INTERNATIONAL STANDARD

ISO 2817

Second edition
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Tobacco and tobacco products — Determination of silicated residues insoluble in hydrochloric acid

Tabac et produits du tabac — Détermination des résidus silicatés insolubles dans l'acide chlorhydrique

Reference number
ISO 2817:1999(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 2817 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, Subcommittee SC 1, *Physical and dimensional tests*.

This second edition cancels and replaces the first edition (ISO 2817:1974), which has been technically revised.

Annex A of this International Standard is for information only.

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Tobacco and tobacco products — Determination of silicated residues insoluble in hydrochloric acid

1 Scope

This International Standard specifies a method for the determination of the percentage of hydrochloric-acid-insoluble extraneous silica particles, especially particles of sand, in tobacco (whole leaf, cut tobacco, tobacco scraps and dust) and tobacco products.

It is particularly useful to know the proportion of these residues, in the following circumstances:

— when buying tobacco leaves, in order to check the cleanness;

— before processing tobacco and tobacco products.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 4874, *Tobacco — Sampling of batches of raw material — General principles*.

ISO 6488-1, *Tobacco — Determination of water content — Part 1: Karl Fischer method*.

3 Term and definition

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

3.1
hydrochloric-acid-insoluble silicated residues
residual material of whole leaf or cut tobacco, tobacco scraps and dust, obtained after incineration and extraction of hydrochloric-acid-soluble material under the conditions specified in this International Standard

4 Principle

A test portion is incinerated at a temperature of 650 °C ± 50 °C. The ashes are leached with hydrochloric acid, then re-incinerated at 650 °C ± 50 °C. The residue is weighed.

5 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

5.1 Hydrochloric acid, c(HCl) = 4 mol l^{-1}.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Muffle furnace, well ventilated, capable of being regulated at 350 °C ± 50 °C and 650 °C ± 50 °C.

6.2 Wide-mouth flat-bottomed crucible, in porcelain or platinum, of capacity and dimensions appropriate to the volume of the test portion. In general, crucibles having a diameter of 50 mm to 70 mm and a height of 30 mm are suitable.

6.3 Screen, with a 2 mm size aperture, in accordance with ISO 565.

6.4 Rapid filter paper, ashless, hardened. [1)]

6.5 Analytical balance, with a resolution of 0,000 1 g.

6.6 Mixer.

7 Sampling

Sampling shall have been carried out in accordance with ISO 4874.

The size of the laboratory sample shall be sufficient to ensure that it is representative of the batch.

8 Preparation of test sample

If necessary, dry the sample to facilitate the grinding. The maximum water content shall be 12 %.

Grind the laboratory sample until the whole sample passes through the screen (6.3).

Thoroughly mix the ground sample, preferably by mechanical means.

If the sample is not to be analysed within 4 days after the preparation, it should be stored at a temperature of between 0 °C and 5 °C in a wide-mouth airtight container of such a capacity that the sample may be adequately mixed by inverting the container at least twice before taking any sample for analysis.

9 Determination of water content

Take a test portion from the prepared test sample following clause 8, and determine the water content as described in ISO 6488-1.

Express the mass fraction of moisture (water content), w_1, of the sample in percent.

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1) It has been proved by experience, that Whatman Grade No. 540 filter paper is suitable.

Whatman filter paper Grade No. 540 the tradename of a product supplied by Whatman. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

10 Procedure

10.1 Test portion

Weigh, to the nearest 0,001 g, the clean crucible (6.2) previously dried at 650 °C ± 50 °C.

From the container take approximately 10 g of the prepared test sample (clause 8) and spread it uniformly over the bottom of the crucible.

Weigh the crucible and the test portion to the nearest 0,001 g.

The mass (m_1) of the test portion can then be calculated by subtraction.

10.2 Determination

Place the crucible containing the test portion (10.1) in the muffle furnace (6.1) and heat to a temperature of 350 °C ± 50 °C until the test portion is completely charred, without any formation of smoke.

Increase the temperature of the furnace to 650 °C ± 50 °C for about 30 min. Allow it to cool to 350 °C ± 50 °C, and then remove the crucible from the furnace. After it has cooled down to ambient temperature, add slowly 40 ml of hydrochloric acid (5.1), allowing it to run down the wall of the crucible.

CAUTION: Add the first few millilitres carefully to avoid vigorous effervescence.

Stir gently from time to time with a glass rod for about 10 min. Filter the contents of the crucible through the filter paper (6.4). Collect all the residue on the filter. Use a rubber-tipped glass rod to remove any residue still adhering to the sides of the crucible. Rinse the crucible with about 25 ml of water and filter. Carefully wash the residue on the filter paper several times with about 25 ml of water, until neutral pH.

Place the filter paper containing the residue in the crucible (6.2) and transfer the crucible into the muffle furnace (6.1), the temperature of which shall be less than 200 °C. Heat to 650 °C ± 50 °C for 30 min. Allow it to cool to 350 °C ± 50 °C. Remove the crucible from the furnace, transfer it to a desiccator containing dried silica gel and allow it to cool to ambient temperature. Weigh the crucible and residue to the nearest 0,001 g.

Calculate the mass (m_2) of the residue.

11 Expression of results

The hydrochloric-acid-insoluble silicated residue, expressed as the mass fraction, w, in percent, is obtained by the equation:

$$w = \frac{m_2}{m_1\left(1 - \frac{w_1}{100}\right)} \times 100\ \%$$

where

w_1 is the mass fraction of water, in percent, of the test portion, as determined according to clause 9;

m_1 is the mass (expressed in grams) of the test portion;

m_2 is the mass (expressed in grams) of the residue.

12 Precision

12.1 Interlaboratory test

Details of an interlaboratory test on 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.

12.2 Repeatability (r)

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:

level 0 %: r = 0,12

level 5 %: r = 0,35

level 10 %: r = 0,47

12.3 Reproducibility (R)

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:

level 0 %: R = 0,12

level 5 %: R = 0,83

level 10 %: R = 1,73

13 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 (in particular the temperature of incineration if it was different from 650 °C ± 50 °C), or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained;

— if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Interlaboratory test

An international collaborative study involving 12 laboratories, 4 samples and 5 repeats was organized by ISO/TC 126/SC 1 in 1996. The results obtained were subjected to statistical analysis in accordance with ISO 5725-2 [1] to give the precision data shown in Table A.1.

Table A.1 — Statistical results of the interlaboratory test
(incineration at the temperature of 650 °C ± 50 °C)

	Proportion of added silica in tobacco		
	0 %	**5 %**	**10 %**
Number of participating laboratories after eliminating outliers	7	9	10
Mean value of silicated residue content, %, on dry matter	0,51	5,56	10,33
Repeatability standard deviation, s_r	0,04	0,12	0,17
Coefficient of variation of repeatability, %	8,21	2,22	1,61
Repeatability limit, r (2,8 s_r)	0,12	0,35	0,47
Reproducibility standard deviation, s_R	0,04	0,29	0,61
Coefficient of variation of reproducibility, %	8,21	5,30	5,92
Reproducibility limit, R (2,8 s_R)	0,12	0,83	1,73
NOTE The results given above were obtained at the temperature of incineration of 650 °C ± 50 °C which is the temperature adopted in this International Standard. However, comparative interlaboratory tests indicated that the results obtained at an incineration temperature of 850 °C ± 50 °C were not statistically different at the 95 % probability level.			

Bibliography

[1] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

[2] ISO/IEC Guide 25:1990, *General requirements for the competence of calibration and testing laboratories*.

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