INTERNATIONAL STANDARD

ISO 15598

First edition
1999-06-01

Tea — Determination of crude fibre content

Thé — Détermination de l'indice d'insoluble dit «cellulosique»

Reference number
ISO 15598:1999(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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 15598 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 8, *Tea*.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

Tea — Determination of crude fibre content

1 Scope

This International Standard specifies a method for the determination of crude fibre content in tea.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1573:1980, *Tea — Determination of loss in mass at 103 °C*.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*.

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
crude fibre content
the whole of the substances which are insoluble and combustible under the operating conditions specified in this International Standard

NOTE It is expressed as a mass fraction, in percent, of the product on a dry basis [formerly expressed as % (m/m)].

4 Principle

The suitably ground sample is successively treated with boiling sulfuric acid solution and sodium hydroxide solution. The residue is separated by filtration, washed, dried, weighed and then ashed. The loss in mass resulting from ashing is called the crude fibre content.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Water, complying with grade 3 of ISO 3696.

5.2 Sulfuric acid stock solution, $c(\frac{1}{2}H_2SO_4)$ = 2,040 ± 0,040 mol/l (corresponding to 100 g of sulfuric acid per litre of solution).

Add 275 ml of concentrated sulfuric acid (ρ_{20} = 1,84 g/ml) to water, cool and dilute to 5 l.

CAUTION — Wear gloves and face protection.

5.3 Sulfuric acid working solution, $c(½H_2SO_4)$ = 0,255 ± 0,005 mol/l (corresponding to 12,5 g of sulfuric acid per litre of solution).

Dilute 125 ml of the sulfuric acid stock solution (5.2) to 1 l.

5.4 Sodium hydroxide stock solution, c(NaOH) = 2,504 ± 0,040 mol/l (corresponding to 100 g of sodium hydroxide per litre of solution).

Dissolve 500 g of sodium hydroxide in water, cool and dilute to 5 l.

CAUTION — Wear gloves and face protection.

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

Dilute 125 ml of the sodium hydroxide stock solution (5.4) to 1 l.

5.6 Octan-1-ol, as antifoaming agent.

5.7 Hydrochloric acid, 1 % solution (volume fraction).

Dilute 10 ml of concentrated hydrochloric acid (ρ_{20} = 1,19 g/ml) to 1 l.

CAUTION — Wear gloves and face protection.

5.8 Ethanol, of minimum purity 95 % (volume fraction).

5.9 Acetone.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Laboratory mill, of hammer or centrifugal type, fitted with a 1 mm screen.

6.2 Conical flasks, of 1 l capacity, with plain neck (or ground glass neck if reflux condensers are to be used).

6.3 Dispenser, capable of dispensing 200 ml of hot liquid.

6.4 Heating unit, equipped for heating a bank of 1 l conical flasks.

6.5 Cold-finger condensers, to fit into the necks of 1 l conical flasks (or reflux condensers if apparatus with ground glass joints is being used).

6.6 Buchner flasks, with rubber adaptors and fitted with Hartley funnels for 12,5 cm diameter filter paper, plus adaptor funnels for 70 ml sintered-glass crucibles.

6.7 Sintered-glass crucibles, of porosity No. 1 or P 160 (pore size 100 µm to 160 µm), of 40 mm plate diameter and 70 ml capacity.

6.8 Oven, fan-assisted, capable of maintaining a temperature of 103 °C ± 2 °C.

6.9 Muffle furnace, capable of maintaining a temperature of 550 °C ± 10 °C.

6.10 Desiccator, containing an efficient desiccant.

6.11 Filter papers, of 12,5 cm diameter, hardened, ashless grade, with a porosity of 20 µm to 25 µm.

NOTE Whatman No. 541[1)] have been found to be suitable.

7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport and storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 1839.

8 Preparation of test sample

Using the mill (6.1), grind the sample to pass through the 1 mm screen.

9 Procedure

NOTE If it is required to check whether the repeatability limit (11.2) is met, carry out two single determinations in accordance with 9.1 to 9.3 under repeatability conditions.

9.1 Determination of dry matter content

Calculate the dry matter content (w) from the moisture content (loss in mass at 103 °C) determined on a portion of the test sample (clause 8) in accordance with ISO 1573.

9.2 Test portion

Weigh, to the nearest 0,001 g, 2 g to 3 g of the test sample (clause 8) into a 1 l conical flask (6.2). Record the mass (m_0).

9.3 Determination

9.3.1 Using the dispenser (6.3), add to the sample 200 ml of the sulfuric acid working solution (5.3), measured at room temperature and brought to boiling.

CAUTION — Take care to avoid splashing the boiling acid. Wear gloves and face protection.

9.3.2 Add two or three drops of the antifoaming agent (5.6), insert the condenser (6.5) into the neck of the flask, and heat using the heating unit (6.4) to boiling within 2 min. Continue boiling gently for 30 min, rotating the flask occasionally to mix the contents and to remove particles of the sample adhering to the sides.

The times given are critical.

9.3.3 Prepare a Buchner flask (6.6) with a Hartley funnel with a wet filter paper (6.11).

9.3.4 At the end of the boiling period, pour the acid digest into a shallow layer of hot water contained in the funnel under gentle suction.

Filtration should be completed within 10 min. If it is not, repeat the determination using a smaller mass of sample.

---

1) Whatman No. 541 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

9.3.5 Rinse out the flask with two portions of approximately 50 ml of boiling water, and pour through the filter funnel.

9.3.6 Using the dispenser (6.3), wash the insoluble matter from the filter paper into the original 1 l conical flask using 200 ml of the working sodium hydroxide solution (5.5), measured at room temperature and brought to boiling.

CAUTION — Take care to avoid splashing the boiling alkali. Wear gloves and face protection.

9.3.7 Add two or three drops of the antifoaming agent (5.6) and boil for 30 min using the same procedure as with the acid treatment (see 9.3.2).

The times given are critical.

9.3.8 Using boiling water, transfer all the insoluble matter into the sintered glass crucible (6.7) fitted to the Buchner flask by means of an adaptor, applying gentle suction.

9.3.9 Wash the residue successively with approximately 50 ml portions of boiling water, the hydrochloric acid solution (5.6) and boiling water again. Finally, wash the residue twice with the ethanol (5.7) and then three times with the acetone (5.8).

9.3.10 Heat the crucible and residue in the oven (6.8) maintained at 103 °C for 2 h. Allow to cool in the desiccator (6.10) and weigh to the nearest 0,001 g. Return the crucible to the oven and heat again for 1 h. Cool in the desiccator and weigh. Repeat these operations until the difference between successive weighings does not exceed 0,001 g.

NOTE It is an acceptable alternative, and it may be more convenient, to heat the crucible and residue overnight.

Record the mass (m_1).

9.3.11 Place the crucible and residue in the muffle furnace (6.9) maintained at 550 °C for at least 1 h. Allow to cool in the desiccator (6.10) and weigh to the nearest 0,001 g. Record the mass (m_2).

10 Calculation

The crude fibre content, w, expressed as mass fraction, in percent, of the sample on a dry basis, is given by the formula:

$$w = \frac{m_1 - m_2}{m_0} \times 100 \times \frac{100}{w_D}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the crucible and residue after drying (9.3.10);

m_2 is the mass, in grams, of the crucible and residue after heating in the furnace (9.3.11);

w_D is the dry matter content, expressed as a mass fraction, in percent, of the test sample (9.1).

11 Precision

11.1 Interlaboratory test

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

11.2 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 in not more than 5 % of cases be greater than the repeatability (r) values given in Table A.1.

11.3 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 in not more than 5 % of cases be greater than the reproducibility (R) values given in Table A.1.

12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained, or, if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Results of interlaboratory test

An interlaboratory test, carried out in 1994 under the auspices of the International Organization for Standardization, gave the statistical results (evaluated in accordance with ISO 5725 [2)]) shown in Table A.1.

Table A.1 — Repeatability and reproducibility values

Sample identification	Black tea				
	1	2	3	4	5
Number of participating laboratories	11	11	11	11	11
Number of accepted test results	11	11	11	11	11
Mean crude fibre content, %, dry matter	28,63	9,67	18,95	22,85	9,35
Repeatability standard deviation, s_r	0,433 8	0,234 1	0,272 8	0,214 3	0,153 7
Repeatability coefficient of variation, %	1,50	2,42	1,44	0,94	1,64
Repeatability limit, r (2,8 × s_r)	1,214 6	0,655 5	0,763 8	0,599 9	0,430 5
Reproducibility standard deviation, s_R	1,103 2	0,674 6	1,235 4	1,185 5	0,540 1
Reproducibility coefficient of variation, %	3,85	6,98	6,52	5,19	5,77
Reproducibility limit, R (2,8 × s_R)	3,089 0	1,888 8	3,459 1	3,319 5	1,512 3

2) ISO 5725:1986 (now withdrawn) was used to obtain the precision data.

Bibliography

[1] ISO 1839:1980, *Tea — Sampling*.

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ICS 67.140.10

Price based on 7 pages