INTERNATIONAL STANDARD ISO 2817

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Tobacco and tobacco products – Determination of silica content

Tabac et produits du tabac — Détermination de la teneur en silice

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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 2817 was drawn up by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, and circulated to the Member Bodies in June 1972.

It has been approved by the Member Bodies of the following countries :

Australia
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India
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Tobacco and tobacco products — Determination of silica content

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies two methods for the determination of the silica content of tobacco :

- the incineration method (method 1);
- the wet digestion method (method 2).

These methods are suitable for leaf and cut tobacco.

2 DEFINITION

By **silica** is meant the residual material of whole leaf or cut tobacco obtained after incineration and extraction with hydrochloric acid (method 1) or by digestion and incineration (method 2) under the conditions specified.

3 SAMPLING

3.1 Sampling method

Carry out sampling by the method described in ISO . . . [1].

3.2 Preparation of the sample

For whole leaf or cut tobacco, air dry the laboratory sample to a water content of between 5 and 9 % and mill it until the whole of the laboratory sample passes a 2 mm mesh screen. Thoroughly mix the milled sample, preferably by mechanical means. If the sample is not to be examined at once, but is to be kept for 4 days or more, it should be stored at a temperature of between 0 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.

4 METHOD 1 : INCINERATION METHOD

4.1 Principle

Incineration of the test portion in air at a temperature of 550 °C, leaching with hydrochloric acid, re-incineration and weighing of the residue.

4.2 Reagents

4.2.1 Hydrochloric acid, 1 N.

4.2.2 Nitric acid, concentrated (ρ_{20} 1,42 g/ml).

4.3 Apparatus

Ordinary laboratory apparatus not otherwise specified, and the following items :

4.3.1 Analytical balance.

4.3.2 Muffle furnace, well ventilated, giving an adequate zone of uniform temperature at 550 ± 25 °C.

4.3.3 Dish, rectangular, approximately 50 to 70 mm by 50 mm and 30 mm deep, of silica, or alternatively a platinum dish of similar dimensions.

4.3.4 Hotplate, electrically heated, capable of attaining a temperature of 400 °C.

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[1] In preparation.

4.4 Procedure

4.4.1 *Test portion*

Weigh, to the nearest 0,001 g, the clean dry dish (4.3.3). Take from the container approximately 10 g of the prepared sample and spread it uniformly over the bottom of the dish. Reweigh the dish and test portion to the nearest 0,001 g. The mass m_1 of the test portion can then be calculated to the nearest 0,001 g.

4.4.2 *Determination*

Place the dish (4.3.3) containing the test portion on the hotplate (4.3.4) and heat slowly at a temperature of 340 to 380 °C in a fume cupboard, until the test portion is completely charred.

Transfer the dish to the muffle furnace (4.3.2), pre-heated to a temperature of 550 ± 25 °C, and heat the test portion for 2 h at this temperature, leaving the furnace door slightly open to provide adequate ventilation. Remove the dish from the furnace and allow to cool. If it is evident that all the carbon is not burnt away at the end of this period, continue the heating until this is achieved.

Moisten the ash with approximately 5 ml of distilled water.

Cover with a watch glass and add 40 ml of hydrochloric acid (4.2.1) down the sides of the dish. Add the first few millilitres carefully to avoid vigorous effervescence. Transfer the dish to a boiling water bath and leave for 30 min, stirring gently from time to time with a glass rod. Filter the contents of the dish through an ashless medium-fine filter paper, transferring all the residue in the dish to the filter paper. Use a rubber-tipped glass rod to remove any residues still adhering to the sides of the dish. Rinse the dish with warm distilled water, transferring the rinsings to the filter paper. Carefully wash the residues on the filter paper several times with warm distilled water.

Transfer the filter paper and residue back to the silica dish. Add a drop of concentrated nitric acid (4.2.2). Heat on the hotplate to charring and transfer the dish as before to the muffle furnace. Heat at 550 ± 25 °C for 30 min, leaving the furnace door slightly open as before.

Remove the dish from the furnace and allow to cool for 10 min. Transfer the dish to a desiccator containing silica gel and cool for a further 15 min. Reweigh the dish and residue to the nearest 0,001 g. The mass m_2 of the residue can then be calculated to the nearest 0,001 g.

5 METHOD 2 : WET DIGESTION METHOD

5.1 Principle

Destruction of the organic material of the test portion by heating with an oxidizing acid mixture, separation by filtration and incineration of the inorganic residue.

5.2 Reagents

5.2.1 Nitric acid, concentrated (ρ_{20} 1,42 g/ml).

5.2.2 Perchloric acid, 72 % (*m/m*).

WARNING : Evaporations with perchloric acid must be carried out in a well-ventilated fume cupboard which is kept clean and free from combustible materials and is suitably constructed to resist exposure to perchloric acid fumes. In addition adequate shielding against possible explosion shall be provided for operators.

5.3 Apparatus

Usual laboratory apparatus not otherwise specified, and the following items :

5.3.1 Analytical balance.

5.3.2 Muffle furnace, well ventilated, giving an adequate zone of uniform temperature at 550 ± 25 °C.

5.3.3 Hotplate, electrically heated, capable of attaining a temperature of 400 °C.

5.3.4 Conical flask, 50 ml capacity, made of heat-resistant glass, provided with ground glass joint.

5.3.5 Condenser, air cooled, provided with ground glass joint.

5.3.6 Silica crucible, approximately 40 mm diameter by 20 mm deep.

5.4 Procedure

5.4.1 *Test portion*

Weigh, to the nearest 0,001 g, the clean dry conical flask (5.3.4). Add approximately 1 g of the prepared sample. Reweigh the flask and test portion to the nearest 0,001 g. The mass m_1 of the test portion can then be calculated to the nearest 0,001 g.

5.4.2 *Determination*

Add 5 ml of concentrated nitric acid (5.2.1) to the test portion in the flask; swirl to mix and leave for 5 min. Add 5 ml of perchloric acid (5.2.2) to the mixture in the flask and attach the reflux air condenser (5.3.5). The condenser is intended to prolong the digestion reactions and to coagulate the siliceous residue.

Transfer the assembly to the electric hotplate (5.3.3) and heat to boiling in a fume cupboard until the evolution of brown fumes ceases. Increase the temperature of the hotplate to produce rapid boiling and evaporate the solution until the volume is reduced to 2 to 3 ml. Remove the assembly from the hotplate and allow to cool.

Wash down the condenser and neck of the flask with hot distilled water using a total of approximately 20 ml and filter the contents of the flask through an ashless medium-fine filter paper. Use a rubber-tipped glass rod to remove any particles of residue still adhering to the sides of the flask. Rinse the flask and the rod with warm distilled water into the filter paper. Finally wash the residues on the filter paper several times with warm distilled water.

Weigh the clean dry silica crucible (5.3.6) to the nearest 0,001 g and transfer the filter paper and residue to it. Place the crucible on the hotplate (5.3.3) at a temperature of about 350 °C and heat until the paper is completely charred. Transfer the crucible to the muffle furnace, preheated to 550 ± 25 °C, and ignite at this temperature for 30 min leaving the furnace door slightly open to provide adequate ventilation.

Remove the crucible from the furnace and allow to cool for 10 min. Transfer the crucible to a desiccator containing silica gel and cool for a further 15 min. Weigh the crucible and ignited residues to the nearest 0,001 g. The mass m_2 of the residue can then be calculated to the nearest 0,001 g.

6 EXPRESSION OF RESULTS

The silica content, S, expressed as a percentage by mass of the test portion, is given by the formula :

$$S = \frac{m_2 \times 10^4}{m_1 \times (100 - E)}$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the ignited residue;

E is the water content as a percentage by mass of the test portion determined according to ISO . . . (1).

7 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

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

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1) In preparation.

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