SLOVENSKI STANDARD

SIST ISO 5498

prva izdaja
maj 1995

Kmetijski pridelki in živilski proizvodi - Določanje vsebnosti surove vlaknine - Splošna metoda (prevzet standard ISO 5498:1981 z metodo platnice)

Agricultural food products - Determination of crude fibre content - General method

Produits agricoles alimentaires - Détermination de l indice d insoluble dit «cellulosique» - Méthode générale

Deskriptorji: kmetijski proizvodi, preskus, določanje, indeksi (razmerja), netopne snovi, ekstrakcijska analiza

ICS 67.040

Referenčna številka
SIST ISO 5498:1995 (en)

Nadaljevanje na straneh od II do III in 1 do 8

UVOD

Standard SIST ISO 5498, Kmetijski pridelki in živilski proizvodi - Določanje vsebnosti surove vlaknine - Splošna metoda, prva izdaja, 1995, ima status slovenskega standarda in je z metodo platnice prevzet mednarodni standard ISO 5498, Agricultural food products - Determination of crude fibre content - General method, first edition, 1981-04-15.

PREDGOVOR

Mednarodni standard ISO 5498:1981 je pripravil tehnični odbor Mednarodne organizacije za standardizacijo ISO/TC 34 Kmetijski pridelki in živilski proizvodi.

Odločitev za prevzem tega standarda po metodi platnice je sprejela delovna skupina WG 10 Analitika krme v okviru tehničnega odbora USM/TC KŽP Kmetijski pridelki in živilski proizvodi.

Ta slovenski standard je dne 1995-05-08 odobril direktor USM.

ZVEZA S STANDARDI

S prevzemom tega mednarodnega standarda veljajo naslednje zveze:

SIST ISO 6651:1995 (en)	Krma - Določanje vsebnosti aflatoksina B_1
SIST ISO 6654:1995 (en)	Krma - Določanje vsebnosti sečnine
SIST ISO 6866:1995 (en)	Krma - Določanje vsebnosti prostega in skupnega gosipola
SIST ISO 6870:1995 (en)	Krma - Določanje vsebnosti zearalenona
SIST ISO 5983:1995 (en)	Krma - Določanje vsebnosti dušika in izračun vsebnosti surovih beljakovin
SIST ISO 5984:1995 (en)	Krma - Določanje surovega pepela
SIST ISO 5985:1995 (en)	Krma - Določanje pepela, netopnega v klorovodikovi kislini
SIST ISO 6490-1:1995 (en)	Krma - Določanje vsebnosti kalcija - 1. del: Titrimetrična metoda
SIST ISO 6490-2:1995 (en)	Krma - Določanje vsebnosti kalcija - 2. del: Metoda atomske absorpcijske spektrometrije
SIST ISO 6491:1995 (en)	Krma - Določanje vsebnosti skupnega fosforja - Spektrofotometrična metoda
SIST ISO 6495:1995 (en)	Krma - Določanje vsebnosti v vodi topnih kloridov
SIST ISO 6496:1995 (en)	Krma - Določanje vsebnosti vlage
SIST ISO 5506:1995 (en)	Sojini proizvodi - Določanje ureazne aktivnosti
SIST ISO 6541:1995 (en)	Kmetijski pridelki in živilski proizvodi - Določanje vsebnosti surove vlaknine - Modificirana Scharrerjeva metoda

OSNOVA ZA IZDAJO STANDARDA

- Prevzem standarda ISO 5498:1981

OPOMBI

- Povsod, kjer se v besedilu standarda uporablja izraz mednarodni standard , to pomeni v SIST ISO 5498:1995 slovenski standard .

- Uvod in predgovor nista sestavni del standarda.

Po mnenju Ministrstva za informiranje Republike Slovenije z dne 18. februarja 1992, štev. 23/96-92, spada ta publikacija med proizvode informativne narave iz 13. točke tarifne številke 3, za katere se plačuje 5-odstotni prometni davek.

International Standard 5498

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Agricultural food products — Determination of crude fibre content — General method

Produits agricoles alimentaires — Détermination de l'indice d'insoluble dit «cellulosique» — Méthode générale

First edition — 1981-04-15

UDC 664 : 543.868 **Ref. No. ISO 5498-1981 (E)**

Descriptors : agricultural products, tests, determination, indexes (ratios), insoluble matter, extraction analysis.

Price based on 8 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5498 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in September 1979.

It has been approved by the member bodies of the following countries :

Australia	France	Netherlands
Austria	Germany, F.R.	Poland
Brazil	Hungary	Portugal
Bulgaria	India	Romania
Canada	Israel	South Africa, Rep. of
Chile	Kenya	Spain
Cyprus	Korea, Rep. of	Thailand
Czechoslovakia	Libyan Arab Jamahiriya	Turkey
Egypt, Arab Rep. of	Malaysia	United Kingdom
Ethiopia	Mexico	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

USA

Printed in Switzerland

Agricultural food products — Determination of crude fibre content — General method

0 Introduction

There are numerous methods for the determination of the crude fibre content of agricultural food products and, for a given method, numerous variants are used according to the products analysed, or, for the same product, from laboratory to laboratory.

This International Standard therefore fulfils a need for harmonization. It specifies a method of general application based on the Weende method, which is the most commonly used, and basically involves acid treatment followed by alkaline treatment.

Studies carried out have demonstrated the need to specify precisely the conditions for the acid and alkaline treatments and to describe the various procedures used for separating the insoluble matter. These procedures are described in annex B.

1 Scope

This International Standard specifies a conventional method for the determination of the crude fibre content of agricultural food products.

2 Field of application

The method is intended for general application; however, it may be necessary, in certain individual cases, to choose a more appropriate method, particularly in the case of yeasts and products containing less than 1 % of crude fibre for which the method described in ISO 6541 shall be used.

3 Reference

ISO 6541, *Agricultural food products — Determination of crude fibre content — Modified Scharrer method.*

4 Definition

crude fibre content : Conventionally, the whole of the substances which are insoluble and combustible under the operating conditions described in this International Standard.

The crude fibre content is expressed as a percentage by mass, referred either to the product as received or to the dry matter content of the product.

NOTE — In French, the term "indice d'insoluble dit cellulosique" has been adopted for "crude fibre" rather than the alternative "cellulose brute".

5 Principle

After any necessary grinding and defatting, boiling with sulphuric acid solution of standard concentration, and separation and washing of the insoluble residue.

Boiling this residue with sodium hydroxide solution of standard concentration, then separation, washing, drying and weighing of the insoluble residue, and determination of the loss in mass on incineration.

6 Reagents and materials

All reagents shall be of recognized analytical quality and the water used shall be distilled water or water of at least equivalent purity.

6.1 Sulphuric acid, standard volumetric solution, $c(1/2\ H_2SO_4)$ = 0,255 ± 0,005 mol/l (corresponding to 12,5 g of sulphuric acid per litre of solution).

6.2 Sodium hydroxide, standard volumetric solution, $c(NaOH)$ = 0,313 ± 0,005 mol/l (corresponding to 12,5 g of sodium hydroxide per litre of solution).

This solution shall be as free as possible from carbonates.

6.3 Acetone, or 95 % (V/V) **ethanol,** or **methanol,** or **propan-2-ol.**

6.4 Extraction solvent :

Technical grade *n*-hexane, or light petroleum (having a boiling range between 40 and 60 °C), or diethyl ether, or another solvent or mixture of solvents more suitable for the extraction of fatty substances from certain products to be analysed.

6.5 Hydrochloric acid, 0,5 mol/l solution (in the case of products rich in carbonates). (See 11.1.)

6.6 Filter aid (in cases where the separation techniques described in clauses B.2 or B.3 of annex B are used).

6.7 Anti-foam agent, if necessary, known to have no effect on the results.

6.8 Anti-bumping agent, if necessary, resistant to attack under the test conditions or known to have no effect on the results.

7 Apparatus

Usual laboratory apparatus, and in particular

7.1 Grinding device, easy to clean, suited to the nature of the product and allowing grinding of the product without causing undue heating or significant change in the moisture content.

7.2 Sieve, of metal wire cloth, aperture size 1 mm, complying with the requirements of ISO 3310/1.

7.3 Drying oven, capable of being controlled at 130 ± 2 °C.

7.4 Wide-mouthed vessel, provided with a condenser, for example a flask having a minimum capacity of 600 ml fitted with a reflux condenser, or a beaker without spout, of capacity 600 ml, covered by a round-bottom flask of 500 ml capacity containing 450 ml of cold water.

7.5 Heating device, for example an electrically heated hot-plate fitted with a magnetic stirrer, capable of maintaining 200 ml of reagents 6.1 and 6.2 at a gentle boil.

7.6 Incineration dish, of capacity 25 to 50 ml, resistant to attack under the test conditions, or a **filter crucible**, suitable for separation and incineration of the residue.

7.7 Muffle furnace, provided with air circulation and temperature control, suitable for carrying out incineration at 550 ± 25 °C.

7.8 Desiccator, containing an efficient desiccant.

7.9 Separating device.

Various types of separating devices are described in annex B.

7.10 Analytical balance.

8 Sampling

Refer to the International Standard appropriate to the product concerned.

9 Procedure

9.1 Preparation of test sample

9.1.1 Preliminary drying

In the case of products having moisture contents too high for them to be mixed or ground as received, carry out a preliminary drying of the product at an appropriate temperature. In this case, weigh the product before the preliminary drying and again just before preparation of the test sample (9.1.2 or 9.1.3).

9.1.2 Products not requiring grinding

9.1.2.1 Products that pass through the sieve (7.2) without leaving a residue do not need to be ground before the determination.

Mix well before taking the test portion.

9.1.2.2 If the results are to be expressed relative to the dry matter content, determine beforehand the dry matter content of the test sample (9.1.2.1) by an appropriate method.

9.1.3 Products requiring grinding

Products that do not pass through the test sieve (7.2) without leaving a residue shall be ground.

9.1.3.1 If the results are to be expressed relative to the product as received, determine beforehand the dry matter content of the sample by an appropriate method.

9.1.3.2 Grind the laboratory sample in the grinding device (7.1) so that the product passes through the sieve (7.2) without leaving a residue.

9.1.3.3 Determine the dry matter content of the test sample (9.1.3.2) by an appropriate method.

9.2 Test portion

NOTE — If it is necessary to remove fatty matter (see 9.3.1) and the technique described in A.1.3 of annex A is to be used, the extraction shall be carried out before the test portion is taken.

Weigh, to the nearest 1 mg, about 3 g of the test sample (except in special cases) which has been prepared as described in 9.1, and which is presumed to contain more than 1 % of crude fibre.

9.3 Determination

9.3.1 Extraction of fatty substances (see annex A)

If the fatty matter content is less than 1 %, extraction of fatty matter is unnecessary.

Extraction is not absolutely essential, but is, nevertheless, recommended if the fatty matter content is between 1 and 10 %.

If the fatty matter content is more than 10 %, prior extraction is essential.

For products containing fatty substances which cannot be removed directly, the extraction shall be carried out after the acid treatment (see A.1.5 of annex A).

9.3.2 Acid treatment

9.3.2.1 Transfer the test portion, which may have had the fat and oils (see 9.3.1) and carbonates (see 11.1) removed, into the vessel (7.4). Add, if appropriate, the prescribed quantity of filter aid (6.6) (in the case of the variants described in clauses B.2 and B.3 of annex B) and, if necessary, the anti-foam agent (6.7) and the anti-bumping agent (6.8).

Measure 200 ml of the sulphuric acid solution (6.1) at room temperature, bring it to a temperature of 95 to 100 °C (see 11.2) and add it to the contents of the vessel (7.4).

Fit the condenser. Bring rapidly to the boil (in about 2 min) using the heating device (7.5) and continue boiling gently for 30 ± 1 min. Swirl the vessel from time to time so that any particles adhering to the interior wall are returned to the solution.

9.3.2.2 After the specified boiling period, add about 50 ml of cold water and separate rapidly the insoluble residue using the separating device (7.9) chosen. Wash the vessel (7.4) with 50 ml portions of hot water (temperature 95 to 100 °C) and pour the washings over the insoluble residue remaining in the separating device.

Repeat the washing of the insoluble residue until the filtrate is substantially neutral to litmus paper. The separation and washing of the insoluble residue shall be completed in less than 30 min.

9.3.3 Alkaline treatment

9.3.3.1 Return the washed insoluble residue to the vessel (7.4) and add, if necessary, the anti-foam agent (6.7) and the anti-bumping agent (6.8).

Measure 200 ml of the sodium hydroxide solution (6.2) at room temperature, bring it to a temperature of 95 to 100 °C (see 11.2), and add it to the contents of the vessel (7.4) in small portions.

Fit the condenser. Bring rapidly to the boil (in about 2 min) using the heating device (7.5) and continue to boil gently for 30 ± 1 min.

9.3.3.2 After the specified boiling period, add about 50 ml of cold water and rapidly separate the insoluble residue using the separating device (7.9) chosen. Wash the residue with 25 ml of the sulphuric acid solution (6.1) measured at room temperature and then raised to a temperature of 95 to 100 °C (see 11.2). Wash with water as described in 9.3.2.2. Dry the residue with one of the reagents (6.3); wash with solvent (6.4) to remove unsaponifiable fatty matter.

According to the filtration technique chosen (see annex B), collect all the residue in the incineration dish (7.6) or in the filter crucible (7.6).

9.3.4 Drying

Dry the incineration dish or the filter crucible with its contents in the oven (7.3) (see the note) at 130 ± 2 °C.

Allow to cool to room temperature in the desiccator (7.8) and quickly weigh to the nearest 0,5 mg.

Repeat these operations until the difference between two successive weighings, following drying in the oven and cooling in the desiccator, does not exceed 1 mg.

NOTE — The length of the periods in the oven depends on the methods of separation used. A total drying period of 2 h is usually sufficient.

9.3.5 Incineration

After drying, incinerate the dry residue in the muffle furnace (7.7) at 550 ± 25 °C to constant mass. Allow to cool to room temperature in the desiccator (7.8) and weigh again to the nearest 0,5 mg.

9.3.6 Number of determinations

Carry out at least two determinations on the same test sample.

9.4 Blank test

If asbestos is used as a filter aid (see clause B.2 of annex B), carry out a blank test under the same conditions as described in 9.3.

10 Expression of results

10.1 Method of calculation and formulae

10.1.1 Crude fibre content relative to product as received

The crude fibre content, expressed as a percentage by mass relative to the product as received, is given by the formula

a) *for products not requiring grinding*[1)]

$$[m_1 - (m_2 + m_3)] \times \frac{100}{m_0}$$

b) *for products requiring grinding*[1)]

$$[m_1 - (m_2 + m_3)] \times \frac{100}{m_0} \times \frac{100}{M'_S} \times \frac{M_S}{100}$$

where

m_0 is the mass, in grams, of the test portion (9.2);

1) If there was no blank test, delete m_3 from the formula.