SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17943:2023

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Karakterizacija odpadkov - Navodilo za določevanje elementov in drugih snovi v odpadkih

Characterization of waste - Guidance on the determination of the content of elements and substances in waste

Charakterisierung von Abfällen - Leitlinien zur Bestimmung des Gehalts von Elementen und Stoffen in Abfällen

Caractérisation des déchets - Guide pour la détermination de la teneur en éléments et substances dans les déchets

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Characterization of waste - Guidance on the determination of the content of elements and substances in waste

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This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 444.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (FprCEN/TS 17943:2023) has been prepared by Technical Committee CEN/TC 444 “Environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This document is currently submitted to the Vote on TS.

Introduction

This document is intended to be used for the characterization of waste when information is needed to fulfil the requirements of the Waste Framework Directive 2008/98/EC and the EC Regulation (EU) No 1357/2014, for the classification of waste according to different hazards. This document can also be used for the Classification Labelling and Packaging (CLP) Regulation (EC) No 1272/2008.

This document deals with the determination of elements and substances in waste to assess their hazards if no information is available on their content.

1 Scope

This document provides guidance to the characterization of waste. It applies to all types of waste, with unknown or partially known composition, by giving examples of EN standards dedicated to waste characterization and analytical methods for parameters not covered by standards. Some requirements concerning the determination of inorganic elements and organic substances content in waste are given to achieve approximately 90 % or the highest possible mass.

In case information on the origin or on the composition of the waste is given by the owner of the waste, it might be sufficient to follow only part of this document to complete missing knowledge about the waste.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
dry matter
DM
mass fraction of sample excluding water expressed as mass fraction calculated by determination of dry residue or water content

3.2
volatile organic compounds
VOC
any organic compound having an initial boiling point less than or equal to 250 °C measured at a standard atmospheric pressure of 101,3 kPa

Note 1 to entry: According to WHO, organic compound whose boiling point is in the range from (50 °C to 100 °C) to (240 °C to 260 °C).

3.3
semi-volatile organic compounds
SVOC
Organic compound whose boiling point is in the range from (240 °C to 260 °C) to (380 °C to 400 °C), according to WHO

Note 1 to entry: Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for classification of compound volatility that can be used for classification of organic chemicals. SVOCs have vapour pressure between 10^{-2} mPa and 10 Pa.

3.4
unidentified volatile or semi-volatile compounds
mass calculated from the unresolved chromatographic areas of the corresponding chromatograms

3.5
non-extractable organic compounds
mass lost by calcination of the dried solid residue remaining after the extraction of semi-volatile substances

Note 1 to entry: Chemical compound that remains un-extractable by a method which do not significantly change the chemical nature of these residues.

3.6
internal calibration standards
compounds added in a known amount to the sample from the beginning of the protocol and enabling analytical coverage throughout the procedure, and that is used to correct for losses during sample preparation and analysis by accounting for all-system matrix effects (recoveries, ionization effect, variability of the detector response of the instrument for example)

3.7
external calibration standards
compounds added in a known amount to a sample which is analysed separately from the unknown sample under identical conditions, and used to facilitate the qualitative identification and/or quantitative determination of the sample components

3.8
unresolved chromatographic peak
portion of a chromatogram where a separation is incomplete, and corresponding to two or more components which may elute together from the chromatographic column

4 Principle

This method consists of determining the content of elements and substances above 0,1 % (weight) in content of waste. It applies to laboratory samples of liquid and solid wastes. It is based on standard methods when available, or otherwise on non-standardized methods as briefly described in this document, specifying any extraction procedure and quantitative or semiquantitative analytical method.

All work should be led in compliance with EN 16457. Sampling should be performed in compliance with EN 14899 and with guidance documents CEN/TR 15310-1 to 5. Laboratory samples should be prepared according to EN 15002. All standards to which this document refers to are listed in Table 1.

5 Example of analytical methods

Table 1 — Overview of examples of analytical methods

Analysis or test	Test method for liquid waste	Test method for solid waste
Sample preparation	EN 15002	EN 15002
Dry matter	EN 15934	EN 15934
Loss on ignition	EN 15935	EN 15935
pH	CEN/TR 16192 EN ISO 10523	CEN/TR 16192 EN ISO 10523
Conductivity	CEN/TR 16192 EN 27888	CEN/TR 16192 EN 27888
Halogens (bromide, chloride, fluoride)	CEN/TR 16192 ISO 10304-1	CEN/TR 16192 ISO 10304-1
Anions (nitrates, orthophosphates, sulfates)	CEN/TR 16192 ISO 10304-1	CEN/TR 16192 ISO 10304-1
Easily liberable cyanides, free cyanides and total cyanides	EN ISO 14403-1 EN ISO 14403-2	ISO 11262 EN ISO 17380
Elements (As, Ba, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Sb, se, Zn)	EN ISO 11885 EN ISO 17294-2	ISO 22036 EN 16170
Elements (semiquantitative analysis)	EN 15309 (X-Ray fluorescence) EN ISO 11885 EN ISO 17294-2	EN 15309 (X-Ray fluorescence) ISO 22036 or EN 16170
Cr VI	EN ISO 23913 or ISO 11083	EN ISO 15192
BTEX	EN ISO 17943	EN ISO 22155
HAP	ISO 28540	EN 17503
Hydrocarbon content C10-C40 range	EN 14039	EN 14039

6 Laboratory sample and preparation of test portions

Given the sheer number of potential analyses and pre-treatments (including any separation of a sample's component liquid and solid phases), it is recommended that the laboratory sample of liquids should be at least 10L and solids (powders and sludges) should be at least 10 kg.

Analytical test portions taken from the laboratory sample should be prepared according to EN 15002. When samples are composed of several non-miscible phases or fractions, the analyses should be carried out on each phase, and the results aggregated to give a complete result covering the whole sample.

By convention, a waste sample is considered liquid if it flows freely from the orifice of a container within a limited time (Annex B of EN 12457-2:2002).

7 Analysis of liquid wastes

7.1 General

The organigram of tasks proposed for the liquid waste analysis is given in Annex A. All the parameters are listed and described below. The standards referred to in this clause are given as examples, but other suitable standards may be used.

7.2 Analysis

When dealing with suspended solids or separated phases in the samples, first separate the fractions using an appropriate method (filtration, centrifugation, decanting), then determine the mass of each fraction and perform the analyses of parameters on the liquid and solid fractions of each phase (EN 15002), provided the concerning fraction contains enough material to perform the test.

Determine the following parameters:

a) **density**: take at least 10 subsamples of the laboratory sample, using class A laboratory glassware, and individually weigh each subsample, it should be the same mass which is weighed. The deviation between the measurements should not be higher than ± 5 % for a series of subsamples. When compared with the mass of same volumes of water, the mean value of these masses gives an adequate evaluation of the density of the liquid sample.

b) **water content**: following EN 15934, either dry at 105 °C or run a Karl-Fisher titration depending on the waste and the presence of volatile substances. The method used should be able to measure water contents up to 99,9 % of gross weight to identify the presence of substances other than water at 0,1 % of gross weight.

c) **ignition residue content**: loss on ignition should be performed according to EN 15935 or equivalent method;

d) **pH and conductivity**: for wastewater and aqueous waste, measure the pH and the electrical conductivity directly in the sample in the field during the sampling if possible or as fast as possible in the laboratory in case of transportation, taking care to minimize exchanges with the ambient air environment.

   For paste and non-aqueous liquids (especially with hydrocarbons): the pH and the electrical conductivity should be measured in the leachate obtained according to EN 12457-2, before filtration, taking care to minimize exchanges with the laboratory air environment.

   Measure the pH and electrical conductivity according to CEN/TR 16192 which refers to EN ISO 10523 for pH determination and to EN 27888 for electrical conductivity determination.

   Record any water-triggered reaction of the waste, as detectable for example by a release of heat or gas.

   Record the leaching test performed, in the report.

e) **determination of halogen content for saline waste (if conductivity > 0,15 S/m)**: it is recommended to determine chlorides and preferably all water-soluble halogens, according to CEN/TR 16192, either in the liquid waste (wastewater or aqueous waste) or in a leachate obtained according to EN 12457-2. For example, the ion chromatography method can be used for chloride, bromide, and fluoride according to EN ISO 10304-1. A selective method can also be used for the determination of the fluoride content.

NOTE This result does not interfere with the mass balance as the halogen compounds are integrated via the residue on ignition.

f) **determination of Total Organic Carbon (TOC)**: according to EN 15936. This result does not interfere with the mass balance because carbon compounds are integrated via the organic part like identified compounds or unidentified compounds.

g) **determination of free or easily liberatable cyanide**: according to EN ISO 14403-1 or to EN ISO 14403-2.

h) **determination of the elements content (excluding the 10 heavy metals, As and Se), sulphur content, phosphorous content**: perform a total digestion according to EN 13656 and a semiquantitative scanning analysis by ICP-AES (inductively coupled plasma-atomic emission spectrometry), ICP-MS (inductively coupled plasma mass spectrometry) or an analysis by X-ray fluorescence according to EN 15309. Quantitatively determine each individual concentration of the following elements (As, Ba, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Sb, Se, Zn) according to EN ISO 11885 or EN ISO 17294-2, on the solution obtained by acid digestion.

i) **determination of the hexavalent chromium content if suspected**: it is recommended to determine the hexavalent chromium content according to EN ISO 23913 or ISO 11083;

j) **determination of the volatile organic compounds content**:

Determine the volatile organic compounds by headspace extraction according to ISO 11423-1 or EN ISO 10301 or after solvent extraction according to ISO 11423-2; the analysis is performed accordingly:

i) add a solution containing internal calibration standards to the solvent sample blanks. This solution is used to standardize the analyte response factors;

ii) first, run a semiquantitative analysis with external calibration standards, preferably using three calibration standards (e.g. toluene, trichloroethylene, hexane);

iii) when possible, and using the results obtained by the semiquantitative analysis, each volatile organic compound detected above the threshold of 0,1 % weight concentration should be quantified by, either:

- individually quantifying each compound detected with their own calibration standards (external calibration), or;

- quantifying with three calibration standards (e.g. toluene, trichloroethylene, hexane). The response factors of the detected compounds can be corrected by comparing their TIC (Total Ion Current) with the TIC responses obtained for the internal calibration standards.

- The compound identification rate should be higher than 80 %.

For the best quantification of the identified compounds, it is recommended to use the same chemical compounds as calibration standards. If different calibration standards are used, then it is preferable to create and use a database of response factors with different families of analytes (aliphatic alkanes, aromatics, ketones, alcohols, glycols, chlorinated solvents, etc.). The GC-MS response factors differ widely, according to the chemical nature of the analyte substance. The quantification may be performed by GC-FID (flame ionization detector) as the response factor depends less on the chemical nature of the analyte substance.

- Quantitatively, determine each individual volatile organo-halogenated compound (see below for the target compound list[1]);
- Quantitatively determine methanol e.g. according to CEN/TS 17847;
- Quantitatively determine each individual BTEX according to EN ISO 17943.
- Calculate the "unidentified volatile compounds" parameters by integrating the unresolved area of the chromatogram for volatile compounds.

k) **determination of the semi-volatile organic compounds content**:

Extraction:

i) for aqueous liquid samples, perform a liquid-liquid extraction of 100 ml of sample using 3 series of 25 ml dichloromethane, then, if required, concentrate the extract under nitrogen down to a final volume of 5 ml;

ii) for non-aqueous samples, prepare a 0,1 g/10 ml dilution of a mixture of hexane-acetone 50/50 v/v or any solvent able to dilute the sample.

Analysis of the extract by GC-MS, using an analytical method according to the following protocol:

i) add a solution containing internal calibration standards to the sample extracts and the blank solvent extracts. This solution is used to standardize the analytes response factors;

ii) first, perform a semiquantitative analysis, with an external calibration of the semi-volatile compounds, preferably using three calibration standards (e.g. C10, C25, C40);

iii) when possible, and using the results obtained by the semiquantitative analysis, each substance detected above the threshold of 0,1 % weight concentration should be quantified by, either:

- individually quantifying each compound detected, with their own specific calibration standards (external calibration), or;
- quantifying with 3 external calibration standards (e.g. C10, C25, C40). The response factors of the compounds detected can be corrected by comparing their TIC (Total Ion Current) with the TIC responses obtained for the internal calibration standards;
- The compound identification rate should be higher than 80 %.

If several substances, which cannot be discriminated, are detected, express the result as the sum of the substances and assign it the CAS Number of the most toxic substance.

[1] target compound list: vinyl chloride, trichlorofluoromethane, 1,1-dichloroethene, 3-chloropropene, dichloromethane, trans-1,2-dichloroethene, 1,1-dichloroethane, cis-1,2-dichloroethene, 2,2-dichloropropane, chloroform, bromochloromethane, 1,1,1-trichloroethane, 1-chlorobutane, 1,1-dichloropropene, 1,2-dichloroethane, tetrachloromethane, chloroacetonitrile, trichlorethene, 1,2-dichloropropane, bromodichloromethane, cis-1,3-dichloropropene, trans-1,3-dichloropropene, 1,1,2-trichloroethane, 1,3-dichloropropane, chlorodibromomethane,tetrachloroethene, chlorobenzene, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane, 1,2,3-trichloropropane, 1,4-dichloro-2-butene, 2-chlorotoluene, 4-chlorotoluene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, hexachloroethane, 1,2-dibromo-3-chloropropane, 1,2,4-trichlorobenzene, hexachloro-1,3-butadiene, 1,2,3-trichlorobenzene.