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Cereals — Determination of cadmium content by graphite furnace atomic absorption spectrometry with diluted nitric acid extraction

Céréales — Détermination de la teneur en cadmium par spectrométrie d'absorption atomique en four graphite et extraction à l'aide d'acide nitrique dilué

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Cereals — Determination of cadmium content by graphite furnace atomic absorption spectrometry with diluted nitric acid extraction

1 Scope

This document specifies a method for the determination of cadmium (Cd) in cereals.

It is applicable to rice, brown rice, wheat and maize by graphite furnace atomic absorption spectrometry (GFAAS) after extraction with diluted nitric acid (HNO_3). The limit of quantification is 0,002 mg/kg; it is approximate and dependent on the sample matrix as well as on the instrument conditions.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Cadmium (Cd) is extracted from the cereals using diluted nitric acid and then determined by graphite furnace atomic absorption spectrometry (GFAAS).

5 Reagents and solution

During the analysis, unless otherwise stated, use only reagents of recognized analytical purity and only water of grade 1 in accordance with ISO 3696.

5.1 Nitric acid (HNO_3), not less than 65 % (mass fraction) of approximately $\rho(HNO_3)$ = 1,4 g/ml.

5.2 Nitric acid solution (0,5 %, volume fraction), mix 0,5 volume parts of HNO_3 (5.1) and 100 ml volume parts of water.

5.3 Nitric acid solution (50 %, volume fraction), mix HNO_3 (5.1) and water in equal volume.

5.4 Palladium nitrate hydrate ($Pd(NO_3)_2$, 99,9 % purity).

5.5 Palladium nitrate ($Pd(NO_3)_2$) solution, c($Pd(NO_3)_2$) = 100 mg/l, dissolve 0,1 g $Pd(NO_3)_2$ (5.4) and dilute to 1 000 ml with HNO_3 (5.2). Other matrix modifiers may also be used if their applicability is proven.

5.6 Cadmium standard solution.

5.6.1 Cadmium stock standard solution, with a cadmium mass concentration of 1 000 mg/l or 500 mg/l.

5.6.2 Cadmium standard solution, stepwise dilute cadmium stock solution (5.6.1) into concentration of 2 µg /l with HNO_3 (5.2).

5.6.3 Cadmium calibration solutions, pipette suitable volumes of cadmium standard solution (5.6.2), e.g. 2 ml, 4 ml, 6 ml, 8 ml and 10 ml, into, for example, a 10 ml volumetric flask (6.7) and dilute to the mark with diluted nitric acid solution (5.2). The concentration of cadmium in the calibration solutions should cover the range of 0,4 µg /l to 2,0 µg/l.

6 Apparatus and equipment

All glassware shall be cleaned several times with water after being soaked overnight with HNO_3 (5.3) and rinsed three times with ultrapure water before use.

6.1 Grinding mill, grinder suitable to obtain the particle sizes of 0,25 mm and 0,40 mm.

6.2 Atomic absorption spectrometer, equipped with graphite furnace, autosampler and background correction capability, such as Zeeman background correction.

6.3 Cadmium hollow cathode lamp, with wavelength 228,8 nm and stable lamp energy.

6.4 Centrifuge, with positions for 10 ml centrifuge tubes and 3 000 r/min of speed.

6.5 Analytical balance, accurate to 1 mg.

6.6 Sieve, with aperture sizes of 0,25 mm and 0,40 mm.

6.7 Plastic centrifuge tube, 10 ml.

7 Procedure

7.1 General

The measurement should be performed as soon as possible after extraction.

7.2 Sampling

A representative sample should be sent to the laboratory. It should not have been damaged or changed during transport and storage.

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 24333.