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Water pipe tobacco — Determination of carbon monoxide in the vapour phase of water pipe tobacco smoke — NDIR method

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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*.

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.

Water pipe tobacco — Determination of carbon monoxide in the vapour phase of water pipe tobacco smoke — NDIR method

1 Scope

This document specifies a method for the determination of carbon monoxide (CO) in the vapour phase of water pipe tobacco smoke.

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 using a water pipe tobacco smoking machine*

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 smoke, which passes the smoke trap during smoking in accordance with ISO/TS 22487 using a machine conforming to ISO 22486

4 Principle

Smoking of water pipe tobacco in accordance with the procedures given in ISO/TS 22487. Collection of the vapour phase of the water pipe tobacco smoke 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 water pipe tobacco test portion.

5 Apparatus

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

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

5.2 Vapour-phase collection system, which can be fitted to the water pipe smoking machine. The use of the system shall ensure collection of all the vapour phase (normally vented to atmosphere) 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 and the consequent determination of total particulate matter and nicotine.

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 vapour phase-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 vapour phase-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 vapour phase 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 are 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 are 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; the 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.

5.3 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 precision of 0,1 % CO, 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 % CO_2 shall not exceed 0,05 % as CO. Its response to 2 % water vapour shall not exceed 0,05 % as CO.

5.4 Heating device, effecting flameless electric heating, as defined in ISO 22486.

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

5.6 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 Storage and conditioning

Water pipe tobacco for testing should be conditioned for at least 12 h at a temperature of 22 °C ± 3 °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.

7.2 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 5.3.

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 smoking.

7.3.2 Smoking procedure

7.3.2.1 Smoke the water pipe tobacco in accordance with the procedure stated in ISO/TS 22487.

7.3.2.2 After completion of smoking remove the residual tobacco portion and take 2 clearing puffs.

7.3.2.3 Record the total number of puffs taken, i.e. smoking puffs plus clearing puffs.

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 flow rate as used during calibration. Read the analyser display giving the carbon monoxide concentration. Recalibration may 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 7.3.2.1.

8 Expression of results

8.1 Calculation of the average volume of carbon monoxide per water pipe tobacco portion

The average volume of carbon monoxide per tobacco portion is given by Formula (1):

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

where

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

C is the percentage by volume of carbon monoxide observed;

V is the puff volume, in millilitres;

N is the number of puffs in the measured test portion (including clearing puffs);

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.

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 mass of carbon monoxide per water pipe tobacco portion

The average mass of carbon monoxide per test portion is given by Formula (2):

$$m = V_{as} \times \frac{M_{CO}}{V_m} \quad (2)$$

where

m is the average mass of carbon monoxide per test portion, 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 water pipe tobacco sample and identification

All necessary details to describe the sample fully such as:

a) name of manufacturer;

b) country of manufacture;

c) water pipe tobacco 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 water pipe tobacco sampled that day);

i) marks on any tax stamp;

j) printed smoke yields (if any).