

**SRI LANKA STANDARD 1570: PART 1: 2017**  
**(ISO 11212-1: 1997)**  
**UDC 664.2**

**METHODS OF TEST FOR**  
**STARCH AND DERIVED PRODUCTS**  
**HEAVY METALS CONTENT**  
**PART 1 : DETERMINATION OF ARSENIC CONTENT**  
**BY ATOMIC ABSORPTION SPECTROMETRY**

**SRI LANKA STANDARDS INSTITUTION**



**Sri Lanka Standard**  
**METHODS OF TEST FOR STARCH AND DERIVED PRODUCTS - HEAVY METALS**  
**CONTENT**  
**PART 1 : DETERMINATION OF ARSENIC CONTENT BY ATOMIC**  
**ABSORPTION SPECTROMETRY**

**SLS 1570: Part 1: 2017**  
**(ISO 11212-1: 1997)**

**Gr. C**

*Copyright Reserved*  
**SRI LANKA STANDARDS INSTITUTION**  
**17, Victoria Place**  
**Elvitigala Mawatha**  
**Colombo - 08**  
**Sri Lanka**

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This Standard does not purport to include all the necessary provisions of a contract.

© SLSI 2017

All right reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the SLSI.

**Sri Lanka Standard**  
**METHODS OF TEST FOR STARCH AND DERIVED PRODUCTS - HEAVY**  
**METALS CONTENT**  
**PART 1 : DETERMINATION OF ARSENIC CONTENT BY ATOMIC**  
**ABSORPTION SPECTROMETRY**

**NATIONAL FOREWORD**

This Sri Lanka Standard was approved by the Sectoral Committee on Food Products and was authorized for adoption and publication as a Sri Lanka Standard by the council of the Sri Lanka Standards Institution on 2017-07-21.

The Standard prescribes the test methods for determining whether the material conforms to the requirements of the relevant individual Standards and thus from a necessary adjunct to series of Sri Lanka Standard Methods of tests for starch and derived products - heavy metals content. However, keeping in view the experience gained during the years and various international standards brought out by the International Organization for Standardization (ISO) on the subject of testing starch and derived products - heavy metals content it was decided to adopt it with a view to updating the existing methods of test.

In order to accommodate large number of test methods within the scope of one Standard, this Standard is published in several parts.

This part of the Standard is identical with **ISO 11212-1: 1997** Starch and derived products - Heavy metals content Part 1: Determination of arsenic content by Atomic Absorption Spectrometry.

**TERMINOLOGY AND CONVENTIONS**

The text of the International Standard has been accepted as a suitable for publication, without deviation, as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standard. Attention is therefore drawn to the following:

- a) Wherever the words “International Standard” appear referring to this Standard, they should be interpreted as “Sri Lanka Standard”.
- b) The comma has been used throughout as a decimal marker. In Sri Lanka Standards it is the current practice to use the full point at the base line as the decimal marker.
- c) Wherever page numbers are quoted, they are ISO page numbers.

.....



INTERNATIONAL  
STANDARD

**ISO**  
**11212-1**

First edition  
1997-03-15

---

---

**Starch and derived products — Heavy  
metals content —**

**Part 1:**

**Determination of arsenic content by atomic  
absorption spectrometry**

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

*Partie 1: Détermination de la teneur en arsenic par spectrométrie  
d'absorption atomique*



Reference number  
ISO 11212-1:1997(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 11212-1 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.

© ISO 1997

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

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 1:

### Determination of arsenic content by atomic absorption spectrometry

#### 1 Scope

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

The hydride 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 arsenic content:** Quantity of arsenic determined in accordance with the conditions specified in this method and expressed as arsenic (As), in micrograms per kilogram of the product as received.

#### 3 Principle

Wet digestion of the organic matrix. Reduction of arsenic ( $\text{As}^{3+}$ ) to arsenic hydride by hydrogen resulting from the action of sodium borohydride on hydrochloric acid. Entrainment of the hydride form by a flow of gas and determination by atomic absorption spectrometry in a heated quartz cell.

Measurement of the absorbance at a wavelength of 193,7 nm.

Determination of the concentration of arsenic 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** ( $\rho_{20} = 1,38$  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 hydride generator (5.3).

### 4.4 Hydrochloric acid solution

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

### 4.5 Arsenic 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.

### 4.6 Calibration solutions

Before each series of measurements, prepare from the standard arsenic solution (4.5) 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 arsenic.

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 Arsenic discharge lamp or arsenic hollow cathode lamp.**

**5.3 Hydride generator**, allowing the generation of hydrides as well as their transport to a heated 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.**

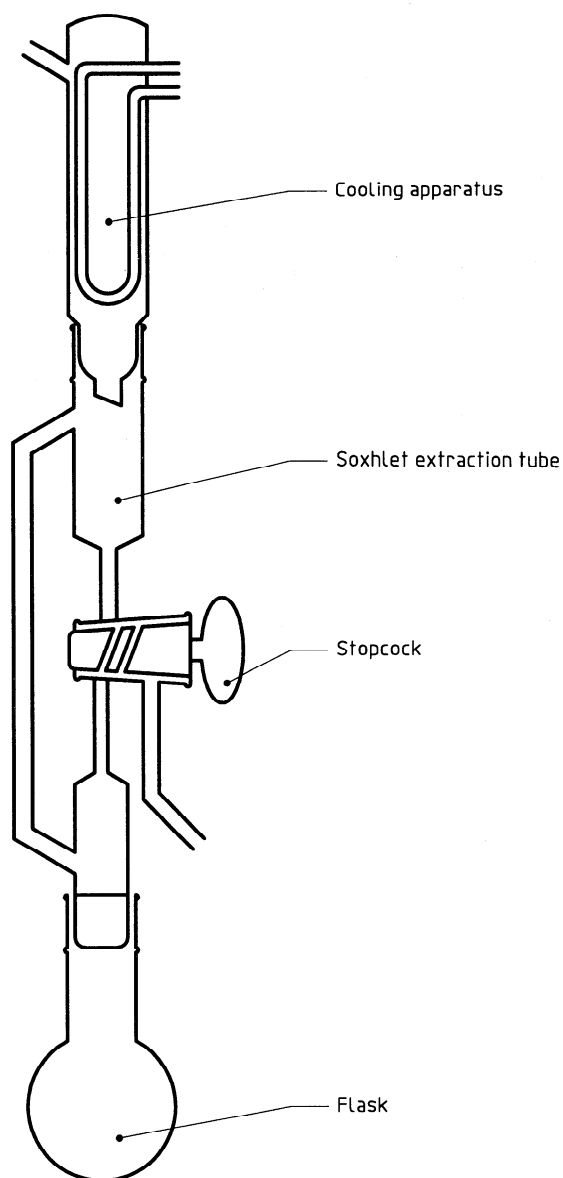


Figure 1 — Digestion apparatus

## 6 Procedure

### 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  $20 \text{ ml} \pm 1 \text{ ml}$  of liquid are recovered in the extraction tube (5.1.1). Stop heating and allow the flask to cool. Separate the flask from the extraction tube. Add 20 ml of water to the digested residue in the flask, bring to the boil for a few minutes, stop heating and allow to cool. Transfer the solution to a 100 ml volumetric flask, dilute to the mark with distilled water and stir.

### 6.3 Blank test

Perform digestion under the same conditions as in 6.2, replacing the test portion by 5 ml of water.

### 6.4 Determination of the calibration curve

Carry out the analysis of the diluted calibration solutions (4.6) with reference to the instructions for use of the hydride generator (5.3), by adding the recommended quantities of hydrochloric acid solution (4.4) and sodium borohydride solution (4.3). Measure the absorbance of each calibration solution at a wavelength of 193,7 nm using the spectrometer (5.2).

Draw the calibration curve by plotting the arsenic concentrations of the calibration solutions, expressed in micrograms per litre, as the abscissa against the corresponding values of the signal, read either in maximum absorbance or in integrated absorbance, as the ordinate. The calibration curve shall be periodically checked depending on the length of the series of analyses.

### 6.5 Determination

Measure the absorbance of the test samples under the same conditions as the calibration solutions and compare the results with the previously plotted calibration curve.

## 7 Expression of results

With reference to the calibration curve, determine the concentrations corresponding to the signals of the test portion and the blank. The arsenic concentration of the sample,  $w$ , expressed in micrograms per kilogram of the product as received, is given by the equation:

$$w = \frac{(\rho_1 - \rho_0) \times 100}{m}$$

where

$\rho_1$  is the numerical value of the arsenic concentration, in micrograms per litre, of the test solution (6.2) read from the calibration curve (6.4);

$\rho_0$  is the numerical value of the arsenic concentration, in micrograms per litre, of the blank test solution (6.3) read from the calibration curve (6.4);

$m$  is the numerical value of the mass, in grams, of the test portion (6.2).

NOTE — When strictly following this method, the quantification limit can reach 20 µg/kg.

## 8 Precision

Details of an interlaboratory test on the precision of the method are summarized in annex A. The values derived from the interlaboratory test may not be applicable to analyte concentration ranges and matrices other than those given in annex A.

### 8.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit  $r$  deduced from table A.1 in more than 5 % of cases.

## 8.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will not exceed the reproducibility value  $R$  deduced from table A.1 in more than 5 % of cases.

## 9 Test report

The test report shall specify:

- the method in accordance with which sampling was carried out, if known;
- the method used;
- the test result(s) obtained; and
- if the repeatability has been checked, the final quoted result obtained.

It shall also mention all operating details not specified in this part of ISO 11212, or regarded as optional, together with details of any incidents which may have influenced the test result(s).

The test report shall include all information necessary for the complete identification of the sample.

## Annex A (informative)

### Results of interlaboratory test

An interlaboratory test at the international level was carried out by 12 laboratories in 1993. The statistical results indicated in table A.1 were determined in accordance with ISO 5725<sup>1)</sup>.

**Table A.1 — Interlaboratory test on corn starch**

Parameter	Sample <sup>1)</sup>		
	LC	HC	VHC
No. of laboratories retained after eliminating outliers	9	9	8
No. of outliers (laboratories)	2	2	3
No. of accepted results	34	36	32
Mean arsenic content (µg/kg)	47,1	117,3	189,3
Repeatability standard deviation, $s_r$ (µg/kg)	10,8	17,9	23,1
Repeatability limit, $r = 2,8 \times s_r$ (µg/kg)	30,5	50,5	65,5
Reproducibility standard deviation, $s_R$ (µg/kg)	17,5	26,9	44,2
Reproducibility limit, $R = 2,8 \times s_R$ (µg/kg)	49,5	76,2	125,2
1) Corn starch with LC: low content HC: high content VHC: very high content			

1) ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn), was used to obtain the precision data.

This page intentionally left blank

This page intentionally left blank



This page intentionally left blank

---

---

**ICS 67.180.20**

**Descriptors:** starches, food starch, chemical analysis, determination of content, heavy metals, arsenic, atomic absorption spectrometric method.

Price based on 6 pages

---

---

## **SLS CERTIFICATION MARK**

*The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI which includes surveillance inspection of the factory, testing of both factory and market samples.*

*Further particulars of the terms and conditions of the permit may be obtained from the Sri Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.*



## SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the National Standards Organization of Sri Lanka established under the Sri Lanka Standards Institution Act No. 6 of 1984 which repealed and replaced the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Science, Technology and Research.

The Principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote Standardization and quality control by educational, consultancy and research and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and Administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in return are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution Endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of Standardization as are of special interest to Sri Lanka.