SLOVENSKI STANDARD
SIST-TS CEN/TS 16229:2012

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Karakterizacija odpadkov - Vzorčenje in analiza lahko sprostljivega cianida iz usedalnikov

Characterization of waste - Sampling and analysis of weak acid dissociable cyanide discharged into tailings ponds

Charakterisierung von Abfällen - Probenahme und Analyse von mit schwachen Säuren freisetzbare Cyanide bei der Einleitung in Absetzteiche

Caractérisation des déchets - Échantillonnage et analyse des cyanures à acide faible dissociable déversés dans des bassins à stériles

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Foreword

This document (CEN/TS 16229:2011) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, (Mandate M/395), which assigned the development of standards on the characterization of waste from extractive industries

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Introduction

As gold typically occurs at very low concentrations it is often extracted from the ore using a cyanide leaching process. The waste from which the gold was removed is referred to as residue or tailings material. Tailings, usually slurry, are pumped to a pond after treatment with for example hydrogen peroxide or sulfur dioxide to destroy cyanides.

In “Directive 2006/21/EC on the management of waste from extractive industries” [1] the following is addressed in Article 13; no.6:

In the case of a pond involving the presence of cyanide, the operator shall ensure that the concentration of weak acid dissociable cyanide in the pond is reduced to the lowest possible level using best available techniques and, in any case, at waste facilities which have previously been granted a permit or have already been in operation on 1 May 2008 that the concentration of weak acid dissociable cyanide at the point of discharge of the tailings from the processing plant into the pond does not exceed 50 ppm as from 1 May 2008, 25 ppm as from 1 May 2013, 10 ppm as from 1 May 2018 and 10 ppm at waste facilities which are granted a permit after 1 May 2008.

Methods for sampling and analysis have been selected to ensure the aim of this directive. Methods described here are either EN or ISO standards with consideration of the Cyanide Code [2].

1 Scope

This CEN Technical Specification specifies methods for sampling and analysis of weak acid dissociable cyanide discharged into tailings ponds.

NOTE The document can be used to support the requirements in the Directive 2006/21/EC of the European Parliament and of the Council of 15 March 2006 on the management of waste from extractive industries.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14899, *Characterization of waste – Sampling of waste materials – Framework for the preparation and application of a Sampling Plan*

EN ISO 14403, *Water quality – Determination of total cyanide and free cyanide by continuous flow analysis (ISO 14403:2002)*

ISO 6703-2, *Water quality – Determination of cyanide – Part 2: Determination of easily liberatable cyanide*

3 Terms and definitions

3.1
tailings pond
engineered facility for managing tailings resulting from ore processing and for clearing and recycling of process water

NOTE Tailings ponds are most of the times formed by a dam construction. They mainly contain tailings along with varying amounts of free water [3].

3.2
cyanide
chemical compound that contains the cyano group (C≡N), occurs either as free cyanide or bound to a metal in complex form

NOTE Free cyanide is very toxic.

3.3
weak acid dissociable (WAD) cyanide
cyanide from substances with cyanide groups and a measurable hydrocyanic acid vapour pressure at pH = 4 and room temperature.

[ISO 6703-2]

NOTE 1 Such substances include all cyanides which will undergo chlorination, especially hydrocyanic acid, alkali- and alkali earth metal cyanides, and complex cyanides of zinc, cadmium, silver, copper and nickel. Complex cyanides of iron and cobalt, nitriles, cyanates, thiocyanates and cyanogen chloride are not included

NOTE 2 Weak acid dissociable cyanide is also referred to as “easily liberatable cyanide”.

4 Safety procedures

Attention is drawn to the toxicity of cyanide and to the need to take extreme care when handling cyanides and their solutions. Safety precautions such as safety glasses, gloves and protective clothing must be rigorously observed.

Carry out all operations regarding sample pretreatment and analysis in a fume cupboard. Avoid contact with the skin and eyes. When pipetting, always use a safety pipette (pipette by bulb). Always measure pH before sample handling. If pH < 10, add a small amount of sodium hydroxide solution and measure again to ensure pH ≥ 10. If necessary, add more sodium hydroxide solution.

Detoxify samples and solutions containing cyanides or heavy metals in accordance with local official regulations.

5 Apparatus and reagents

5.1 Apparatus

5.1.1 Clean sampling vessel, with a volume of 0,5 l to 1 l, made of plastic (e.g. polyethylene) or metal, preferably with wide neck and a screw cap

5.1.2 Separating funnel, either filter funnel or Büchner type funnel

5.1.3 Filter made of e.g.cellulose (preferably wet-strengthened) or glass fiber, plain, pore size 5 µm to 10 µm

5.1.4 Filter made of e.g. cellulose acetate, pore size 0,45 µm

5.1.5 pH meter, with a glass electrode

5.2 Reagents

5.2.1 Sodium hydroxide, solution, c(NaOH) = 5 mol/l

5.2.2 Lead acetate test paper

5.2.3 Potassium iodide/starch test papers

5.2.4 Sodium arsenite ($NaAsO_2$)

5.2.5 Lead carbonate ($PbCO_3$) or lead nitrate ($Pb(NO_3)_2$) powder

6 Sampling procedures

6.1 General

Before setting up a sampling plan, check what the purpose of sampling and analysis will be. The results obtained should reflect the actual situation. The place of sampling has to be chosen that it is representative for the effluents (tailings) pumped to the pond. In addition, the number of individual samples per time period has to be chosen according to the constancy of operation. With a steady state operation of the plant, fewer samples per time unit are required, with intermittent or batch wise operations more samples are needed. It has to be ensured that the limit values set by the directive or national authorities are met with the sampling and consequent analysis scheme.

6.2 Sampling plan

Prepare a sampling plan according to EN 14889. This sampling plan shall specify at least:

- place of sampling;
- method of sampling;
- number of samples per time period (e.g. day);
- minimum sample size;
- use of individual or composite samples for analysis (or both, if appropriate).

NOTE Individual or spot sampling only gives information on the concentrations of weak acid dissociable cyanide during a very short period of time while composite samples cover a wider time range.

- sample handling in the field;
- transport to the laboratory;
- chain of custody.

6.3 Choice of the sampling location

6.3.1 General

According to the directive, the sample should be taken at the point of discharge. This, however, is not always possible as the point of discharge is often not accessible without endangering sampling personnel. In this case sampling may take place at a convenient and accessible site at the slurry pipeline or near the outlet of the cyanide destruction tank. This will not lead to different results as the travel time of the slurry usually is less than one hour before reaching the pond and concentration of cyanides will not increase during pumping through the pipeline.

6.3.2 Slurry sampling at the pipeline

Open the spigot at the tailings pipeline and purge for a sufficient time, e.g. 1 min. Hold the sampling vessel into the slurry stream flowing out of the spigot and purge the vessel for a sufficient time, e.g. 30 s. Fill the sampling vessel completely and screw on the cap. Label the sampling vessel for further identification.

Transfer the sample to the laboratory without undue delay.

6.3.3 Slurry sampling at the destruction tank

Completely fill the sampling vessel at the outlet of the destruction tank. Discard the content back to the tank and fill again. Label the sampling vessel for further identification.

Transfer the sample to the laboratory without undue delay.

6.4 Sample handling in field

Transport the samples to the laboratory without undue delay. If this is not possible, store samples in a portable cool box (with some ice packs) and keep dark during transport and bring to the analytical laboratory as soon as possible

If the analytical laboratory in off-site, first check for oxidizing matter and remove it, if present, according to 7.3. Then use the fastest transportation available.