



SLOVENSKI STANDARD

SIST EN 13804:2013

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Nadomešča:
SIST EN 13804:2002

Živila - Določevanje elementov in njihovih kemijskih oblik - Splošna navodila in posebne zahteve

Foodstuffs - Determination of elements and their chemical species - General considerations and specific requirements

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Allgemeines und spezielle Festlegungen

Dosage des éléments et de leurs espèces chimiques - Considérations générales et exigences spécifiques

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ICS:

67.050

Splošne preskusne in
analizne metode za živilske
proizvode

General methods of tests and
analysis for food products

SIST EN 13804:2013

en,fr,de

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 13804

March 2013

ICS 67.050

Supersedes EN 13804:2002

English Version

Foodstuffs - Determination of elements and their chemical species - General considerations and specific requirements

Produits alimentaires - Détermination des éléments et de leurs espèces chimiques - Considérations générales et exigences spécifiques

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Allgemeines und spezielle Festlegungen

This European Standard was approved by CEN on 12 January 2013.

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, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 13804:2013) 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 2013, and conflicting national standards shall be withdrawn at the latest by September 2013.

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

This document supersedes EN 13804:2002.

Compared to EN 13804:2002, the following changes were made:

- a) the Scope was updated;
- b) former Clause 3, "Terms and definitions" was deleted;
- c) former Clause 4, "Performance characteristics" was deleted;
- d) former Clause 5, "Sampling" was deleted;
- e) former Clause 6, "Sample preparation" was updated and wording is now more precise and appears in the new Clause 2;
- f) Subclause 2.3.2, "Storage for **speciation purposes**" was added;
- g) Subclause 2.4.2, Table 2 on "Examples of sample preparation procedures for some foodstuffs" was thoroughly updated;
- h) Subclause 2.4.3, "Drying and lyophilisation" was added;
- i) Former Subclause 7.1, "Reagents" was revised and introduced in 3.1;
- j) Former Clause 8, "Laboratory Quality standards" was completely revised and appears now as Clause 4, "Performance requirements and characteristics";
- k) Former Clause 9, "Expression of results" was revised and appears now as Clause 5, "Interpretation and expression of results";
- l) Former Clause 10, "Test report" was specified and appears now as Clause 6;
- m) the Bibliographic references were completed;
- n) the whole document was editorially revised.

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

Introduction

The Working Group, CEN/TC 275/WG 10 "Elements and their chemical species", selects and elaborates methods of analysis of elements and their chemical species in foodstuffs.

There are many methods of analysis for the determination of elements in foodstuffs which have been validated and published; the analyst is often required to make a choice between several established methods all of which purport to be applicable to the same analyte/matrix combination. The Working Group decided to establish specific criteria to guide the analyst in the selection between several methods of analysis. As a general rule, analysts should give preference to methods of analysis which comply with the provisions given in Clauses 1 and 2 of the annex to [1], with the Decision in [2] and with the General Principles for Methods of Analysis of the Codex Alimentarius Commission (CAC), as defined in the CAC Procedural Manual and further developed in the "criteria approach" to methods of analysis developed by the Codex Committee of Methods of Analysis and Sampling (CCMAS).

The performance criteria laid down in this European Standard are based on published data or collected from official reports on European interlaboratory studies. When such performance characteristics are not available, the criteria were established based on the experience and opinions of the experts of CEN/TC 275/WG 10.

The criteria included in this European Standard have also been used as guidance in the Working Group 10 for the selection of specific methods of analysis of trace elements to be standardised.

In addition, the Working Group 10 also decided to provide some general information on sample handling and sample preparation, laboratory organisation and equipment.

This document does not deal with sampling issues. It provides an overview of the processes involved from receipt of the laboratory sample to the end result.

General sample preparation procedures are specified, as well as examples for some foodstuffs.

1 Scope

This European Standard specifies performance criteria for the selection of methods of analysis of elements and their chemical species in foodstuffs and contains performance requirements and characteristics, guidelines for laboratory set-up, sample preparation and test reports.

2 Sample preparation

2.1 General

Food samples are commonly treated in the same way as is done before consumption (washed, peeled, removal of non-edible parts). Apart from the treatments that are just mentioned and that are clarified in some more detail in 2.4.1 and 2.4.2, samples should not undergo further processing (i.e. samples should not be boiled, steamed, fried, heated or the like). Extra treatments, like drying, may be performed to prevent the sample from decaying. In particular, for speciation analyses, the potential loss or change of analytes shall be considered carefully though. Finally the sample needs to be homogenised (for example by grinding) to obtain a homogenous test portion. Contamination and uncontrolled loss of liquid, e.g. during operations such as grinding and cutting, shall be avoided to the greatest extent possible.

2.2 Reagents, apparatus and equipment

No reagents and chemicals other than water shall be used in sample preparation. Water with the quality of drinking water may be used for washing some samples, e.g. vegetables. However, depending on the purpose of the analysis and the element to be determined, it may be necessary to use purified water (see 3.1). Apparatuses and equipment should preferably be designated for trace element analysis.

Typical kitchen utensils (e.g. plastic dishes, plastic salad spinner, and coffee grinder) may also be used for sample preparation steps as long as they do not affect both the sample and the elements to be determined.

Any equipment coming into direct contact with the sample shall, if necessary, be cleaned with detergent and hot water. Equipment cleaned with detergent, and containers for storage of homogenised samples, should be treated with diluted nitric acid (approximately 6 % by volume) followed by purified water, if appropriate.

Contamination problems may arise when stainless steel or iron equipment is used for analysis of elements such as chromium, molybdenum, nickel and iron. Special apparatus shall be used in such cases, e.g. titanium or ceramic knives, agate mortar or ball mill for size reduction and homogenisation. Ceramic materials show a high degree of mechanical and chemical stability, but they might have varying compositions (e.g. Al-oxides, Zr-oxides).

Homogenisers shall be chosen carefully as not to cause losses of volatile elements (e.g. by heating up the sample during mixing) or to cause contamination of the elements to be determined or modification of species (e.g. due to warm-up by friction) in the case of speciation analyses.

2.3 Storage

2.3.1 General

Both laboratory samples and test samples shall be stored in such a way that the composition and sample mass does not change as a result of, for instance, drying out, evaporative loss, spoilage or decay. The test sample should be prepared as quickly as possible after arrival of the laboratory sample.

Test samples may be stored in a refrigerator or frozen depending on the nature of the material and the expected storage time. Suitable storage material may be made of glass, plastic containers closed with a screw cap or plastic bags. Dry products may be stored at room temperature.

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2.3.2 Storage for speciation purposes

Some considerations shall be kept in mind when storing samples for speciation purposes. Parameters with a strong influence in speciation analysis are (see Table 1):

- a) Temperature: Storage temperature shall be low enough to prevent microbial activity resulting in reactions, e.g. methylation and biodegradation (see Table 1).
- b) pH: The pH of the media may strongly affect the stability of the inorganic species. Samples intended for species analysis shall not be changed in their acidity for preservation purposes.
- c) Light: Light may cause instability of organometallic compounds by photodegrading. When analysing organometallic compounds storage shall be done in dark or in opaque containers.
- d) Storage time: Generally, storage should be kept as short as possible.

Table 1— Examples of storage conditions for samples intended for speciation purposes

Element (species)	Storage conditions
Hg (Hg(II), methylmercury)	Keep samples at 4 °C or lower in darkness. Dilution shall be done only immediately before the analysis.
Sn (butylated organotin compounds)	PVC containers shall not be used to avoid contamination by organotin compounds. Store samples in borosilicate glass bottles in the dark, at least at 4 °C or lower.
Cr (Cr (III) and Cr (VI))	Only borosilicate or quartz glass should be used for handling and storage. Some glassware may cause contamination with chromium. Keep samples at 4 °C or lower. The pH has different effect on the stability of Cr (III) and Cr (VI).
Se (Se (IV), Se (VI) and methylated compounds)	Avoid the use of poly tetra fluoro ethylene containers [3]. Keep at -20 °C or lower to avoid any change of the inorganic species. Acid media (pH 1,5 to 2) avoid oxidation of Se (IV) to Se (VI).
As (As (III), As (V), and organic As-species)	Keep samples at 4 °C or lower, in darkness. Avoid the use of glassware as this may cause contamination with arsenate.

2.4 Procedures for sample preparation

2.4.1 General

For some trace elements sampling preparation procedures are laid down in EU legislation. Commission Regulation (EC) No 1881/2006 [4] defines the foodstuffs to which the EU maximum levels apply. Further sample preparation methods for official control of the levels of these elements are laid down in Regulation (EC) No 333/2007 [5]. Other specific legislation may also apply to trace elements.

Without prejudice to the specific provisions of EU legislation for official control, in food analysis only the edible part should be investigated and the results should refer to this part. Parts which are usually not intended for consumption should be removed, e.g. outer leaves, shell, skin and bones. Surface contamination like soil should be thoroughly removed by rinsing with drinking water or purified water. For preparation of the test portion a sufficient and representative amount should be available from the edible part of the laboratory sample. Please note that the edible part can differ between countries. The prepared and analysed part of the test portion has to be described in the test report (e.g. potatoes peeled or potatoes washed and unpeeled).

Rinse samples with water before cutting to avoid leaching from cut surfaces. After rinsing with drinking water samples shall be rinsed with purified water. Excess water should be physically removed, e.g. by shaking, using a sieve or a strainer (e.g. for leafy vegetables). Removing excess water by wiping with a soft tissue paper can cause random sample contamination and should not be used for elemental trace analysis. The washing step can be omitted for samples designated as “ready to eat”.

2.4.2 Examples of sample preparation for some foodstuffs

There is a huge variety of foodstuffs on the European market. Preparation practices vary between products as well as between geographical areas. It is therefore not possible to cover every possible preparation procedure in this standard. Examples of preparation procedures for some foodstuffs are given in Table 2. It is advisable to check if there is specific legislation or, e.g., traditions that could give some guidance in their preparation.

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