
**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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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 13276:2017), which has been technically revised. The main changes compared to the previous edition are as follows:

- the Warning notice has been updated;
- the error in [Formula \(1\)](#) in [Clause 8](#) has been corrected.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of any other restrictions prior to use.

1 Scope

This document 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.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <http://www.electropedia.org/>

4 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.

5 Reagents

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

5.1 Tungstosilicic acid solution (CAS of tungstosilicic acid: 12027-43-9).

Dissolve 12 g of dodeca-tungstosilicic acid ($\text{H}_4[\text{Si}(\text{W}_3\text{O}_{10})_4] \cdot x\text{H}_2\text{O}$) in 100 ml of water.

Avoid the use of the other forms of tungstosilicic acid such as $4\text{H}_2\text{O} \cdot \text{SiO}_2 \cdot 10\text{WO}_3 \cdot 3\text{H}_2\text{O}$ or $4\text{H}_2\text{O} \cdot \text{SiO}_2 \cdot 12\text{WO}_3 \cdot 20\text{H}_2\text{O}$ as they do not yield crystalline precipitates with nicotine. Tungstosilicic acid of the CAS 11130-20-4 and 12027-38-2 may be used for this method provided sufficiently crystalline precipitation occurs.

5.2 Hydrochloric acid solution, HCl, a volume fraction of 20 %.

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

5.3 Hydrochloric acid solution, HCl, a volume fraction of 0,1 %.

Dilute 5 ml of hydrochloric acid solution (5.2) to 1 l with water.

5.4 Nicotine solution, $\rho(\text{C}_{10}\text{H}_{14}\text{N}_2) = 0,1$ mg/ml.

Dissolve 2,5 mg of nicotine ($\text{C}_{10}\text{H}_{14}\text{N}_2$) in water using a volumetric flask (6.1) and dilute to 25 ml with water.

6 Apparatus

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

6.1 Volumetric flask, of 25 ml capacity.

6.2 Beakers, of 250 ml capacity.

6.3 Watch glasses.

6.4 Glass stirring rods.

6.5 Desiccator, containing an effective drying agent.

6.6 Apparatus for glass filter filtration procedure.

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

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6.6.2 Filter flask (Buchner flask).

6.6.3 Vacuum source.

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

6.7 Apparatus for filter paper filtration procedure.

6.7.1 Ashless filter paper.¹⁾

6.7.2 Porcelain or platinum crucibles.

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

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

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

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 document and does not constitute an endorsement by ISO of this product.