International Standard ISO 6541

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

Agricultural food products — Determination of crude fibre content — Modified Scharrer method

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

First edition — 1981-06-01

UDC 664 : 543.868 **Ref. No. ISO 6541-1981 (E)**

Descriptors : agricultural products, food products, cereal products, tests, determination, indexes (ratios), insoluble matter.

Price based on 4 pages

Foreword

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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 6541 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 :

Austria
Brazil
Bulgaria
Canada
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Egypt, Arab Rep. of
Ethiopia
France
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Hungary
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Netherlands
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Poland
Portugal
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Spain
Thailand
Turkey
United Kingdom
USSR
Yugoslavia

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

NOTE — This International Standard is based on Standard No. 113 of the International Association for Cereal Chemistry (ICC).

Printed in Switzerland

Agricultural food products — Determination of crude fibre content — Modified Scharrer method

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 applicable to cereals and cereal products, as well as to certain products containing less than 1 % of crude fibre, for example yeasts, which are excluded from the field of application of ISO 5498.

3 Reference

ISO 5498, *Agricultural food products — Determination of crude fibre content — General 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 a mixture of acetic acid, nitric acid and trichloroacetic acid (Scharrer reagent). Separation and washing of the insoluble residue on a filter crucible.

Drying and weighing of the insoluble residue, and determination of the loss of 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 Scharrer reagent, consisting of a mixture having the following composition :

— acetic acid solution, prepared by diluting 730 g of 96 % (*m*/*m*) glacial acetic acid with water to 1 000 g	900 ml
— nitric acid, concentrated, ϱ_{20} = 1,38 g/ml	60 ml
— trichloroacetic acid, crystalline	24 g

6.2 Acetone.

6.3 Diethyl ether.

6.4 Sea sand, prepared as follows.

Sieve the sand successively through sieves of wire gauze of aperture sizes 160 µm and 125 µm, complying with the requirements of ISO 3310/1.

Boil the sand retained by the 125 µm aperture size sieve for 30 min in 4 mol/l hydrochloric acid solution. Rinse the sand with water until the rinsing water is free from chloride, as indicated by the absence of a reaction with silver nitrate solution, and incinerate in the muffle furnace (7.9) at 550 ± 25 °C to constant mass (usually at least 6 h are required).

6.5 Boiling aid : crushed porcelain.

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 Wide-mouthed vessel, provided with a condenser, for example a conical flask of capacity 200 to 300 ml, fitted with a reflux condenser.

7.4 Suction flask.

7.5 Water-jet pump.

7.6 Filter crucible, of silica, having a fritted silica plate of porosity grade P 100 (pore diameter 40 to 100 µm), prepared as indicated in 11.1.

7.7 Desiccator, containing an efficient desiccant.

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

7.9 Muffle furnace, capable of being controlled at 550 ± 25 °C.

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 of which 95 % pass through the sieve (7.2) 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 of which less than 95 % pass through the sieve (7.2) 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 at least 95 % of the product passes through the sieve (7.2).

NOTE — Such a degree of fineness of grinding is unnecessary for products containing parts which are rich in cellulosic substances, such as grains in husk or husks of grain.

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

Weigh, to the nearest 1 mg, a mass of the prepared test sample (9.1) corresponding to 0,05 to 0,15 g of crude fibre [i.e. 1 to 3 g for a crude fibre content of 5 % (*m*/*m*)].

For products with low crude fibre contents, and in particular those having crude fibre contents less than 1 % (*m*/*m*), take a test portion of 3 g.

9.3 Determination

NOTE — Products rich in fatty substances may require defatting beforehand (methods are described in annex A of ISO 5498). This operation is not required for cereal and cereal products or for yeasts.

9.3.1 Digestion

Transfer the test portion to the digestion vessel (7.3) and suspend it in about one-third of the total volume of the Scharrer reagent (6.1). Generally, the total volume, in millilitres, of the Scharrer reagent is numerically twenty times the mass, in grams, of the test portion (in no case shall the volume used be less than 40 ml).

Using a glass rod, which shall be left in the digestion vessel, break up any large lumps that may have been formed.

Carefully rinse the interior walls of the digestion vessel with the remaining two-thirds of the Scharrer reagent in order to remove any particles of the product adhering to the walls. Fit the condenser. Bring the contents of the vessel to the boil in 3 ± 0,5 min and maintain boiling for 30 ± 1 min (see 11.2). Do not stir or shake while boiling.

9.3.2 Separation and washing of the residue

After the specified boiling period, transfer the boiling solution to the filter crucible (7.6) prepared as described in 11.1, and filter under reduced pressure using the suction flask (7.4) and the water-jet pump (7.5).

Rinse the vessel and the glass rod with 50 to 70 ml portions of water (temperature between 95 and 100 °C) and quantitatively transfer the insoluble residue to the filter crucible using a glass rod fitted with a rubber cap. Repeat the washing until the filtrate is substantially neutral to litmus paper; this usually requires 300 to 400 ml water.

After washing, disconnect the water-jet pump, immediately empty the suction flask and fill the filter crucible three times with the acetone (6.2), allowing the solvent to drain through under gravity. If this operation requires too much time, apply gentle suction to obtain a flow rate not exceeding one drop per second.

Rinse twice with the diethyl ether (6.3) and remove the ether by filtration under reduced pressure using the suction flask and water-jet pump.

9.3.3 Drying

Dry the filter crucible with its contents in the drying oven (7.8) maintained at 130 ± 2 °C.

Allow them to cool to room temperature in the desiccator (7.7) and weigh them rapidly to the nearest 0,5 mg.

Repeat these operations until the difference between two successive weighings, separated by a period in the oven followed by cooling in the desiccator, does not exceed 1 mg.

NOTE — Drying for 1 h is generally sufficient.

9.3.4 Incineration

After drying, incinerate the dry residue in the muffle furnace (7.9) at 550 ± 25 °C for 30 min.

Place the crucible on a refractory plate and allow to cool to room temperature in the desiccator (7.7). Weigh rapidly to the nearest 0,5 mg.

9.3.5 Number of determinations

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

10 Expression of results

10.1 Method of calculation and formulae

10.1.1 Crude fibre content relative to the 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*

$$(m_1 - m_2) \times \frac{100}{m_0}$$

b) *for products requiring grinding*

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

where

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

m_1 is the total mass, in grams, of the dry residue and its support after drying (9.3.3);

m_2 is the total mass, in grams, of the dry residue and its support after incineration (9.3.4);

M_S is the dry matter content of the product as received, determined as indicated in 9.1.3.1;

M'_S is the dry matter content of the test sample, determined as indicated in 9.1.2.2 or 9.1.3.3.

10.1.2 Crude fibre content relative to the dry matter content of the product

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

$$(m_1 - m_2) \times \frac{100}{m_0} \times \frac{100}{M'_S}$$

where m_0, m_1, m_2 and M'_S have the same meanings as in 10.1.1.

10.1.3 Case of preliminary drying

If a preliminary drying was carried out (see 9.1.1), the crude fibre content, expressed as a percentage by mass relative to the product as received, is obtained by multiplying the result calculated according to 10.1.1 by the ratio

$$\frac{m_5}{m_4}$$

where

m_4 is the mass, in grams, of the initial moist sample before preliminary drying;

m_5 is the mass, in grams, of the same sample after preliminary drying.

10.1.4 Result

Take as the result the arithmetic mean of the two determinations (9.3.5), provided that the requirements for repeatability (see 10.2) are satisfied.

10.2 Repeatability

The difference between the results of the two determinations (9.3.5), carried out simultaneously or in rapid succession by the same analyst, shall not exceed :

0,1 (absolute value) for crude fibre contents below 2 % (*m*/*m*);

5 % (relative value) for crude fibre contents greater than 2 % (*m*/*m*).

11 Notes on procedure

11.1 Preparation of filter crucibles

11.1.1 Prior to first use, carefully clean the filter crucibles (7.6) and heat in the muffle furnace (7.9), controlled at 550 ± 25 °C, for 6 h.

11.1.2 Spread on the crucible plate 5 to 6 g of the sea sand (6.4). Level the surface and spread over the top 4 to 5 g of crushed porcelain (6.5) and again level the surface. Using gentle pressure, place a perforated porcelain disc on top of these two layers. This filling may be reused without cleaning or preliminary treatment, but care should be taken to ensure that the layers remain in their initial positions.

11.2 Precautions

During digestion (see 9.3.1), heating shall be performed with care in order to avoid over-heating and too rapid boiling. The foam formed in the vessel shall never be allowed to exceed a height of 10 mm. Control is exerted by varying the intensity of heating.

12 Test report

The test report shall show the method used and the result obtained (in particular, whether it is expressed relative to the product as received or to the dry matter content). It shall also mention all operating conditions not specified in this International Standard or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all the details required for the complete identification of the sample.