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Tobacco and tobacco products — Determination of nicotine purity — Gravimetric method using tungstosilicic acid

Tabac et produits du tabac — Détermination de la pureté de la nicotine — Méthode gravimétrique à l'acide tungstosilicique

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Foreword

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International Standard ISO 13276 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

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Annex A of this International Standard is for information only.

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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
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Tobacco and tobacco products — Determination of nicotine purity — Gravimetric method using tungstosilicic acid

1 Scope

This International Standard specifies a method for the gravimetric determination of the purity of nicotine using tungstosilicic acid.

The method is applicable to pure nicotine or nicotine salts used to calibrate analytical methods for the determination of nicotine in the field of tobacco, tobacco products and smoke analysis.

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2 Principle

Complex formation of nicotine or its salts with tungstosilicic acid to form insoluble nicotine silicotungstate. Determination of the precipitate mass by filtration using either a sintered glass crucible in combination with oven-drying or an ashless filter paper in combination with incineration.

3 Reagents

Use only reagents of recognized analytical reagent grade and distilled water or water of at least equivalent purity.

3.1 Tungstosilicic acid solution

Dissolve 12 g of dodeca-tungstosilicic acid ($SiO_2 12WO_3 \cdot 26H_2O$) in 100 ml of water.

NOTE 1 Avoid the use of the other forms of tungstosilicic acid such as $4H_2O \cdot SiO_2 10WO_3 \cdot 3H_2O$ or $4H_2O \cdot SiO_2 12WO_3 \cdot 20H_2O$ as they do not yield crystalline precipitates with nicotine.

3.2 Hydrochloric acid solution, HCl, 20 % (*V/V*).

Dilute 20 ml of hydrochloric acid, ρ_{20}(HCl) = 1,18 g/ml, to 100 ml with water.

3.3 Hydrochloric acid solution, HCl, 0,1 % (*V/V*).

Dilute 5 ml of hydrochloric acid solution (3.2) to 1 litre with water.

3.4 Nicotine solution, $\rho(C_{10}H_{14}N_2)$ = 0,1 mg/ml.

Dissolve 2,5 mg of nicotine ($C_{10}H_{14}N_2$) in water using a volumetric flask (4.1) and dilute to 25 ml with water.

4 Apparatus

Usual laboratory apparatus and, in particular, the following items:

4.1 Volumetric flask, of 25 ml capacity.

4.2 Beakers, of 250 ml capacity.

4.3 Watch glasses.

4.4 Glass stirring rods.

4.5 Desiccator, containing an effective drying agent.

4.6 Apparatus for glass filter filtration procedure.

4.6.1 Sintered glass crucible (Gooch type), of porosity 2 (40 µm to 100 µm).

4.6.2 Filter flask (Buchner flask).

4.6.3 Vacuum source.

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4.6.4 Laboratory oven, capable of maintaining a temperature of (120 ± 5) °C.

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4.7 Apparatus for filter paper filtration procedure.

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4.7.1 Ashless filter paper. [1)]

4.7.2 Porcelain or platinum crucibles.

4.7.3 Gas or electric Bunsen burner, capable of maintaining a temperature higher than 600 °C.

4.7.4 Furnace, capable of maintaining a temperature higher than 600 °C (optional).

4.8 Analytical balance, with a resolution of 0,1 mg.

5 Procedure

5.1 Precipitation procedure

Weigh, to the nearest 0,0001 g, approximately 0,1 g of the nicotine alkaloid (or the equivalent amount of nicotine salt) (*m*) in each of five 250 ml beakers (4.2) equipped with glass stirring rods (4.4).

Add 100 ml of water to each beaker. Add 2 ml of 20 % hydrochloric acid solution (3.2) to each beaker and stir. Do not remove the stirring rod.

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1) The ashless filter paper Whatman No. 42 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.

Add slowly 15 ml of the tungstosilicic acid solution (3.1) while stirring constantly during the addition. Cover each beaker with a watch glass (4.3) leaving the stirring rod in place and allow to stand overnight. Before filtering, stir the precipitate to ensure that it settles quickly and is of a crystalline form. Check for complete precipitation with a few extra drops of the tungstosilicic acid solution.

5.2 Filtration procedure

Filtration can be performed by either of the procedures given in 5.2.1 or 5.2.2

5.2.1 Glass filter filtration procedure

Dry each glass filter crucible (4.6.1) in the oven (4.6.4) at 120 °C to constant mass (± 1 mg). Store in the desiccator (4.5).

Weigh, to the nearest 0,0001 g, each glass crucible (4.6.1) (m_1) and filter the precipitate directly into the glass filter using the Buchner flask (4.6.2) and vacuum source (4.6.3). Ensure that the precipitate is removed from the sides of the beaker and the glass stirring rod by washing into the filter with hydrochloric acid solution (3.3) approximately three times using 15 ml each. Discard the washings.

Rinse with a further aliquot portion of hydrochloric acid solution (3.3) (up to 400 ml may be required) which should be collected and tested with a few drops of nicotine solution (3.4) to ensure that no opalescence occurs; i.e. all tungstosilicic acid has been removed.

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Dry each glass crucible and precipitate in the oven (4.6.4) for 3 h at 120 °C. Allow to cool in the desiccator and weigh to the nearest 0,0001 g (m_2). Place the filters back in the oven for 1 h, allow to cool and reweigh. Repeat if necessary until a constant mass (± 1 mg) is obtained.

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5.2.2 Filter paper filtration procedure

Filter the precipitate directly onto an ashless filter paper (4.7.1). Ensure that the precipitate is removed from the sides of the beaker and the stirring rod by washing into the filter with hydrochloric acid solution (3.3) approximately three times using 15 ml each. Discard the washings.

Rinse with a further aliquot portion of hydrochloric acid solution (3.3) (up to 400 ml may be required) which should be collected and tested with a few drops of nicotine solution (3.4) to ensure that no opalescence occurs, i. e. all tungstosilicic acid has been removed.

Dry each crucible (4.7.2) on the Bunsen burner (4.7.3) or in the furnace (4.7.4) at 600 °C until constant mass (± 1 mg). Store in the desiccator (4.5).

Weigh, to the nearest 0,0001 g, each crucible (4.7.2) (m_1). Transfer the filter paper with the precipitate to the crucible. Place the crucible on a silica triangle resting on a tripod, heat gently at first and then ignite with the Bunsen burner (4.7.3). The crucible contents have to be broken up very carefully to ensure complete removal of the carbon. The final residue should be greenish/yellow in colour. Allow to cool in the desiccator (4.5) and weigh to the nearest 0,0001 g (m_2). Repeat the heating process until a constant mass (± 1 mg) is obtained.

NOTE 2 After ignition of the filter paper it may be convenient to leave the crucible in the furnace (4.7.4) at above 600 °C overnight. This technique ensures that no further heating is required.

6 Expression of results

The nicotine purity or the nicotine salt purity, NP, expressed as a percentage by mass, is given by the following formula:

$$NP = \frac{(m_2 - m_1) \times C}{m} \times 100 \quad (1)$$

where

m_1 is the mass, in milligrams, of the dried empty crucible;

m_2 is the mass, in milligrams, of the crucible with precipitate after drying (5.2.1) or ignition (5.2.2);

C is a factor depending on the filtration procedure;
~ 0,1012 for the glass filter filtration procedure (5.2.1);
~ 0,1141 for the filter paper filtration procedure (5.2.2);

m is the nicotine equivalent mass, in milligrams, of the sample.

When the method is used to determine the purity of a nicotine salt, calculate the nicotine equivalent mass m from the mass of nicotine salt m_s by the following formula:

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$$m = m_s \times \frac{162,2}{M_s} \quad (2)$$

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where M_s is the molecular mass of the nicotine salt.

Take as the test result the arithmetic mean of the five determinations, report the result to one decimal place.

7 Repeatability and reproducibility

An international collaborative study involving 17 laboratories and 2 samples conducted in 1993 showed that when pure nicotine and a degraded sample of the pure nicotine were analysed by this method, the following values for the repeatability limit (r) and the reproducibility limit (R) were obtained.

The difference between two test results found on different analyses by one operator using the same apparatus within a short time interval will exceed the repeatability limit (r) on average not more than once in 20 cases in the normal and correct operation of the method.

Single test results reported by two laboratories will differ by more than the reproducibility limit (R) on average not more than once in 20 cases in the normal and correct operation of the method.

Data analysis gave the estimates summarized in table 1.

Table 1 - Estimates given by data analysis

Nicotine type	Mean purity of nicotine %	Repeatability limit *r*	Reproducibility limit *R*
Pure (> 99 %)	98,8	2,2	3,8
Degraded	96,7	1,6	3,2

For the purpose of calculating *r* and *R*, one test result was defined as the yield obtained from analysing one sample once.

NOTE 3 Estimation of *R* based on average of five single determinations gave the following values: 2,9 for degraded nicotine and 3,3 for pure nicotine.

8 Test report

The test report shall show the method used and the result obtained. 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 affected the result.

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

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Annex A
(informative)

Bibliography

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

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