# Toiduained. Raskemetallide määramine. Määramise tingimused ja üldpõhimõtted.

Foodsuffs - Determination of trace elements - Performance criteria and general considerations



#### **EESTI STANDARDI EESSÕNA**

#### **NATIONAL FOREWORD**

Käesolev Eesti standard EVS-EN 13804:2002 sisaldab Euroopa standardi EN 13804:2002 ingliskeelset teksti.

Käesolev dokument on jõustatud 18.10.2002 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.

Standard on kättesaadav Eesti standardiorganisatsioonist.

This Estonian standard EVS-EN 13804:2002 consists of the English text of the European standard EN 13804:2002.

This document is endorsed on 18.10.2002 with the notification being published in the official publication of the Estonian national standardisation organisation.

The standard is available from Estonian standardisation organisation.

#### Käsitlusala:

This European Standard specifies performance criteria for the selection of methods of analysis of trace elements in foodstuffs. It provides general considerations about the special requirements on sample preparation, apparatus, equipment and reagents for trace elements analysis. In selecting a method of analysis for a specific food matrix, the analyst should give preference to any method which has been developed by the appropriate vertical Technical Committee rather than using a method which has been developed by the horizontal Technical Committee CEN/TC 275/WG 10. However it is the responsibility of the analyst to determine whether an applicable vertical standard has been published.

#### Scope:

This European Standard specifies performance criteria for the selection of methods of analysis of trace elements in foodstuffs. It provides general considerations about the special requirements on sample preparation, apparatus, equipment and reagents for trace elements analysis. In selecting a method of analysis for a specific food matrix, the analyst should give preference to any method which has been developed by the appropriate vertical Technical Committee rather than using a method which has been developed by the horizontal Technical Committee CEN/TC 275/WG 10. However it is the responsibility of the analyst to determine whether an applicable vertical standard has been published.

#### ICS 67.050

**Võtmesõnad:** analysis, chemical analysis, chemical analysis and testin, food technology, metals, methods of analysis, residues, samples, sampling, sampling methods, specification (approval), specifications, specimen preparation, testing, trace element analysis, trace elements

## EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN 13804

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ICS 67,050

#### **English version**

# Foodstuffs - Determination of trace elements - Performance criteria, general considerations and sample preparation

Produits alimentaires - Dosage des éléments traces -Critères de performance, généralités et préparation des échantillons Lebensmittel - Bestimmung von Elementspuren -Leistungskriterien, allgemeine Festlegungen und Probenvorbereitung

This European Standard was approved by CEN on 20 March 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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



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#### **Foreword**

This document EN 13804:2002 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 November 2002, and conflicting national standards shall be withdrawn at the latest by November 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. " O POR LICE OF THE SERVICE OF THE S

#### Introduction

The Working Group CEN/TC275/WG10 – Trace Elements (Heavy Metals) selects and elaborates methods of analysis of trace elements in foodstuffs.

There are many methods of analysis for the determination of trace 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 [9], 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 these performance characteristics are absent or not available, the criteria were established based on the experience and opinions of the experts of CEN/TC275/WG10.

The criteria included in this European Standard have also been used as a 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 equipment, apparatus, reagents, glassware, etc., which are applicable to the selected methods.

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 trace elements in foodstuffs.

It provides general considerations about the special requirements on sample preparation, apparatus, equipment and reagents for trace elements analysis.

In selecting a method of analysis for a specific food matrix, the analyst should give preference to any method which has been developed by the appropriate vertical Technical Committee rather than using a method which has been developed by the horizontal Technical Committee CEN/TC 275/WG 10. However it is the responsibility of the analyst to determine whether an applicable vertical standard has been published.

#### 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).

ISO 5725-1:1994, Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions.

ISO 5725-2:1994, Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories.

#### 3 Terms and definitions

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

#### 3.1

#### method validation

process of acquiring a set of data indicating the accuracy and precision that a method is capable of producing for specified analytes and sample types

#### 3.2

#### precision

closeness of agreement between independent test results obtained under stipulated conditions [ISO 5725-1:1994]

#### 3.3

#### repeatability

precision under repeatability conditions [ISO 5725-1:1994]

#### 3.4

#### repeatability conditions

conditions where independent tests results are obtained with the same method on identical test items, in the same laboratory, by the same operator, using the same equipment within short intervals of time [ISO 5725-1:1994]

#### 3.5

#### reproducibility

precision under reproducibility conditions [ISO 5725-1:1994]

#### 3.6

#### reproducibility conditions

conditions where tests results are obtained with the same method on identical test items in different laboratories with different operators using different equipment [ISO 5725-1:1994]

#### 3.7

#### RSD,

(relative within laboratory standard deviation): precision characteristic which relates to the within laboratory variability of a method. It is the standard deviation, expressed as a percentage of the mean, obtained under repeatability conditions. It is given by the following formula:

$$RSD_{\rm r} = \frac{S_{\rm r}}{r} \times 100\% \tag{1}$$

#### 3.8

#### RSD<sub>R</sub>

(relative between laboratory standard deviation): precision characteristic which relates to the between laboratory variability of a method. It is the standard deviation, expressed as a percentage of the mean, obtained under reproducibility conditions. It is given by the following formula:

$$RSD_{R} = \frac{S_{R}}{x} \times 100\% \tag{2}$$

#### 3.9

#### Ho.

(Horrat index for repeatability): the observed  $RSD_r$  divided by the  $RSD_R$  estimated from the Horwitz equation using the assumption  $r = 0.66 \ R$