SLOVENSKI STANDARD
SIST EN ISO 11212-2:1998

01-november-1998

Škrob in derivati škroba - Delež težkih kovin - 2. del: Določevanje živega srebra z atomsko absorpcijsko spektrometrijo (ISO 11212-2:1997)

Starch and derived products - Heavy metals content - Part 2: Determination of mercury content by atomic absorption spectrometry (ISO 11212-2:1997)

Stärke und Stärkederivate - Schwermetallgehalt - Teil 2: Bestimmung des Quecksilbergehaltes durch Atomabsorptionsspektrometrie (ISO 11212-2:1997)

Amidons, fécules et produits dérivés - Teneur en métaux lourds - Partie 2: Détermination de la teneur en mercure par spectrométrie d'absorption atomique (ISO 11212-2:1997)

Ta slovenski standard je istoveten z: **EN ISO 11212-2:1997**

ICS:

67.180.20	Škrob in izdelki iz njega	Starch and derived products

SIST EN ISO 11212-2:1998 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 11212-2

March 1997

ICS 67.180

Descriptors: see ISO document

English version

Starch and derived products - Heavy metals content - Part 2: Determination of mercury content by atomic absorption spectrometry (ISO 11212-2:1997)

Amidons, fécules et produits dérivés - Teneur en métaux lourds - Partie 2: Détermination de la teneur en mercure par spectrométrie d'absorption atomique (ISO 11212-2:1997)

Stärke und Stärkederivate - Schwermetallgehalt - Teil 2: Bestimmung des Quecksilbergehaltes durch Atomabsorptionsspektrometrie (ISO 11212-2:1997)

This European Standard was approved by CEN on 1997-02-28. 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 Central Secretariat or to any CEN member.

The European Standards exist 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 Central Secretariat has the same status as the official versions.

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

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 11212-2:1997 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 11212-2:1997 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 11212-2

First edition
1997-03-15

Starch and derived products — Heavy metals content —

Part 2:
Determination of mercury content by atomic absorption spectrometry

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

Partie 2: Détermination de la teneur en mercure par spectrométrie d'absorption atomique

Reference number
ISO 11212-2:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 11212-2 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.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

Starch and derived products — Heavy metals content —

Part 2:
Determination of mercury content by atomic absorption spectrometry

1 Scope

This part of ISO 11212 specifies a method for the determination of the mercury content of starch, including derivatives and by-products, by atomic absorption spectrometry with cold-vapour generation.

The cold-vapour generators currently available use very different techniques; it is thus impossible to propose a comprehensive method likely to ensure the attainment of satisfactory results on all types of apparatus. 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 mercury content: Quantity of mercury determined in accordance with the conditions specified in this method and expressed as mercury (Hg), in micrograms per kilogram of the product as received.

3 Principle

Wet digestion of the organic matrix. Reduction of mercury (Hg^{2+}) to metallic mercury by hydrogen resulting from the action of sodium borohydride (or tin(II) chloride) on hydrochloric acid. Entrainment of the mercury vapour by a flow of gas and determination of monoatomic mercury vapour by atomic absorption spectrometry in a quartz cell.

Measurement of the absorbance at a wavelength of 253,7 nm.

Determination of the concentration of mercury 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 Sodium borohydride solution

Prepare a solution at the concentration recommended in the instructions for use of the cold-vapour generator (5.3).

4.4 Tin(II) chloride solution

Prepare a solution at the concentration recommended in the instructions for use of the cold-vapour generator (5.3).

4.5 Hydrochloric acid solution

Prepare a solution at the concentration recommended in the instructions for use of the cold-vapour generator (5.3).

4.6 Mercury standard solution, 1 g/l.

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

4.7 Calibration solutions

Before each series of measurements, prepare from the standard mercury solution (4.6) 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).

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 mercury.

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 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 Mercury discharge lamp or **mercury hollow cathode lamp.**

5.3 Generator of cold mercury vapour, allowing the generation of cold mercury vapour as well as its transport to a measuring cell whose wavelength is adapted to the spectrometer, and equipped with an automatic sampling device which is necessary to obtain good repeatability and to reduce the risk of contamination.

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

5.5 Analytical balance.

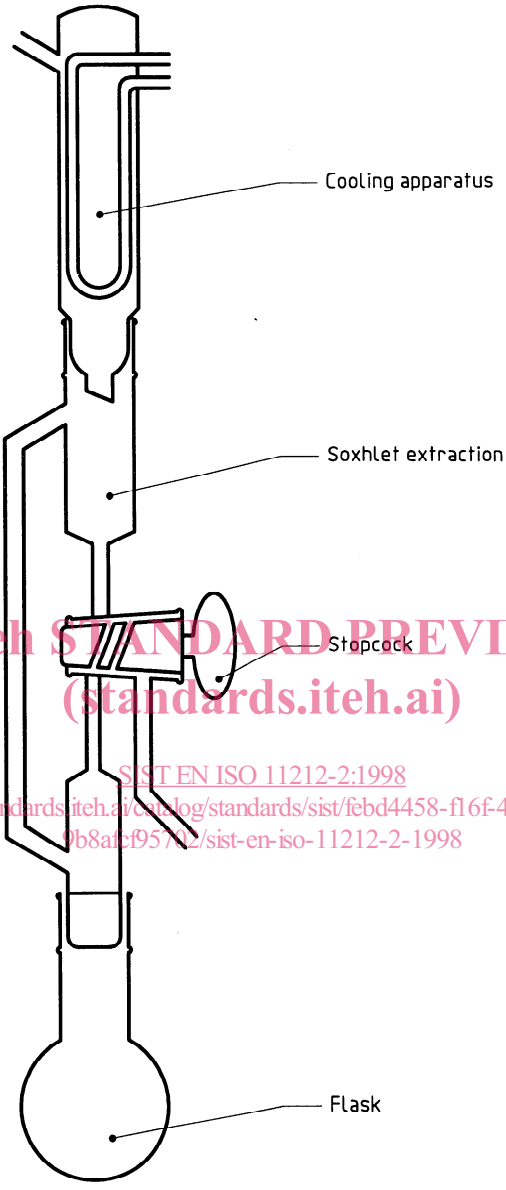

Figure 1 — Digestion apparatus

6 Procedure

WARNING — Mercury is highly toxic and is a very volatile element. It is thus necessary to verify the tightness of the ground joints and of the tap and to avoid any overheating at digestion in order to prevent any loss of mercury vapour.

6.1 Preparation of test sample

Thoroughly homogenize the sample.