
**Water pipe tobacco products —
Determination of carbon monoxide
emission of glowing water pipe
charcoal — NDIR method**

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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).

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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*.

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

For the testing of water pipe tobacco a routine analytical water pipe tobacco smoking machine is used, heating the water pipe tobacco with an electrical heater. This is done to prevent contamination of the collected phase by the emission of glowing water pipe charcoal. Nevertheless, most of the users use glowing water pipe charcoal to heat up the water pipe tobacco for smoking.

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Water pipe tobacco products — Determination of carbon monoxide emission of glowing water pipe charcoal — NDIR method

1 Scope

This document specifies a method for the determination of carbon monoxide (CO) emission of glowing water pipe charcoal.

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

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

ISO/TS 22487, *Water pipe tobacco — Determination of total collected matter and nicotine*

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

vapour phase

portion of gas, which passes the glowing charcoal and the water bottle during smoking in accordance with ISO/TS 22487 using a machine conforming to ISO 22486

4 Principle

Light up a sample of charcoal for water pipe smoking, place it in the sample holder of a routine analytical water pipe tobacco smoking machine and take puffs in accordance with the procedures given in ISO/TS 22487. Collection of the vapour phase and measurement of the carbon monoxide using a non-dispersive infrared (NDIR) analyser calibrated for carbon monoxide. Calculation of the amount of carbon monoxide per sample.

5 Apparatus

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

5.1 Conditioning enclosure, maintained accurately in accordance with the conditions specified in ISO 3402, for conditioning the cigarette sample prior to smoking (see also 7.1).

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

5.3 Gas-phase collection system, which can be fitted to the water pipe tobacco smoking machine. The use of the system shall ensure collection of all the generated vapour phase to be stored in a previously evacuated container for subsequent sampling through an NDIR analyser.

The collection system shall not cause interference with the normal performance of the smoking machine.

The impermeability of the vapour phase collecting device to a vapour phase shall be checked with a vapour phase containing a volume fraction of 4 % to 6 % of CO. The CO concentration shall be measured directly after filling the previously evacuated gas-collecting device. After a period of not less than 2 h, the measured value of CO concentration in the vapour phase in the device shall not differ by more than a volume fraction of 0,2 % from the value expected from the first determination.

When a bag is used as the gas-collecting device, it shall be large enough to avoid the final pressure of its contents exceeding the ambient atmospheric pressure. The volume of the bag should also be no greater than twice the volume of the gas content collected at atmospheric pressure. In practice, the collection of the vapour phase from 175 puff requires a bag volume of 120 l to 185 l.

It can be inconvenient to collect all of the vapour phase generated from a single smoked test portion in one single 120 l bag. Other possibilities exist and may be considered for inclusion in this document.

- a) Use two or more smaller bags, which are changed at the same time as the TCM collection pad is changed after every 35 puff. The practical bag size for this option is roughly 30 l; at least two bags are required. Both should be evacuated prior to commencement of the smoking process. The first bag is filled during the first 35 puffs, then removed for analysis and re-evacuated while the next bag is in use and so on. A modified version of [Formulae \(1\) and \(2\)](#) given in [Clause 8](#) is required in order to combine the partial vapour phase concentrations measured during each bag fill.
- b) Use a constant flow gas splitting system to deliver a known fraction of the total vapour phase to an appropriate-sized collection bag. A ratio 20:1 splitting system requires a 10 l bag (connected to the low flow output of the splitter) to collect the vapour phase output for a complete smoked test portion. The vapour phase from the high flow output of the splitter is routed directly to the waste smoke exhaust system. The contents of the collection bag are then analysed in the normal way. The relative volumes of the split sample are not required; [Formula \(1\)](#) or [Formula \(2\)](#) in [Clause 8](#) only needs the total volume which is the puff volume time the number of puffs. This system works correctly provided that the vapour phase sample is homogeneous at the entrance to the splitter and that the split flows remain at a constant ration throughout the smoking process.
- c) The vapour phase for a single puff only is collected, analysed and disposed of on a puff by puff basis. The CO is calculated on the basis of mg per puff and the total CO per sample is the sum of the mass for all puffs.

5.4 Non-dispersive infrared (NDIR) analyser, selective and calibrated for the measurement of carbon monoxide in vapours.

Analysers are available from several manufacturers and should have a suitable measurement range. The sampling rate should be between 0,5 l/min and 5 l/min. The analyser shall have a linearity of 0,1 % CO and a repeatability of 0,2 % CO, under conditions of constant temperature and pressure. In terms of volume fractions its response to 10 % CO₂ shall not exceed 0,05 % as CO. Its response to 2 % water vapour shall not exceed 0,05 % as CO.

5.5 Flame or heating device, capable to ignite the charcoal.

5.6 Barometer, capable of measuring atmospheric pressures to the nearest 0,1 kPa.

5.7 Thermometer, capable of measuring temperature to the nearest 0,1 °C.

6 Standard gas mixtures

Make-up gas shall be nitrogen as other gases can change the detected response of carbon monoxide. Gases used should be of high purity (with low content of carbon dioxide) and used within the manufacturer's time limits.

The NDIR analyser should be calibrated with at least three standard gas mixtures of accurately known concentrations within a relative error of 2 %, covering the expected range in such a way as to avoid extrapolation of the calibration curve. Typically used concentrations are approximately 25 %, 50 % and 75 % of the analyser's measurement range.

7 Procedure

7.1 Conditioning

Condition the test portion taken from and representative of the laboratory sample in accordance with ISO 3402. Verify that equilibrium has been properly attained as described in ISO 3402.

The atmosphere in the laboratory where the smoking is to be carried out shall also be in accordance with ISO 3402. Place the conditioned test portion in a vapour-tight container (just large enough to contain the portion) and remove from the container just before smoking.

7.2 Calibration of the NDIR analyser

7.2.1 Warm up the instrument according to the manufacturer's recommendations, purge the instrument with air and adjust to read zero.

7.2.2 Fill a previously evacuated vapour phase collection container with the standard gas mixture of a known volume fraction, re-evacuate and refill with gas. Ensure that the gas in the container is at ambient temperature and pressure. Introduce the gas into the measuring cell using the system sampling pump allowing 5 s to 10 s for equilibration of pressure of the analyser. Note the reading on the analyser concentration display when a steady value has been obtained.

If necessary, adjust the analyser reading to agree with the certified value of the standard gas.

7.2.3 Repeat the procedure as specified in [7.2.2](#) for at least two other standard gas mixtures. If there is a difference of greater than a volume fraction of 0,2 % CO between the observed and expected values, attention should be given to the analyser linearity.

7.2.4 Recalibrate the instrument at least once a week, using the standard gases. The calibration shall be linear within the limits reported in [5.4](#).

7.2.5 Check the calibration prior to the measurement using the same standard gas used under [7.2.2](#). If there is a difference greater than a volume fraction of 0,2 % CO between observed and expected values, repeat the full calibration.

7.3 Smoking and collection of vapour phase

7.3.1 Preparation of vapour phase collection system

Prepare the system using the instructions pertinent to the equipment fitted.

Ensure that the vapour phase collecting device has been completely flushed with ambient air and evacuated before the start of the smoking process. There shall not be any residual vacuum upstream of the collection device before puffing.

7.3.2 Selection of the charcoal

Randomly select 10 pieces of charcoal from the conditioned portion. Weigh the samples to at least 0,1 g and calculate the average. Select three samples with the weight closest to the average. Note the average weight as well as the individual weights.

7.3.3 Preparation of the routine analytical water pipe tobacco smoking machine

Set up the routine analytical water pipe tobacco smoking machine in accordance to ISO 22486 without use of any water pipe tobacco and the electrical heating device.

7.3.4 Testing procedure

Ignite the charcoal sample to be tested following the manufacturers recommendations. Alternatively, place the charcoal on an electric stove or, in the case of quick light charcoal, light the charcoal holding it with pliers and wait until any sparking has fully finished. No fire starters shall be used in any case.

Wait until the sample is homogeneously glowing. Place the sample into the holder of the water pipe. Take 175 puffs with regards to ISO/TS 22487. Collect the vapour phase (see 5.3). After 60 puffs add the second glowing charcoal. After 120 puffs replace the first charcoal by the third glowing charcoal.

It is recommended to register the time needed from lighting to homogeneously glowing when preparing the first charcoal. This time is needed in advance for the preparation of the second and third charcoal before adding to the process.

7.4 Measurement of carbon monoxide volume concentration

7.4.1 Recheck the calibration of the analyser (see 7.2.5) and introduce the vapour phase into the measuring cell of the analyser under the same conditions of ambient temperature and pressure as for sampling and the same gas flow rate as used during calibration. Read the analyser display giving the carbon monoxide concentration. Recalibration can be necessary when the barometric pressure has changed for more than 10 kPa and the CO analyser has no internal compensation.

7.4.2 At the end of each smoking, the vapour phase collection container shall be emptied. The apparatus is then ready for the next smoking starting at step 7.3.2.

8 Expression of results

8.1 Calculation of the average emission of carbon monoxide per piece of charcoal

The average volume of carbon monoxide per piece of charcoal is given by [Formula \(1\)](#):

$$V_{as} = \frac{C \times V \times N \times p \times T_0}{100 \times p_0 \times (t + T_0) \times 3} \quad (1)$$

where

V_{as} is the average volume of carbon monoxide per piece of charcoal, in millilitres;

C is the percentage by volume of carbon monoxide observed;

V is the puff volume, in millilitres;

- N is the total number of puffs (i.e. 175);
- p is the ambient pressure, in kilopascals;
- p_0 is the standard atmospheric pressure, in kilopascals;
- T_0 is the temperature for the triple point of water, in Kelvin;
- t is the ambient temperature, in degrees Celsius;
- 3 is the number of charcoal pieces used.

In the calculation the following values can be used: $V = 530$ ml and rounded values of p_0 (101,3 kPa) and T_0 (273 K).

8.2 Calculation of the average emission of carbon monoxide per puff

The average volume of carbon monoxide per puff is given by [Formula \(2\)](#):

$$V_{as} = \frac{C \times V \times N \times p \times T_0}{100 \times p_0 \times (t + T_0) \times 175} \quad (2)$$

where

- V_{as} is the average volume of carbon monoxide per puff, in millilitres;
- C is the percentage by volume of carbon monoxide observed;
- V is the puff volume, in millilitres;
- N is the total number of puffs (i.e. 175);
- p is the ambient pressure, in kilopascals;
- p_0 is the standard atmospheric pressure, in kilopascals;
- T_0 is the temperature for the triple point of water, in Kelvin;
- t is the ambient temperature, in degrees Celsius;
- 175 is the number of puffs taken.

In the calculation the following values can be used:

$V = 530$ ml and rounded values of p_0 (101,3 kPa) and T_0 (273 K).

8.3 Calculation of the average mass of carbon monoxide per piece of charcoal

The average mass of carbon monoxide per sample portion is given by [Formula \(3\)](#):

$$m = V_{as} \times \frac{M_{CO}}{V_m} \quad (3)$$

where

- m is the average mass of carbon monoxide per piece of charcoal, in milligrams;
- M_{CO} is the molar mass of carbon monoxide, in grams per mole;
- V_m is the molar volume of an ideal gas, in litres per mole.

In the calculation the following values can be used: Rounded values of M_{CO} (28 g/mol) and V_{m} (22,4 l/mol).

8.4 Calculation of the average mass of carbon monoxide per puff

The average mass of carbon monoxide per sample portion is given by [Formula \(4\)](#):

$$m = V_{\text{as}} \times \frac{M_{\text{CO}}}{V_{\text{m}}} \quad (4)$$

where

m is the average mass of carbon monoxide per puff, in milligrams;

M_{CO} is the molar mass of carbon monoxide, in grams per mole;

V_{m} is the molar volume of an ideal gas, in litres per mole.

In the calculation the following values can be used: Rounded values of M_{CO} (28 g/mol) and V_{m} (22,4 l/mol).

9 Repeatability and reproducibility

An interlaboratory study is required in order to provide an estimate of the repeatability and reproducibility of this method.

10 Test report

10.1 General

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 listed in [10.2](#) to [10.5](#) shall be recorded.

10.2 Characteristic data about the charcoal sample and identification

All necessary details to describe the sample fully such as:

- a) name of manufacturer;
- b) country of manufacture;
- c) charcoal brand name;
- d) date of sampling;
- e) place of purchase or sampling;
- f) kind of sampling point;
- g) sampling point (e.g. address of retail outlet or machine number);
- h) packet number (of the charcoal sampled that day);
- i) mass of contents, if available;
- j) carbon content as indicated on the packaging;
- k) other additives.