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

EESTI STANDARDIKESKUS
ESTONIAN CENTRE FOR STANDARDISATION

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 13656:2020 sisaldab Euroopa standardi EN 13656:2020 ingliskeelset teksti.	This Estonian standard EVS-EN 13656:2020 consists of the English text of the European standard EN 13656:2020.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 04.11.2020.	Date of Availability of the European standard is 04.11.2020.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

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EUROPEAN STANDARD

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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

Sols, bio-déchets traités, boues et déchets - Digestion par un mélange d'acides chlorhydrique (HCl), nitrique (HNO_3) et tétrafluoroborique (HBF_4) ou fluorhydrique (HF) pour la détermination ultérieure des éléments

Boden, behandelter Bioabfall, Schlamm und Abfall - Aufschluss mit einem Gemisch aus Salzsäure (HCl), Salpetersäure (HNO_3) und Tetrafluorborsäure (HBF_4) oder Fluorwasserstoffsäure (HF) für die anschließende Bestimmung der Elemente

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

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.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

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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.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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

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.

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.

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*

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]