SLOVENSKI STANDARD
SIST EN 13805:2002

01-september-2002

Živila - Določevanje elementov v sledovih - Razklop pod tlakom

Foodstuffs - Determination of trace elements - Pressure digestion

Lebensmittel - Bestimmung von Elementspuren - Druckaufschluss

Produits alimentaires - Détermination des éléments-traces - Digestion sous forte pression

Ta slovenski standard je istoveten z: **EN 13805:2002**

ICS:

67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products

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English version

Foodstuffs - Determination of trace elements - Pressure digestion

Produits alimentaires - Détermination des éléments-traces - Digestion sous forte pression

Lebensmittel - Bestimmung von Elementspuren - Druckaufschluss

This European Standard was approved by CEN on 22 February 2002.

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 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 Management Centre has the same status as the official versions.

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

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Contents

Foreword

This document (EN 13805:2002) has been prepared by Technical Committee CEN/TC 275 "Food analysis – Horizontal methods", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by September 2002, and conflicting national standards shall be withdrawn at the latest by September 2002.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Annex A is normative.

1 Scope

This European Standard specifies a method for the digestion of foodstuffs under pressure intended for use in the determination of trace elements. This method has been collaboratively tested in combination with atomic absorption (flame, furnace, hydride, cold-vapour) techniques. ICP-MS. ICP-OES and voltametry can be used in combination with the measurement standards, which make reference to this one.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 13804, *Foodstuffs — Determination of trace elements — Performance criteria, general considerations and sample preparation.*

3 Principle

Pressure digestion, physicochemical method described in this document for mineralising the sample material and for preparing a test solution containing the trace elements prior to their determination according to the standards that make reference to this one and have been validated in combination with it.

Apparatus with a low level of contamination is used to homogenise the sample, which is then digested in a sealed vessel in a pressure container at high temperature and pressure by conventional or microwave assisted heating [1], [2], [3].

4 Reagents

4.1 General

The concentration of the trace elements in the chemicals and water used shall be low enough not to affect the results of the determination.

4.2 Nitric acid, not less than 65 %, (mass fraction), having a density of approximately ρ (HNO_3) = 1,4 g/ml. In case of insufficient purity it is necessary to purify the acid in a distillation apparatus (5.5).

4.3 Diluted nitric acid, to be prepared by mixing nitric acid (4.2) and water in a proportion minimum of 1 + 9 parts by volume.

4.4 Hydrochloric acid, not less than 30 %, (mass fraction), having a density of approximately ρ (HCl) = 1,15 g/ml.

4.5 Hydrogen peroxide, not less than 30 %, (mass fraction).

5 Apparatus and equipment

5.1 General

To minimise the contamination, carefully clean all the apparatus which comes into direct contact with the sample by treatment with diluted nitric acid (4.3) and then with water. It is advisable to use a stripping apparatus (5.6) for cleaning vessels or flasks.

5.2 Pressure digestion apparatus, commercially obtainable, tested safety pressure vessels made of acid-resistant materials and having holders for the sample of acid-resistant material with low level of contamination. Apparatus is available which uses a high-pressure incinerator with or without ambient autoclave pressure.

Instead of polytetrafluoroethylene (PTFE) holders, it is better to use graduated quartz holders, perfluoro ethylene propylene (FEP) holders [4], [5] or perfluoro alkoxy (PFA) holders. Quartz is advisable to be used for the mercury determination and for decomposition temperatures above 230 °C.

5.3 Heating device, temperature-controllable (e.g. heating block or microwave oven)

5.4 Ultrasonic bath

5.5 Distillation apparatus, quartz glass or equivalent made of high purity fluoropolymers, according to Figure A.1.

5.6 Stripping apparatus, according to Figure A.2.

6 Sampling

Proceed according to prEN 13804.

7 Procedure

7.1 General

At every stage in the method, steps shall be taken to ensure that contamination is as low as possible.

WARNING — The use of this European Standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

It shall be borne in mind that the digestion of carbon-rich materials (e.g. carbohydrates, fats etc.) can result in explosions.

Before the pressure digestion apparatus is used, read operating manual and observe safety instructions. Pay particular attention to the risk to the laboratory staff posed by nitrous gases.

Detailed descriptions of the procedure shall be available in every laboratory in the form of working instructions.

7.2 Sample preparation

Prepare the samples in a way as usual for the preparation of foodstuffs in the normal household. Avoid contamination with elements to be determined to the greatest possible extent (e.g. for the determination of chromium and nickel stainless steel knives shall not be used during the sample preparation). See prEN 13804.

7.3 Pressure digestion conditions

7.3.1 Initial sample mass and acid volumes

Match the initial sample mass to the capacity of the digestion vessel, with the manufacturer's instructions being strictly observed for safety reasons.

If the capacity is e.g. 70 ml, up to 400 mg (to the nearest milligram) of dry matter or up to 4 ml of liquid, equivalent to a carbon content of 200 mg [5], can as a rule be digested. If the carbon content is lower, the test portion may be increased. The volume of acid necessary for the digestion will depend on the nature of the sample material. Usually 3 ml of nitric acid will be sufficient to digest the amounts mentioned above. For pure fat, it may be necessary to reduce the initial sample weight and to increase the amount of acid. 0,5 ml to 1 ml hydrogen peroxide may be added to prevent cementing of the sample on the wall of the digestion vessel and to achieve complete mixing of the sample with the acid.

NOTE If iron is subsequently to be determined, it can be necessary to add 0,5 ml of hydrochloric acid (4.4) to prevent losses due to adsorption on the vessel wall.

7.3.2 Digestion temperature

Determine the necessary digestion temperature and consequently the completeness of the digestion, by the measuring method subsequently used (e.g. higher temperatures result in lower values of residual carbon in the digestion solutions. Thereby the background in ET-AAS and ICP-AES measurements is reduced. Interferences in the chromium determination by ICP-MS are minimised [6] and trouble free voltametric measurements can be done).

A smooth rise in temperature at the beginning of the digestion is advantageous.

NOTE In general, it applies that the quality of the digestion will become better with increasing digestion temperature, [7], [8]. If it is likely that organic arsenic compounds are present in the food, a temperature of 320 °C can be necessary, if HG-AAS is used for subsequent determination of arsenic [9].

7.3.3 Digestion time

A suggested digestion time for homogenised sample material is about 3 h. In case of microwave systems digestion time is typically 15 min to 30 min. For some samples the digestion will be gentler if a preliminary reaction is allowed to take place at room temperature, e.g. overnight, after adding the acid.

7.3.4 Cooling

To reduce the pressure inside the digestion vessel, cool the still sealed pressure vessel to near the ambient temperature.

7.3.5 Preparation of test solution

After the digestion vessel has been cooled and opened, initially place it under a fume hood until brown fumes are no longer visible. It is highly recommended to degas the digestion solution in an ultrasonic bath. Fill up to a specified volume with water (test solution) and transfer the test solutions to vessels made of quartz (highly recommended for mercury determinations), FEP or PFA.

The digestion solution shall be clear and its volume roughly the same as before digestion. A marked reduction in volume indicates that the pressure vessel was not leak-tight and in such cases, the digestion shall be repeated.

7.3.6 Blank solution

To check for contamination, prepare a reagent blank containing the same amount of acids as in the sample and up to 4 ml of water (depending on the initial sample weight), then carry out all the steps of the method (7.3.1 to 7.3.5).

7.3.7 Reference samples

For the purpose of analytical control, analyse reference samples having reliably known contents of the elements to be determined, in parallel with all the series of samples analysed, the reference samples being subjected to all the steps of the method, starting from digestion.

7.4 Example of microwave digestion

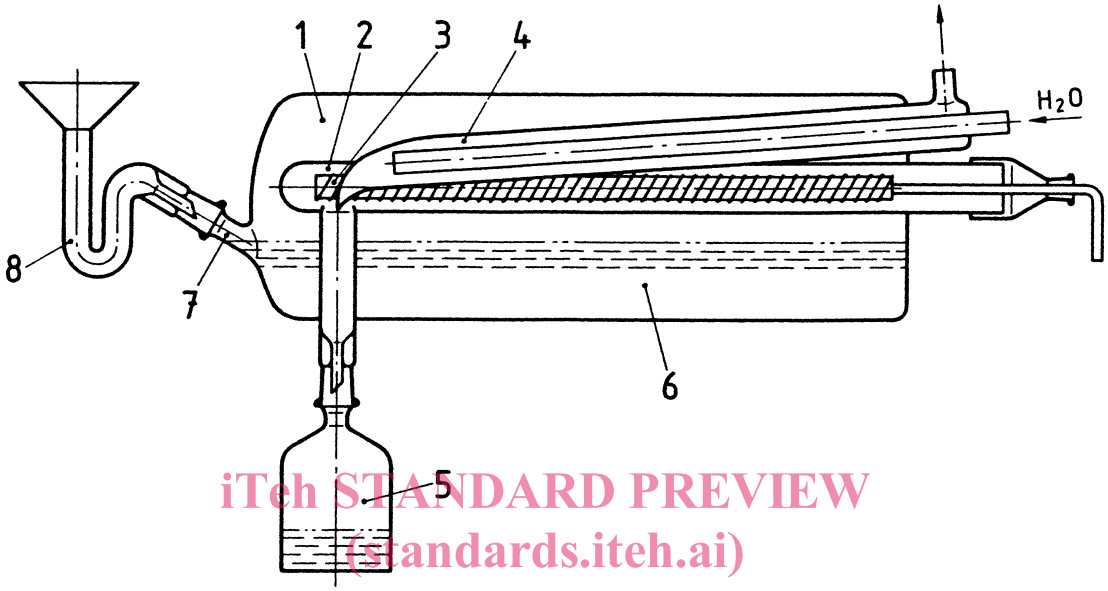
When using 70 ml to 100 ml vessels, weigh 1 g to 2 g of meat or 3 g of lettuce (fresh weight). Add 3 ml of nitric acid and 0,5 ml of hydrogen peroxide, seal the digestion vessel and the pressure holders in the correct manner. Apply low microwave energy at the beginning of the digestion and slowly raise the energy to the maximum power, e.g. start with 100 W, raise up to 600 W within 5 min, hold for 5 min, raise to 1000 W, hold for 10 min, cool down for minimum 20 min to 25 min.

7.5 Example of a high pressure asher digestion

When using a 70 ml vessel, weigh 1 g to 2 g of meat or 3 g of lettuce (fresh weight). Add 3 ml of nitric acid, seal the digestion vessel and the pressure vessel in the correct manner and heat from room temperature to 150 °C in 60 min, then to 300 °C in 40 min and keep 300 °C for 90 min before cooling down.

Annex A
(normative)

Figures of the apparatus



Key

1 Distillation chamber
2 Quartz glass tube
3 Heating filament
4 Cold finger
5 Bottle containing purified acid
6 Acid to be distilled
7 Connecting piece
8 Charging funnel

Figure A.1 — Quartz glass distillation apparatus