastes from extractive industries (FprCEN/TR 16365).

2 Methods

2.1 General

It is necessary to have a good understanding of the waste material before kinetic (mineral reaction rate) testing is performed. This together with well-defined objectives will aid in selecting the methods. This clause describes the planning of kinetic testing, key elements to analyse for, and the main methods used by the industry.

2.2 Planning

Figure 1 shows a flow chart of the different steps to consider when planning for kinetic testing. A number of the steps in the flow chart are not further discussed in this document. More details on topics related to sampling are found in FprCEN/TR 16365, e.g. supporting information, data quality, documentation and reporting are discussed in overall guidance document (WI 00292066). Additional information that puts kinetic testing in a wider context may also be found in the overall guidance document.

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Figure 1 — General outline of the steps involved when performing kinetic testing for assessing acid/neutral generation potential of sulfidic waste from extractive industries

The only kinetic test method that has been standardized is the so-called Humidity cell test (ASTM D5744 - 96:2001 and ASTM D5744 - 07:2007; Sobek et al, 1978). This method has been used extensively in the mining sector. The method is designed to evaluate long-term acid generation potential and not to predict long term mineral reactions and mineral leaching in the actual tailings management facility (TMF) or waste rock dump, as pointed out by Sobek et al. (1978) and re-emphasized by Lapakko et al. (2003) and EIPPCB (2004).

Kinetic tests can be designed as small laboratory tests or large-scale field tests. During the exploration phase only smaller amounts of material are available and Humidity cell tests are the most common kinetic test used. The interpretation of the Humidity cell tests may help in defining feasible waste management options.

Most of the laboratory tests are run with relatively small amounts of crushed material (a few hundred grams to a few kilograms) with an optimal amount of oxygen available. The amount of rinse solution used is intended to be high enough to ensure removal of all reaction products, so that secondary precipitates do not limit reactions. However, at higher pH (> 4 to 5) iron oxides are likely to precipitate.

If the exploration project proceeds into mining, larger amounts of material will become available for testing. This may give the opportunity to design and run tests that are larger and/or more suited to site-specific conditions (column tests, lysimeter tests, field tests, etc.). These tests will give more reliable results for evaluating the long-term oxidation and leaching rates.

Kinetic testing may be performed several times through the lifetime of an extractive operation. It is common to establish field tests with extensive instrumentation at an early stage of operation. These field tests can be considered kinetic verification tests and will give valuable information for the final planning for closure.

In summary, the main kinetic tests designs used by extractive industries internationally are:

- Humidity cell tests;
- Column tests;

Lysimeter tests; and

— Field tests.

The Humidity cell has a standard protocol while the other methods are site specific and not standardized. In practise, also the Humidity cell tests that are being run are for or by the extractive industry commonly deviate from the standard design by introducing more site-specific aspects.

If the Humidity cell protocols are followed, the reaction products are to be flushed out at cyclic intervals. Column experiments can, however, be designed to allow for build-up of secondary minerals by reducing the water amount for flushing. The column is likely to induce a concentration gradient along the length of the axis in the flow direction.

There are also other tests methods that can be useful for testing certain processes and reaction rates under given conditions. The listed four most commonly used tests are described in the following sections complemented by a few additional tests that may be useful for evaluating reaction and leaching rates.

2.3 Testing data

The kinetic testing data to be obtained from the different tests will depend on the defined objective(s). The primary data commonly include pH, alkalinity, sulfate and weight of the sample. However, when analysing leachate samples, it is often beneficial for the understanding of the processes within the tested material to do a multi-element analysis. Kinetic testing requires collecting and analysing many samples over a long period of time (months to years). Only a few basic parameters are normally analysed on a regular basis. When there is a significant change in the basic parameters (e.g. pH and sulfate, see below), a full chemical analysis of the leachate may be performed to better understand the processes taking place and to provide input data for estimations/evaluations of drainage water quality.

The key parameters will commonly include:

- Alkalinity; <u>SIST-TP CEN/TR 16363:2013</u> https://standards.iteh.ai/catalog/standards/sist/52996adc-cbcc-4745-acb9 pH; fb37d67ca2ba/sist-tp-cen-tr-16363-2013
- Sulfate;
- Total dissolved solids;
- Key metals (copper for copper mines, nickel for nickel mines, etc.); and
- Element concentrations (anions and cations) in the leachate.

In order to understand the geochemical processes taking place within the tests columns information may also be needed on test conditions and on the tested material. Relevant information may include:

- Flow rate of air and water;
- Oxygen / carbon dioxide (during the test run, under sealed conditions);
- Temperature (during the test run);
- Mineralogy/speciation (before and after testing);
- Speciation/element availability (before and after testing) and;
- Grain size or surface area evaluation (before testing).

The data is commonly plotted in time versus concentration or accumulated concentrations (Figure 2). These types of plots help in understanding the processes taking place within the testing material, under the given conditions of the tests.





2.4 Humidity cell test

In the late 1960s, kinetic tests were defined to evaluate and predict acid drainage from coal wastes (Caruccio, 1968), then called Humidity cell tests. However, the method that has been most commonly used is the method designed by Sobek et al. (1978) called simulated weathering cells, also referred to as Humidity cell tests. This test setup has been modified to be more applicable for waste rock material and larger samples. The original method used 200 g material crushed to less than 2 mm placed in a "shoe box" container; while the later setup (ASTM D5744 - 96, 1996 and 2001, and ASTM D5744 - 07:2007) suggests using a 1 kg to 2 kg sample crushed to less than 6,5 mm grain size in a column rather than a shoe box.

The Humidity cell test is designed to: 1b37d67ca2ba/sist-tp-cen-tr-16363-2013

- determine if the material can go acidic or not; and
- assess the rate of oxidation under laboratory conditions.

The tests are commonly performed using 2 kg to 5 kg crushed material (< 6 mm). The material is placed in a column with a lid. Air is pumped through the column. The original procedure specifies alternating dry air-humid air, three days each while Price (2009) and EPA method 1627 (2009) recommends using only humid air. EPA method 1627 also recommends adding 10 % CO₂ to the humid air. Once a week, the sample is rinsed with a specific volume of water and drained. The collected water is measured and analysed for parameters as listed above (2.2). The tests are commonly run for at least 20 weeks, but in many cases up to a year or more.

The size of the columns may vary depending on the type of material used. Figure 3 shows a typical column test setup based on the concepts of the Humidity cell test (ASTM D5744 - 96:2001 and ASTM D5744 - 07:2007; Price, 2009).