International Standard

ISO 8454

Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke — NDIR method

Cigarettes — Dosage du monoxyde de carbone dans la phase gazeuse de la fumée de cigarette — Méthode IRND

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Contents

Page

Foreword

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

This fourth edition cancels and replaces the third edition (ISO 8454:2007), which has been technically revised. It also incorporates the Amendment(s) ISO 8454:2007/Amd. 1:2019 and ISO 8454:2007/Amd. 2:2019.

The main changes are as follows:

— the scope was edited to improve clarity;

— the repeatability and reproducibility values in Table 1 were updated to include those from ISO/TR 19478 and the total particulate matter values were included;

— Clause 10, Test Report, was harmonized with ISO 10315;

— subclauses 7.2.2 and 7.2.3 were edited to better describe the calibration and verification processes.

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

No machine smoking regime can represent all human smoking behaviour:

— it is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this document;

— machine smoking testing is useful to characterize cigarette 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.

Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke — NDIR method

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 determination of carbon monoxide (CO) in the vapour phase of mainstream cigarette smoke collected with the smoking regime specified in ISO 4387.

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 3308, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

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

ISO 4387, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine*

3 Terms and definitions

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

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

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

— IEC Electropedia: available at https://www.electropedia.org/

3.1
vapour phase
portion of smoke, which passes the particulate phase trap during smoking in accordance with ISO 4387 using a machine conforming to ISO 3308

3.2
clearing puff
any puff taken after a cigarette has been extinguished or removed from the cigarette holder

4 Principle

Smoking of cigarettes in accordance with the procedures given in ISO 4387. Collection of the vapour phase of the cigarette 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 cigarette.

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 cigarette-smoking machine and accessories, conforming with the requirements of ISO 3308.

5.3 Vapour-phase collection container, (i.e. gas collection bag) which is used for collection of the vapour phase. The impermeability of the vapour-phase collection container shall be checked with a standard gas mixture containing a volume fraction of 4 % to 6 % of CO. To check the impermeability of the vapour-phase collection container, the CO concentration shall be measured immediately after filling the previously evacuated vapour-phase collection container and a second time after a period of not less than 2 h. The measured value of the CO concentration shall not differ by more than a volume fraction of 0,2 % from the first and second determinations.

The vapour-phase collection container shall be large enough to avoid the final pressure of its contents exceeding the ambient atmospheric pressure. The volume of the vapour-phase collection container 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 5 cigarettes requires a volume of 3 l and the collection of the vapour phase from 20 cigarettes requires a volume of 10 l.

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

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

5.5 Vapour-phase collection system, which can be fitted to one or more of the smoking machine channels. The vapour-phase collection system consists of the apparatus for directing the vapour phase to the vapour-phase collection container (5.3) and the NDIR analyser (5.4). 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.

5.6 Ignition device, effecting flameless ignition. Experience has shown that the lighting process can influence the CO yield considerably. The lighters should preferably light the cigarettes at the first attempt without either touching or pre-charring the cigarettes. The CO yields are increased by higher lighting intensity.

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

5.8 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 timeframe stated in the manufacturer's certificate of analysis.

The NDIR analyser should be calibrated with a high-range standard gas mixture and then verified with at least two lower concentration standard gas mixtures. All as mixtures should have accurately known concentrations within a relative error of 2 % covering the expected range of CO in the samples. Typically, volume fractions for low-range, mid-range and high-range standard gas mixture are appropriate about 1 %, 3 % and 5 % of CO in nitrogen, respectively.

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 an airtight container (just large enough to contain the portion) and remove each cigarette 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 Calibration for the NDIR analyser: Fill a previously evacuated vapour-phase collection container with the high-range standard gas mixture, 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.

Where the standard gas mixture is a volume fraction of certified 5,0 % CO, the maximum allowed range for the observed value is 4,8 % to 5,2 %.

7.2.3 Verify the calibration of NDIR analyser: Using at least two other standard gas mixtures verify the linearity of the calibration. Fill a previously evacuated vapour-phase collection container with the mid-range standard gas mixture, 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 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. Repeat with the low-range gas mixture.

Where the standard gas mixture is a volume fraction of certified 1,0 % CO, the maximum allowed range for the observed value is 0,8 % to 1,2 %.

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 high-range standard gas. 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 collection container has been completely flushed with ambient air and evacuated before the start of the smoking process. The pressure between the puffing engine and the vapour-phase collection container shall be at atmospheric pressure prior to smoking.

7.3.2 Smoking procedure

7.3.2.1 Smoke the cigarettes in accordance with ISO 4387 and collect the vapour phase for subsequent analysis.

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. Record the result of carbon monoxide concentration displayed on the analyser. Recalibration may be necessary when the barometric pressure has changed for more than 10 kPa and the NDIR analyser has no internal compensation.

7.4.2 At the end of each smoking, the vapour-phase collection container shall be emptied. The pressure between the puffing engine and the vapour-phase collection container shall be at atmospheric pressure prior to smoking. 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 cigarette

The average volume of carbon monoxide per cigarette, V_{as}, expressed in millilitres, is given by the Formula (1):

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

where

V_{as} is the volume of carbon monoxide per cigarette, 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 sample (including clearing puffs);

p is the ambient pressure, in kilopascals;

p_0 is the standard atmospheric pressure, in kilopascals;

S is the number of cigarettes smoked;

T_0 is the temperature for the triple point of water, in Kelvin;

t is the ambient temperature, in degrees centigrade.

In the calculation, the following values can be used:

$V = 35\,\text{ml}$ 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 cigarette

The average mass of carbon monoxide per cigarette, m_{cig}, expressed in milligrams, is given by the Formula (2):

$$m_{\text{cig}} = V_{\text{as}} \times \frac{M_{\text{co}}}{V_{\text{m}}} \tag{2}$$

where

m_{cig} is the average mass of carbon monoxide per cigarette, 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

A major interlaboratory study involving 35 laboratories and 10 samples, conducted in 2010, showed that the resulting vapour phase smoke is successfully analysed by this method. The following values for the repeatability (r) and reproducibility limits (R) were obtained[4].

The difference between two single results found on matched cigarette samples by the same operator using the same apparatus within the shortest feasible time interval will exceed the repeatability, r, on average not more than once in 20 cases in the normal and correct operation of the method.

Single results on matched cigarette samples reported by two laboratories will differ by more than the reproducibility, R, on average not more than once in 20 cases in the normal and correct operation of the method.

The test results were subjected to statistical analysis according to ISO 5725-1 and ISO 5725-2 to give the precision data shown in Table 1.

Table 1 — Estimates given by data analysis

TPM **Mean value** m_{cig} mg per cigarette	**CO** **Mean value** m_{cig} mg per cigarette	**CO** **Repeatability limit** r mg per cigarette	**CO** **Reproducibility limit** R mg per cigarette
1,28	1,32	0,31	0,48
2,08	3,20	0,41	1,09
5,21	5,15	0,53	1,35
10,81	7,79	0,76	1,85
11,54	9,08	0,88	1,46
10,03	9,34	0,91	1,64
10,65	9,88	0,79	1,50

Table 1 *(continued)*

TPM **Mean value** m_{cig} mg per cigarette	**CO** **Mean value** m_{cig} mg per cigarette	**CO** **Repeatability limit** r mg per cigarette	**CO** **Reproducibility limit** R mg per cigarette
12,05	10,96	0,85	1,93
11,08	13,61	1,05	2,19
17,30	14,44	0,90	1,92

For the purpose of calculating r and R, one test result was defined as the mean yield obtained from smoking 20 cigarettes in a single run.

For further details of the interaction of *r* and *R* with other factors, see ISO/TR 19478-1.

10 Test report

The test report shall state the yield of carbon monoxide per cigarette smoked and the method used and shall include all conditions not stated in this document which might affect the result (e.g. atmospheric test conditions during smoking differ as required in 7.1 Conditioning). It shall also give all details necessary for the identification of the cigarettes smoked.

Bibliography

[1] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[2] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[3] ISO 8243, *Cigarettes — Sampling*

[4] ISO/TR 19478-1, *ISO and Health Canada intense smoking parameters — Part 1: Results of an international machine smoking study*

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