

Foodstuffs — Determination of trace elements — Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP- MS) after pressure digestion

ICS 67.050

National foreword

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The UK participation in its preparation was entrusted to Technical Committee AW/-/3, Food analysis - Horizontal methods.

A list of organizations represented on this committee can be obtained on request to its secretary.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 January 2010
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ISBN 978 0 580 61085 1

Amendments/corrigenda issued since publication

Date	Comments

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15763

December 2009

ICS 67.050

English Version

Foodstuffs - Determination of trace elements - Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion

Produits alimentaires - Dosage des éléments traces - Dosage de l'arsenic, du cadmium, du mercure et du plomb par spectrométrie d'émission avec plasma induit par haute fréquence et spectromètre de masse (ICP-MS) après digestion sous pression

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Arsen, Cadmium, Quecksilber und Blei in Lebensmitteln mit induktiv gekoppelter Plasma-Massenspektrometrie (ICP-MS) nach Druckaufschluss

This European Standard was approved by CEN on 7 November 2009.

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Contents

Page

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Principle	4
4 Reagents	4
5 Apparatus and equipment	6
6 Procedure	6
7 Calculation.....	10
8 Analytical quality control	11
9 Limit of quantification	11
10 Precision	11
11 Test report	13
Annex A (informative) Results of the collaborative test.....	14
Bibliography	18

Foreword

This document (EN 15763:2009) 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of an identical text or by endorsement, at the latest by June 2010, and conflicting national standards shall be withdrawn at the latest by June 2010.

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1 Scope

This European Standard specifies a method for the determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS).

The collaborative study included foodstuffs such as carrots, fish homogenate, Mushrooms (CRM), graham flour, a simulated diet E (CRM), scampi, mussel and a Tort-2 CRM having an arsenic mass fraction ranging from 0,06 mg/kg to 21,5 mg/kg dry matter (d. m.), cadmium ranging from 0,03 mg/kg to 28,3 mg/kg d. m., mercury ranging from 0,04 mg/kg to 0,56 mg/kg d. m. and lead from 0,01 mg/kg to 2,4 mg/kg d. m.

2 Normative references

The following referenced documents are indispensable for the application 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 13805, *Foodstuffs — Determination of trace elements — Pressure digestion*

3 Principle

The test solution, obtained by pressure digestion, is nebulised and the aerosol transferred to a high frequency inductively coupled argon plasma. The high temperature of the plasma is used to dry the aerosol and to atomise and ionise the elements. The ions are extracted from the plasma by a set of sampler and skimmer cones and transferred to a mass spectrometer where the ions are separated by their mass/charge ratio and determined by a pulse-count and/or analogue detector.

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

4 Reagents

4.1 General

The concentration of the trace elements in the reagents and water used shall be low enough not to affect the results of the determination. Using a multielemental method of high sensitivity like ICP-MS, the control of the blank levels of water and acid is very important. Generally ultrapure water and acid of high purity, e.g. cleaned by sub boil distillation, are recommended. Special facilities should be used in order to avoid contamination during the steps of preparation and measurements (e.g. use of laminar flow benches or comparable clean room facilities).

4.2 Nitric acid

Mass fraction not less than $w(\text{HNO}_3) = 65\%$, with a density of approximately 1,4 g/ml.

4.3 Element stock solutions

Commercially available single or multielemental standards with a mass concentration of $\rho = 1\,000\text{ mg/l}$ of As, Au, Cd, Hg, Lu, Rh and Pb are recommended. Such standards are available in suitable concentrations from different suppliers. Stock solutions in diluted nitric acid are preferred.

4.4 Diluted mercury stock solution, $\rho(\text{Hg}) = 10\text{ mg/l}$

Pipette 1 ml of Hg stock solution of $\rho(\text{Hg}) = 1\,000\text{ mg/l}$ (4.3) and 1 ml of nitric acid (4.2) in a 100 ml volumetric flask and dilute with water to mark.

4.5 Diluted multi-element stock solution

The concentration levels of the elements in the diluted multi-element stock solution may be chosen in the relation to the type of samples analysed.

EXAMPLE $\rho(\text{As}) = 20\text{ mg/l}$, $\rho(\text{Cd})$, $\rho(\text{Pb}) = 10\text{ mg/l}$. Pipette 2 ml of As, 1 ml of Cd and Pb, respectively of each stock solution into a 100 ml volumetric flask, add 1 ml of nitric acid (4.2), dilute with water to the mark and transfer the solution into a suitable vessel.

4.6 Multi-element calibration solution

According to the example given under 4.5, the multi-element calibration solution contains: $\rho = 100\text{ }\mu\text{g/l}$ As, $\rho = 50\text{ }\mu\text{g/l}$ Cd, Hg, Pb. Pipette 0,5 ml diluted mercury stock solution (4.4) and 0,5 ml of the diluted multi-element stock solution (4.5) to a 100 ml volumetric flask, add 1 ml nitric acid (4.2), dilute with water to the mark and transfer the solution into a suitable vessel (PFA or quartz is recommended).

4.7 Internal standard solution

The internal standard solution contains Rhodium and Lutetium with a mass concentration of $\rho = 1\,000\text{ mg/l}$. Gold is used to stabilise mercury in the solution and reduce memory effects. The internal standard/s should cover the mass range used for determination of the elements. Their concentrations in the test solutions should be negligible.

4.8 Diluted internal standard solution

The concentration of the diluted internal standard solution should be high enough to give sufficient signal intensity. For an internal standard solution of $\rho(\text{Au, Rh, Lu}) = 5\text{ mg/l}$, pipette 0,5 ml of Au, Rh and Lu internal standard solution (4.7) each into a 100 ml flask, add 1 ml of nitric acid (4.2), dilute to volume with water and transfer the solution into a suitable vessel.

4.9 Optimising solution

The optimising solution is used for check and optimising procedures during set up of the ICP-MS. It is used for mass calibration purposes and for adjustment of maximum sensitivity at low rates of oxides and doubly charged ions. The optimising solution should contain elements that cover the whole mass range giving a high rate of oxides and doubly charged ions. The solutions recommended by the manufacturer of the ICP-MS instrument may be used. A solution containing e.g. Y, Rh, Ce and Pb is suitable for those purposes. The concentration of these elements should be chosen in order to achieve a count rate of 10 000 to 100 000.

4.10 Blank solution

The blank solution contains water and the same amount of acid used in the calibration solution.