SLOVENSKI STANDARD
SIST EN 13656:2020

01-december-2020

Nadomešča:
SIST EN 13656:2004

Tla, obdelani biološki odpadki, blato in odpadki - Razklop z zmesjo klorovodikove kisline (HCl), dušikove(V) kisline (HNO3) in tetrafluoroborove kisline (HBF4) ali fluorovodikove kisline (HF) za določevanje elementov

Soil, treated biowaste, sludge and waste - Digestion with a hydrochloric (HCl), nitric (HNO3) and tetrafluoroboric (HBF4) or hydrofluoric (HF) acid mixture for subsequent determination of elements

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Boden, behandelter Bioabfall, Schlamm und Abfall - Aufschluss mit einem Gemisch aus Salzsäure (HCl), Salpetersäure (HNO3) und Tetrafluorborsäure (HBF4) oder Fluorwasserstoffsäure (HF) für die anschließende Bestimmung der Elemente

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Sols, bio-déchets traités, boues et déchets - Digestion par un mélange d'acides chlorhydrique (HCl), nitrique (HNO3) et tétrafluoroborique (HBF4) ou fluorhydrique (HF) pour la détermination ultérieure des éléments

Ta slovenski standard je istoveten z: **EN 13656:2020**

ICS:

13.030.40	Naprave in oprema za odstranjevanje in obdelavo odpadkov	Installations and equipment for waste disposal and treatment

SIST EN 13656:2020 **en,fr,de**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 13656

November 2020

ICS 13.030.40

Supersedes EN 13656:2002

English Version

Soil, treated biowaste, sludge and waste - Digestion with a hydrochloric (HCl), nitric (HNO_3) and tetrafluoroboric (HBF_4) or hydrofluoric (HF) acid mixture for subsequent determination of elements

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This European Standard was approved by CEN on 21 September 2020.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (EN 13656:2020) has been prepared by Technical Committee CEN/TC 444 “Environmental characterization of solid matrices”, of which the secretariat is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2021 and conflicting national standards shall be withdrawn at the latest by May 2021.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 13656:2002.

In comparison with EN 13656:2002, the following modifications have been made:

— addition of HBF_4 as acid. For safety reasons the use of HBF_4 is preferred over HF;

— addition of a heating block digestion procedure;

— addition of a microwave digestion procedure temperature-controlled;

— removal of the microwave digestion with semi-open vessel system.

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Introduction

The methods specified in this document are providing a multi-element digestion of soil, treated biowaste, sludge and waste prior to analysis. Elements extractable by this procedure can in many instances be described as “total”, although this will be sample dependent. On the other hand, they cannot be regarded as available for leaching, as the extraction procedure is too vigorous to represent natural processes.

This document is validated for several types of matrices as indicated in Table 1.

Table 1 — Matrices for which EN 13656 is validated

Materials used in the validation test	Validated for digestion with $HCl:HNO_3:HBF_4$ (see Annex B)	Validated for digestion with $HCl:HNO_3:HF$ (see Annex C)
City waste incineration ash (BCR176/BCR176R)	X	X
Ink waste sludge (organic matrix)	X	X
Electronic industry sludge (“metallic” matrix)	X	X
Sediment	X	
Coal fly ash	X	
Steel slag	X	
Copper slag	X	
City waste incineration fly ash (“oxidised” matrix)		X
City waste incineration bottom ash (“silicate” matrix)		X
Sewage sludge (BCR 146R)		X

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WARNING — Persons using this document should be familiar with usual laboratory practice. Most of the reagents used in this document are extremely corrosive and very toxic. Safety precautions are absolutely necessary, not only due to the strong corrosive reagents employed, but also to the high temperature and pressures employed.

The use of laboratory-grade microwave equipment with isolated and corrosion resistant safety devices is essential. Domestic (kitchen) type microwave ovens should not be used, as corrosion by acid vapours may compromise the function of the safety devices and prevent the microwave magnetron from shutting off when the door is open, which could result in operator significant hazardous exposure to microwave energy.

All procedures should be performed in a fume hood or in closed force-ventilated equipment. By the use of strong oxidising reagents, the formation of explosive organic intermediates is possible, especially when dealing with samples with a high organic content. Do not open pressurized vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff. People performing the test should be informed on the specific risks associated with the use of HBF_4 and HF.

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1 Scope

This document specifies three methods for the digestion of soil, treated biowaste, sludge and waste by the use of an acid mixture composed of hydrochloric (HCl), nitric (HNO_3) and tetrafluoroboric (HBF_4) or hydrochloric (HCl), nitric (HNO_3) and hydrofluoric (HF) acid as the digestion solution.

Digestion with these acids is effectively considered as a total decomposition of the sample. Elements extractable by this procedure can in many instances be described as “total”, although this will be sample dependent.

This document is applicable for the following elements:

Aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), nickel (Ni), phosphorus (P), potassium (K), selenium (Se), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tellurium (Te), thallium (Tl), tin (Sn), titanium (Ti), vanadium (V), and zinc (Zn).

This document can also be applied for the digestion of other elements, provided the user has verified the applicability.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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EN 15002, *Characterization of waste - Preparation of test portions from the laboratory sample*

EN 15934, *Sludge, treated biowaste, soil and waste - Calculation of dry matter fraction after determination of dry residue or water content*

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EN 16179, *Sludge, treated biowaste and soil - Guidance for sample pretreatment*

3 Terms and definitions

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

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

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
digestion
mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacting with a reagent mixture

3.2
dry residue
remaining mass fraction of a sample after a drying process at 105 °C under specified conditions

[SOURCE: EN 15934:2012, 3.1]

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3.3
dry matter fraction
(dm)
mass fraction of a sample excluding water expressed as mass fraction calculated by determination of dry residue or water content

[SOURCE: EN 15934:2012, 3.3]

3.4
laboratory sample
sample intended for laboratory inspection of testing

[SOURCE: EN ISO 11074:2015, 4.3.7]

3.5
sample
portion of material selected from a larger quantity of material

3.6
test portion
quantity of material, of proper size for measurement of the concentration or other property of interest, removed from the test sample

Note 1 to entry: The test portion may be taken from the laboratory sample directly if no preparation of sample is required (e.g. with liquids), but usually it is taken from the prepared test sample.

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Note 2 to entry: A unit or increment of proper homogeneity, size and fineness, needing no further preparation, may be a test portion.

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[SOURCE: EN ISO 11074:2015, 4.3.15]

3.7
test sample
portion of material resulting from the laboratory sample by means of an appropriate method of sample pretreatment and having the size (volume/mass) necessary for the desired testing or analysis

[SOURCE: EN ISO 11074:2015, 4.3.16]

4 Principle

A test portion is digested according to one of the following heating procedures:

— Method A: Heating block digestion

  — A: Digestion with HCl, HNO_3 and HBF_4 using a heating block at (105 ± 5) °C for 2 h, followed by filtration/centrifugation.

— Method B: Microwave digestion

  — B1: Digestion with HCl, HNO_3 and HBF_4 using a microwave oven with temperature-controlled procedure to an end temperature at (175 ± 5) °C in a closed vessel followed by filtration/centrifugation.

  — B2: Digestion with HCl, HNO_3 and HF using a microwave oven with power-controlled procedure in a closed vessel followed by filtration/centrifugation.

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NOTE Alternatively, microwave digestion with temperature-controlled procedure in a closed vessel can be used, provided the user has verified the applicability.

Due to safety considerations, the digestion with HCl, HNO_3 and HBF_4 is strongly recommended over the much more hazardous digestion using HCl, HNO_3 and HF.

5 Interferences and sources of errors

5.1 General information

The container in which the sample is delivered and stored can be a source of errors. Its material shall be chosen according to the elements to be determined (e.g. elemental Hg can penetrate polyethylene walls very rapidly in both directions. Glass can contaminate samples with elements that it contains: - e.g. Na, K, Al).

Grinding or milling samples includes a risk of contamination of the sample by the environment (air, dust, wear of milling equipment). Due to elevated temperature, losses of volatile elements/compounds are possible.

For the determination of elements forming volatile elements/compounds (e.g. Hg, As, Pb), special care has to be taken at sample pre-treatment.

In the case of filtration of the digested solution, it is necessary to take care that the filtration procedure does not introduce contaminants.

Ensure that all of the test portion is brought into contact with the acid mixture in the digestion vessel.

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Some elements of interest can be lost due to precipitation with ions present in the digest solution, e.g. low soluble chlorides, fluorides and sulphates.

5.2 Closed vessel system for microwave digestion

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The upper limits of mass of the test portion according to the manufacturer's specifications have to be taken into account.

Very reactive or volatile materials that may create high pressures when heated may cause a venting of the vessels with potential loss of sample and analytes. The complete decomposition of either carbonates, or carbon based samples, may cause sufficient pressure to vent the vessel.

After digestion, the vessel shall be cooled to room temperature before opening.

6 Reagents

Use only acids and reagents of recognized analytical grade or verify the content of the elements to be analysed to avoid high blank values for subsequent analytical measurements. Use a test blank solution throughout the procedure applying all steps with the same amount of acids, but without a sample.

6.1 Water, e.g. deionized.

6.2 Tetrafluoroboric acid (HBF_4), c(HBF_4) approximately 6 mol/l, w(HBF_4) = 38 % (m/m) to 48 % (m/m).

6.3 Hydrofluoric acid (HF), c(HF) approximately 23 mol/l, w(HF) = 40 % (m/m) to 45 % (m/m).

6.4 Boric acid ($B(OH)_3$), solid.

6.5 Boric acid ($B(OH)_3$) solution, e.g. 4 % (m/m) solution.

Dissolve 40 g of boric acid (6.4) in water and dilute to 1 l of water (6.1).

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6.6 Hydrochloric acid, c(HCl) approximately 12 mol/l, w(HCl) = 35 % (m/m) to 37 % (m/m).

6.7 Nitric acid, c(HNO_3) approximately 15 mol/l, w(HNO_3) = 65 % (m/m) to 70 % (m/m).

7 Apparatus

Usual laboratory apparatus. All glassware and plastics ware shall be adequately cleaned and stored in order to avoid any contamination.

Depending on the concentration of the element of interest, particular caution shall be taken with respect to the cleaning of the vessels.

7.1 Method A: heating block digestion

7.1.1 Digestion tube, 50 ml polypropylene tube with a screw cap from polyethylene.

The material of the tube and screw cap shall be tested in order to be sure that release of elements of interest does not take place. Other materials and vessels with other volumes than mentioned above are allowed to be used if suitability has been proven.

7.1.2 Temperature-controlled heating block, heating block able to heat the tube(s) to a temperature of (105 ± 5) °C.

7.2 Method B: microwave digestion, temperature and/or power-controlled, closed vessels

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7.2.1 Digestion vessel, for pressurized microwave digestion, typically of 100 ml volume, reagent-, temperature- and pressure-resistant and capable of containing the mixture of sample and digest solution. The vessel shall be suitable for the safe application in the temperature and pressure range applied, capable of withstanding pressures of at least 3 000 kPa.

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Digestion vessels made of modified polytetrafluoroethylene (PTFE) and equipped with a safety pressure releasing system to avoid explosion of the vessel, shall be used. The inner wall of the vessel shall be inert and shall not release contaminations to the digest solution.

NOTE It can be necessary to periodically clean the digestion vessels with a suitable surfactant to remove persistent deposits.

7.2.2 Microwave digestion system, temperature-controlled, corrosion resistant and well ventilated. All electronics shall be protected against corrosion for safe operation.

Use a laboratory-grade microwave oven with temperature feedback control mechanisms.

The microwave digestion system shall be able to control the temperature with an accuracy of at least ± 5 °C and automatically adjust the microwave field output power within 2 s of sensing. Temperature sensors shall be accurate to ± 2 °C, including the final reaction temperature of (175 ± 5) °C. Temperature feedback control provides the primary performance mechanism for the method. Due to the variability in sample matrix types and microwave digestion equipment (i.e. different vessel types and microwave designs), control of the temperature during digestion is important for reproducible microwave heating and comparable data.

The accuracy of the temperature measurement system shall be periodically tested at an elevated temperature according to the manufactures instructions. If the temperature deviates by more than 2 °C from the temperature measured by an external, calibrated temperature measurement system, the microwave oven temperature measurement system shall be re-calibrated.

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7.2.3 Microwave digestion system, power-controlled, corrosion resistant and well ventilated. All electronics shall be protected against corrosion for safe operation.

The microwave unit shall be able to provide programmable power which can be programmed to within ± 10 W of the required power. Typical units provide a nominal 600 W to 1 200 W of power. If necessary (referring to manufactures specifications) calibration of the microwave unit shall be performed (see Annex A).

The microwave unit shall be designed in a way that guarantees homogeneous heating of the samples.

The microwave unit cavity shall be built in a way that even in case of leakage or explosion of the vessels the safety of the operators can be guaranteed. Household instruments are not suitable for laboratory use.

The microwave unit shall include a temperature and/or pressure control system.

7.3 Sample containers, plastic and glass containers are both suitable.

7.4 Filter, usually with a pore size of 0,45 µm and resistant to the employed acid mixture.

7.5 Volumetric flasks, usually of nominal capacity of 50 ml or 100 ml.

7.6 Analytical balance, with an accuracy of 1 mg or better.

8 Procedure

8.1 General

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Pre-treat, if not otherwise specified, soil, sludge and biowaste samples according to EN 16179 and waste samples according to EN 15002.

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Determine the dry matter content, depending on the matrix of the sample, according to EN 15934.

Pre-treatment should include drying and grain size reduction below a particle size of 250 µm, or homogenizing by use of a high speed mixer or sonification for liquid samples.

The mass of test portion for a single digestion shall be selected in a way that:

— it is representative for the laboratory sample;

— it complies with the specifications of manufacturer of the digestion unit.

If the required representative test portion exceeds the manufacturers' specifications, the test portion should be divided into smaller quantities and digested separately. The individual digests should be combined prior to analysis.

8.2 Blank test

Carry out a reagent blank test digestion in parallel with the determination, using the same procedure and the same quantities of all the reagents as in the determination, but omitting the test portion. The laboratory shall define acceptable limits.

NOTE The measurement of a blank is introduced to determine the contribution of the extracting solution, digestion tube and filter used to the measured value.

8.3 Method A: Heating block digestion using HCl, HNO_3 and HBF_4

Weigh approximately 0,2 g to 0,5 g of the dried and pre-treated test portion with an accuracy of 0,001 g and transfer to the digestion tube (7.1.1).

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The amount of the test sample depends on the amount of organic matter. The maximum amount of organic carbon shall not exceed 0,15 g when 8 ml of HCl/HNO_3 mixture is used. Per additional 0,1 g organic carbon (more than this 0,15 g), 1 ml of additional concentrated HNO_3 (6.7) shall be added before the digestion process is started.

If necessary, moisten the test portion with a few drops of water (6.1). Add (6,0 ± 0,1) ml hydrochloric acid (6.6) followed by (2,0 ± 0,1) ml nitric acid (6.7) and (4,0 ± 0,1) ml tetrafluoroboric acid (6.2). Let stand at room temperature until any effervescence almost ceases to allow for slow oxidation of the organic matter in the sample.

Turn the screw cap gently (not very tight!) and place the digestion tube on the heating block (7.1.2) and slowly increase the temperature to (105 ± 5) °C. Keep this temperature during 120 min.

Let the tube cool down to room temperature and add water (6.1) to the volume mark.

If a non-graduated digestion tube is used, transfer quantitatively the solution content into a suitable sized volumetric flask and add water (6.1) to the volume mark. Alternatively, another procedure can be applied, such that the adjustment to volume shall be carried out immediately after digestion.

If the measurement solution contains suspended particles which may clog nebulizers or interfere with an injection of the sample into the instrument, the sample may be centrifuged or filtered (7.4).

If the measurement solution contains suspended particles, the digestion is not complete. This should be stated in the report.

The measurement solution is now ready for the determination of the elements of interest using appropriate elemental analysis techniques.

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8.4 Method B: Microwave digestion

8.4.1 Method B1: Digestion with HBF_4, HNO_3 and HCl using microwave heating, temperature-controlled, closed vessels

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Weigh an amount between 0,2 g to 0,5 g of the dried and pre-treated test portion with an accuracy of 0,001 g and transfer it into the digestion vessel (7.2.1).

The amount of the test sample depends on the amount of organic matter. The maximum amount of organic carbon shall not exceed 0,15 g when 8 ml of HCl/HNO_3 mixture is used. Per additional 0,1 g organic carbon (more than this 0,15 g), 1 ml of additional concentrated HNO_3 (6.7) shall be added before the digestion process is started.

Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

If necessary, moisten the test portion with a few drops of water (6.1). Add (6,0 ± 0,1) ml hydrochloric acid (6.6) followed by (2,0 ± 0,1) ml nitric acid (6.7) and (4,0 ± 0,1) ml tetrafluoroboric acid (6.2).

If a vigorous reaction occurs, allow the reaction cease before capping the vessel.

Cap the digestion vessel according to the manufacturer's instructions. Place in all positions of the microwave oven carrousel (usually 6, 12, 16 or 40 positions) sample vessels. If a lower number of samples are available compared to the vessel positions, place vessels filled with same amount of the acid mixture without sample. This is to ensure the same microwave energy absorption occurs during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions. The temperature of the digestion mixture in each vessel shall be raised with a heating rate of approximately 10 °C/min to 15 °C/min to (175 ± 5) °C and remain at (175 ± 5) °C for (10 ± 1) min. Cool down to room temperature.

WARNING — A too high temperature increase may cause a vigorous, exothermic reaction in the digestion solution with high pressure increase and blow-off of the security valve. Losses of analytes are then possible.