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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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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of an identical text or by endorsement, at the latest by November 2002, and conflicting national standards shall be withdrawn at the latest by November 2002.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

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*$_r$**
(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:

$$RSD_r = \frac{s_r}{\bar{x}} \times 100\,\% \qquad (1)$$

3.8
***RSD*$_R$**
(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:

$$RSD_R = \frac{s_R}{\bar{x}} \times 100\,\% \qquad (2)$$

3.9
***Ho*$_r$**
(Horrat index for repeatability): the observed RSD_r divided by the RSD_R estimated from the Horwitz equation using the assumption $r = 0{,}66\ R$

3.10
***Ho*$_R$**
(Horrat index for reproducibility): the observed RSD_R, divided by the RSD_R calculated by the Horwitz equation

3.11
trueness
closeness of agreement between the average value obtained from a large series of test results and an accepted reference value [ISO 5725-1:1994]

3.12
practicability
non-standard characteristic of an analytical procedure. It is dependent of the scope of the method, and is determined by requirements such as sample throughput and costs [9]

3.13
applicability
list of the commodities for which the candidate method can be applied as presented or with minor modifications [9]

3.14
specificity
ability of the method to distinguish between the analyte of interest and other substances present in the sample [9]

3.15
limit of detection
smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty. The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations ($n > 20$) [9]

3.16
limit of quantification
lowest content of the analyte which can be measured with reasonable statistical certainty. If both accuracy and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to six times the standard deviation of the mean of blank determinations ($n > 20$) [9]

3.17
reference material (RM)
material or substance one or more of whose properties are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to the materials [4]

3.18
certified reference material (CRM)
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence [4]

3.19
blank
solution resulting of executing the analytical procedure in all respects apart from the addition of the test portion [4]

3.20
laboratory sample
specific quantity of a product in the condition in which it was received at the laboratory for analysis

3.21
test sample
part taken from the laboratory sample that is representative in composition of the laboratory sample and has been prepared in such a way that the eventual analysis can be carried out with a part of it