SLOVENSKI STANDARD
SIST EN ISO 11212-4:1998

01-december-1998

Škrob in škrobni derivati - Delež težkih kovin - 4. del: Določevanje kadmija po metodi atomske absorpcijske spektrometrije z elektrotermijsko atomizacijo (ISO 11212-4:1997)

Starch and derived products - Heavy metals content - Part 4: Determination of cadmium content by atomic absorption spectrometry with electrothermal atomization (ISO 11212-4:1997)

Stärke und Stärkederivate - Schwermetallgehalt - Bestimmung des Cadmiumgehaltes durch Atomabsorptionsspektrometrie mit elektrothermischer Atomisierung (ISO/DIS 11212-4:1996)

Amidons, fécules et produits dérivés - Teneur en métaux lourds - Partie 4: Détermination de la teneur en cadmium par spectrométrie d'absorption atomique avec atomisation électrothermique (ISO 11212-4:1997)

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Starch and derived products - Heavy metals content - Part 4: Determination of cadmium content by atomic absorption spectrometry with electrothermal atomization (ISO 11212-4:1997)

Amidons, fécules et produits dérivés - Teneur en métaux lourds - Partie 4: Détermination de la teneur en cadmium par spectrométrie d'absorption atomique avec atomisation électrothermique (ISO 11212-4:1997)

Stärke und Stärkederivate - Schwermetallgehalt - Bestimmung des Cadmiumgehaltes durch Atomabsorptionsspektrometrie mit elektrothermischer Atomisierung (ISO/DIS 11212-4:1996)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard ISO 11212-4:1997 has been prepared by Technical Committee ISO/TC 93 "Starch (including derivatives and by-products)" in collaboration with CEN/CS.

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of an identical text or by endorsement, at the latest by September 1997, and conflicting national standards shall be withdrawn at the latest by September 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 11212-4:1997 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 11212-4

First edition
1997-03-15

Starch and derived products — Heavy metals content —

Part 4:
Determination of cadmium content by atomic absorption spectrometry with electrothermal atomization

Amidons, fécules et produits dérivés — Teneur en métaux lourds —

Partie 4: Détermination de la teneur en cadmium par spectrométrie d'absorption atomique avec atomisation électrothermique

Reference number
ISO 11212-4:1997(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11212-4 was prepared by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*.

ISO 11212 consists of the following parts, under the general title *Starch and derived products — Heavy metals content*:

— *Part 1: Determination of arsenic content by atomic absorption spectrometry*

— *Part 2: Determination of mercury content by atomic absorption spectrometry*

— *Part 3: Determination of lead content by atomic absorption spectrometry with electrothermal atomization*

— *Part 4: Determination of cadmium content by atomic absorption spectrometry with electrothermal atomization*

Annex A of this part of ISO 11212 is for information only.

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Starch and derived products — Heavy metals content —

Part 4:
Determination of cadmium content by atomic absorption spectrometry with electrothermal atomization

1 Scope

This part of ISO 11212 specifies a method for the determination of the cadmium content of starch, including derivatives and by-products, by atomic absorption spectrometry with electrothermal atomization.

The number of parameters for the procedure involved in the electrothermal atomization is far larger than in flame atomization; it is thus impossible to propose a comprehensive method likely to ensure the attainment of satisfactory results on all types of apparatus currently available. Each analyst should therefore optimize the conditions of use of his/her own apparatus on the basis of general or particular instructions.

2 Definition

For the purposes of this part of ISO 11212, the following definition applies.

2.1 cadmium content: Quantity of cadmium determined in accordance with the conditions specified in this method and expressed as cadmium (Cd), in micrograms per kilogram of the product as received.

3 Principle

Wet digestion of the organic matrix. Injection of an aliquot portion of digested sample, in the presence of a matrix modifier, into the furnace of an electrothermal atomization atomic absorption spectrometer.

Measurement of the absorbance at a wavelength of 228,8 nm.

Determination of the concentration of cadmium in the sample by means of a calibration curve.

4 Reagents

Use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

4.1 Nitric acid (ρ_{20} = 1,38 g/ml).

4.2 Hydrogen peroxide, 30 % (*V/V*) solution.

4.3 Matrix modifier, consisting of a 1 g/l solution of palladium nitrate.

4.4 Cadmium standard solution, 1 g/l.

Standard solutions are commercially available at this concentration. These solutions may also be prepared by weighing and dissolving the salt or metal of known purity.

4.5 Calibration solutions

Before each series of measurements, prepare from the standard cadmium solution (4.4) at least five calibration solutions covering the range of concentrations to be determined. 100 ml of each calibration solution shall contain 7,5 ml of nitric acid (4.1) and 20 ml of the matrix modifier solution (4.3) if the latter is not distributed by the automatic injection device.

5 Apparatus

All the glassware used shall be previously washed by means of suitable products (such as nitric acid) and rinsed with distilled water to eliminate any trace of cadmium.

Use ordinary laboratory apparatus and, in particular, the following.

5.1 Digestion apparatus (see figure 1), made of borosilicate glass and consisting of three elements terminating with conical ground joints (5.1.1 to 5.1.3).

5.1.1 Soxhlet extraction tube, of capacity 200 ml, equipped with a stopcock and a lateral tube connected directly to the flask (5.1.3).

5.1.2 Cooling apparatus, 35 cm long, connected to the top of the Soxhlet extraction tube (5.1.1).

5.1.3 Round-bottom flask, of capacity 250 ml, connected to the lower part of the Soxhlet extraction tube (5.1.1).

When the stopcock is open, the device is under reflux; when it is closed, the Soxhlet extraction tube (5.1.1) retains the condensed water and acid vapours.

5.2 Atomic absorption spectrometer, consisting of five elements (5.2.1 to 5.2.5).

5.2.1 High-resolution monochromator, allowing a 0,2 nm bandwidth slit.

5.2.2 Correcting device for non-specific absorption.

5.2.3 Measuring and photoelectric reception device, with a response time not exceeding about 10 ms.

5.2.4 Detector and signal processing system, allowing recording of the maximum and/or integrated absorbance signal.

5.2.5 Cadmium discharge lamp or **cadmium hollow cathode lamp.**

5.3 Electrothermic atomizer

The most widely used atomizer, for which the general conditions of use are suggested, is a graphite tubular furnace placed in the optical axis of the spectrometer, heated by the Joule effect. The furnace shall be maintained in an inert atmosphere to avoid its destruction by oxidation when heated at a high temperature, and shall be equipped with an automatic injection device which is necessary to obtain good repeatability and to reduce the risk of contamination.

5.4 Pyrocoated graphite tube, with Lvov platform.

5.5 Pipettes and **micropipettes**, of suitable capacity.

5.6 Analytical balance.

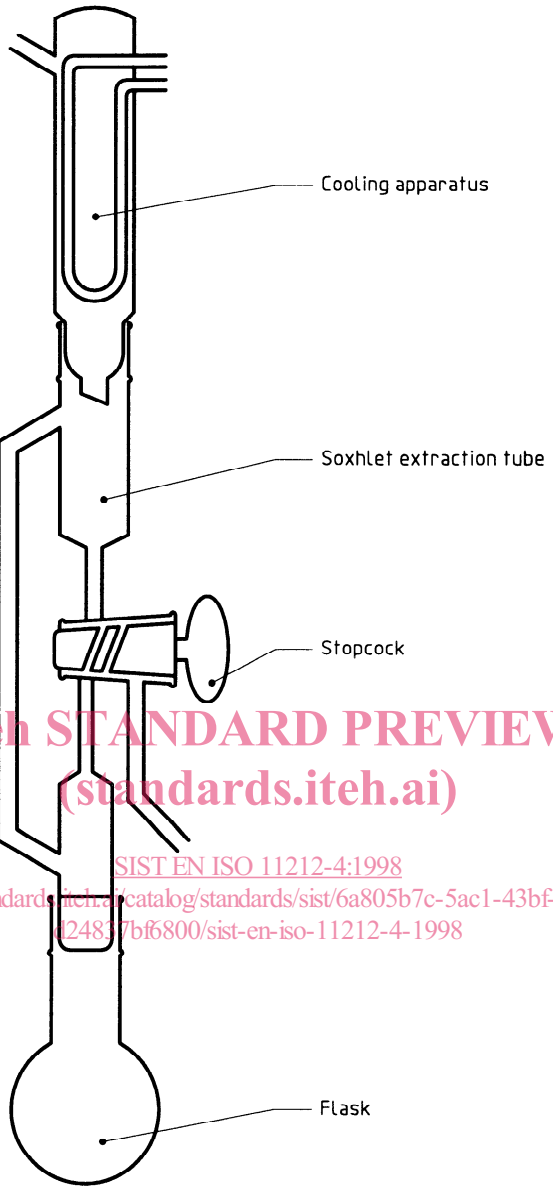

Figure 1 — Digestion apparatus

6 Procedure

6.1 Preparation of test sample

Thoroughly homogenize the sample.

6.2 Digestion

Use the digestion apparatus described in 5.1.

Weigh, to the nearest 1 mg, about 5 g of the test sample into the flask (5.1.3). Add 27,5 ml of nitric acid (4.1) and 1 ml of hydrogen peroxide (4.2). Distil under reflux for 4 h leaving the stopcock open. Turn the stopcock off, continue heating and distil until about 20 ml – 1 ml of liquid are recovered in the extraction tube (5.1.1). Stop heating