
**Water pipe tobacco — Determination
of total collected matter and nicotine
using a water pipe tobacco smoking
machine**

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

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.

Introduction

Tobacco smoke is a complex mixture consisting of many individual chemical constituents. These compounds exist as gases, vapours and condensed aerosol particles. Additionally, various rapid ageing processes, together with diffusional and intersolubility effects, start occurring immediately after the formation of the smoke which further complicates its composition. These processes and effects are particularly relevant to water pipe tobacco smoke where the smoke ages and passes through a water trap before it reaches the smoker.

Historically, when tobacco products are smoked in a laboratory setting, the particulate matter in smoke is collected on a glass fibre filter and this approach has been followed in this document for water pipe tobacco smoking.

The parameters used for “puffing” on the laboratory water pipe used in this document are based on published studies of human behaviour and data reported to the ISO/TC 126. It is convenient to use the term “puffing”, however it is, in strict physiological terms, incorrect. Smokers of cigarettes and many other tobacco products use a two-step process to draw the smoke from the product into the mouth (the puff), followed usually by inhalation of ambient air into the lungs through either the nose or mouth. Smokers of water pipes use a one-step process to inhale smoke directly into the lungs.

It is important to note that no machine smoking regime can represent all human smoking behaviour:

- machine smoking testing is useful to characterize water pipe tobacco emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;
- smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

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Water pipe tobacco — Determination of total collected matter and nicotine using a water pipe tobacco smoking machine

1 Scope

This document specifies methods for the determination of total collected matter and for the subsequent determination of nicotine present in the smoke from water pipe tobacco generated and collected using a water pipe tobacco smoking machine.

This document is only applicable for devices known as “arghile”, “hookah”, “nargile” or “shisha” in which tobacco is only heated, not combusted. Other types, such as “Chinese water pipe”, are not covered.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

ISO 10315, *Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method*

ISO 22486, *Water pipe tobacco smoking machine — Definitions and standard conditions*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/>

3.1

total collected matter

TCM

portion of the mainstream smoke which is trapped in the *smoke trap* (3.2), expressed as milligrams

3.2

smoke trap

device for collecting such parts of the smoke from a sample as is necessary for determination of specified smoke components

[SOURCE: ISO 3308:2012, 3.15]

3.3

smoking process

use of a smoking machine to smoke the water pipe tobacco from first to final puff

3.4

smoking run

specific *smoking process* (3.3) to produce such smoke from a sample of water pipe tobacco as is necessary for the determination of the smoke components

3.5

test portion

water pipe tobacco prepared for a single determination

4 Principle

The water pipe tobacco is sampled and then smoked on a water pipe tobacco smoking machine with collection of total collected matter in a glass fibre filter trap. The mass of the total collected matter so collected is determined gravimetrically. The total collected matter is extracted from the trap for determination of nicotine content by gas chromatography.

In laboratories that are not in a position to use gas-chromatographic methods, reference should be made to ISO 3400 for the determination of total nicotine alkaloids. In such cases, values obtained for nicotine in smoke condensate may be used with the addition of a note made in the expression of the result.

5 Apparatus

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

5.1 Routine analytical water pipe tobacco smoking machine, complying with the requirements of ISO 22486.

5.2 Soap bubble meter, graduated at 530 ml to an accuracy of ± 5 ml and with a resolution of 5 ml.

5.3 Apparatus for the determination of puff duration and frequency.

5.4 Analytical balance, suitable for measuring to the nearest 0,1 mg.

The weighing of filter holders may be affected by static electricity, necessitating the use of an antistatic device.

5.5 Conditioning enclosure, carefully maintained under the conditions specified in ISO 3402.

5.6 Smoke trap sealing device, end caps made from a non-hygroscopic and chemically inert material.

5.7 Gloves, made of cotton, or the non-talc surgical type.

6 Sampling

A representative 300 g sample shall be taken from the lot received by the laboratory for testing.

This representative sample will normally contain water pipe tobacco taken from different parts of the population. Make up the test portion (3.5) required for the test by randomly selecting the water pipe tobacco from the different parts of the population represented in the representative sample.

7 Determination of total collected matter

7.1 Preparation of the water pipe tobacco for smoking

7.1.1 General

Mix the representative sample until homogeneous before the test portions are taken. Fill a loose portion of the water pipe tobacco sample into the tobacco sample holder and ensure that the surface of the water pipe tobacco sample and the upper surface of the tobacco holder are equal without pressing the tobacco. Weigh the used water pipe tobacco and note the weight in the test report.

7.1.2 Replicate test portions

A minimum of three independent replicate determinations should be undertaken per water pipe tobacco.

However, the user should define the number of replicates based upon the final use of the data.

7.2 Storage and conditioning

Water pipe tobacco for testing should be conditioned for at least 12 h at a temperature of $22\text{ °C} \pm 3\text{ °C}$ in vapour tight containers just large enough to contain the sample, until smoke run preparation.

Once opened, the tobacco should be stored at room temperature in vapour tight containers just large enough to contain the sample to avoid the loss of volatile constituents and prevent the building of mould.

The testing atmosphere in the laboratory where the smoking is to be carried out shall be in accordance with ISO 3402.

7.3 Preliminary measurement before smoking

Measuring the mass of the conditioned water pipe tobacco selected for the smoking operation (in grams per portion) is required before smoking by first taking the weight of the unfilled tobacco holder and calculate the weight in difference to the filled tobacco holder.

7.4 Smoking and collection of collected matter

7.4.1 Preparation of smoke traps

For all operations, the operator shall prevent contamination from the fingers by wearing gloves of a suitable material (5.7).

Insert glass fibre filter, which have been conditioned in the test atmosphere for at least 12 h into the holder, and assemble, placing the rough side of the glass fibre filter so that it will face the oncoming smoke. After assembly, examine the filter holders to ensure that the glass fibre filters have been properly fitted. Fit the sealing devices (end caps) (5.6). Weigh the assembled smoke traps to the nearest 0,1 mg.

If necessary, prepare a sample blank by treating an additional smoke trap (at least one per batch/session/day) in the same manner as that used for smoke collection by drawing 35 puffs without tobacco in the water pipe tobacco holder.

7.4.2 Setting up the smoking machine

7.4.2.1 General

If necessary, replace any protective filters on the machine. Switch on the machine and allow it to warm up on automatic cycling for at least 20 min.

After the machine is warmed up, check that the puff duration and puff frequency are in accordance with the standard conditions. The puff volume should be checked daily.

7.4.2.2 Measurement of puff duration

A timer shall be used to measure the period of time which elapses between the triggering operations which begin and end a puffing action of the smoking machine. The accuracy of the timing device shall be such as to ensure that a 1 % error in the puff duration can be detected. The timer should be coupled directly to the triggering circuits.

NOTE It is not possible to specify the method of measurement beyond a statement of principle because of the variety of types of suitable timers and smoking machines available.

7.4.2.3 Checking of puff frequency

Measure the period of time which elapses between the triggering operations which begin successive puffing actions of the smoking machine, thus determining the puff frequency. The timer used shall be suitable for measuring to the nearest 0,1 s and should, preferably, be coupled directly to the triggering circuits.

7.4.2.4 Measurement of puff volume

The displacement of the bubble in a soap bubble meter (5.2) gives a direct measurement of puff volume and also provides a check for leaks in the system. A suitable indicator graduated at 530 ml shall have a resolution of 5 ml. It shall be connected to the suction tube of the water pipe after removing the head of the water pipe. Before use for a series of measurements, wet the instrument twice with detergent solution and then allow it to drain for a period of between 30 s and 45 s.

It is recommended to use the detergent solution as specified by the supplier of the soap bubble flow meter in the corresponding manual.

Fit the prepared smoking trap onto the machine. Prepare the soap bubble flow meter by wetting the inside of the tube with the detergent solution to above the top graduation mark. Connect the bubble meter to the holder and determine the puff volume; adjust if necessary to (530 ± 10) ml.

Repeat the determinations until the necessary precision of measurement is obtained. If the number of replicates exceeds three, continue until the correct precision is obtained but replace the glass fibre filter before smoking, reweigh the smoke trap and recheck the puff volume with the new glass fibre filter in place. Measure and record the temperature and relative humidity of the air surrounding the smoking machine and note the atmospheric pressure.

7.4.3 Procedure for smoking run

Prepare the water pipe according to ISO 22486. Adjust the suction tube equivalent to the water level $(30 \text{ mm} \pm 1 \text{ mm})$ insertion depth). Fill the bottle with 750 ml water of at least quality grade 3 according to ISO 3696.

Fill the head up to the top with the water pipe tobacco to be tested. Weigh the amount of water pipe tobacco used.

Place the water pipe tobacco holder into the head and ensure that the tobacco will be level with the top of the head. Connect the water pipe to the filter holder. Avoid any leaks.

Ensure the heating device has reached the desired operating temperature.

Set the puff counter to zero and place the preheated heating device on the water pipe tobacco holder. Wait 5 min and then take 175 puffs according to the conditions described in ISO 22486. The filter holder

including the glass fibre filter should be replaced every 35 puffs without interfering with the smoking process.

NOTE A clearing puff, i.e. an additional puff after removal of the water pipe tobacco and heating device, is not necessary due to the minimum contribution of a clearing puff to the total amount of 175 puffs.

After completing the smoking process, leave the water pipe hose in place for at least 30 s to enable deposition of any residual smoke in the trap.

7.5 Determination of total collected matter

Remove the smoke trap and cover the front and back apertures of the trap with the sealing devices (5.6).

Immediately after smoking, weigh the smoke trap to the nearest 0,1 mg.

7.6 Calculation of total collected matter

The TCM content, m_{TCM} , for each smoke trap, expressed in milligrams, is given by Formula (1):

$$m_{\text{TCM}} = m_1 - m_0 \quad (1)$$

where

m_0 is the mass of the smoke trap before smoking, in milligrams;

m_1 is the mass of the smoke trap after smoking, in milligrams.

The TCM content for each test portion, expressed in milligrams, is given by Formula (2):

$$TCM_{\text{tot}} = \sum_{i=1}^n m_{\text{TCMi}} \quad (2)$$

The TCM content may also be expressed as milligrams per gram water pipe tobacco placed in the tobacco holder. This can be calculated by Formula (3):

$$m_{\text{TCM}} = \frac{TCM_{\text{tot}}}{m_{\text{tobacco}}} \quad (3)$$

where m_{tobacco} is the mass of the water pipe tobacco placed in the tobacco holder, in grams.

7.7 Handling of glass fibre filter

Remove the sealing devices from the smoke trap (gloves shall be worn). Open it and remove the glass fibre filter with forceps. Fold it twice, total collected matter inwards, being careful to handle only the edge with forceps and gloved fingers. Place the folded glass fibre filter in an appropriately shaped 500 ml dry flask. Wipe the inner surface of the filter holder front with two separate quarters of an unused conditioned glass fibre filter and add these to the flask. Repeat this for the rear part of the filter holder with two further quarters of an unused conditioned glass fibre filter and add these to the flask. Each smoking run will produce a further four glass fibre filters and a further 16 quarter glass fibre filters which should all be added to the same flask.

7.8 Determination of nicotine

Pipette 250 ml solvent (propan-2-ol containing the internal standard for nicotine determination) into the flask (see ISO 10315).

Stopper the flask immediately and shake gently on an electric shaker for at least 10 min, ensuring that the glass fibre filters do not disintegrate. The shaking time should be adjusted to ensure full extraction of the nicotine.

If used, follow the same procedure with the blank smoke trap.

Carry out the determination of nicotine in the solution in each flask in accordance with ISO 10315.

Adjust the concentration of the calibration curve accordingly, if needed.

8 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this document, or regarded as optional, as well as any circumstances that may have influenced the results. The test report shall include all details required for complete identification of the sample. If appropriate, the information given in a) to d) shall be recorded.

a) Characteristic data about the water pipe tobacco

All details necessary for the identification of the water pipe tobacco smoked shall be given. In the case of commercial water pipe tobacco this should include:

- name of manufacturer and country of manufacture;
- water pipe tobacco name;
- packet number (of the water pipe tobacco sampled that day) (if any);
- marks on any tax stamp (if any);
- printed smoke yields (if any);
- digital photograph of the packet.

b) Data about sampling

- type of sampling procedure;
- date of sampling;
- place of purchase or sampling;
- kind of sampling point;
- sampling point (e.g. address of retail outlet or machine number);
- number of portions in the laboratory sample.

c) Description of test

- a reference to this document, i.e. ISO/TS 22487:2019;
- date of test;
- type of smoking machine used;
- type of smoke trap used;
- total number of test portions smoked;
- room temperature [in degrees Celsius (°C)] during smoking operation;
- relative humidity [in percent (%)] during smoking operation;