
Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke with an intense smoking regime — NDIR method

Cigarettes — Dosage du monoxyde de carbone dans la phase gazeuse de la fumée de cigarette obtenue avec un régime de fumage intense — Méthode IRND

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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).

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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

Historically, a set of ISO standards have been developed to specify the requirements of analytical cigarette smoking machines and their use for the quantitative determination of a number of cigarette smoke constituents (such as total particulate matter, nicotine-free dry particulate matter, water, nicotine or benzo[a]pyrene) with a unique standard smoking regime. The description of this smoking regime is provided in ISO 3308.

Later, requirements to provide smoke constituents data with an intense smoking regime, different from the ISO 3308 standard smoking regime, originated from different countries and the Conferences of the Parties to the Framework Convention on Tobacco Control, resulting in a need to specify the conditions for the use of the intense smoking regime on analytical cigarette-smoking machines. The specifications for the use of the intense smoking regime on analytical cigarette-smoking machines are provided in ISO 20778.

This document took into account practical work conducted in the framework of an interlaboratory study involving 35 laboratories (published as ISO/TR 19478-1 and ISO/TR 19478-2). It provides specifications for the determination of carbon monoxide in the vapour phase of cigarette smoke obtained with an intense smoking regime using NDIR method.

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.

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Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke with an intense smoking regime — 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 determination of carbon monoxide (CO) in the vapour phase of cigarette smoke with an intense smoking regime.

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 20778, *Cigarettes — Routine analytical cigarette smoking machine — Definitions and standard conditions with an intense smoking regime*

ISO 20779, *Cigarettes — Generation and collection of total particulate matter using a routine analytical smoking machine with an intense smoking regime*

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 particulate phase trap during smoking in accordance with ISO 20779 using a machine conforming to ISO 20778

[SOURCE: ISO 8454:2007, 3.1, modified — ISO 4387 and ISO 3308 have been replaced by ISO 20779 and ISO 20778 respectively.]

3.2 clearing puff

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

[SOURCE: ISO 20778:2018, 3.22]

4 Principle

Smoke generation in accordance with the procedures given in ISO 20779. 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, complying with the requirements of ISO 20778.

5.3 Vapour-phase collection system, which can be fitted to one or more of the smoking machine channels. 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 gas-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 3 cigarettes requires a volume of 3 l and the collection of the vapour phase from 10 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 % CO₂ shall not exceed 0,05 % as CO. Its response to 2 % water vapour shall not exceed 0,05 % as CO.

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

5.6 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 date stated on the manufacturer's certificate of analysis.

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, volume fractions of about 1 %, 3 % and 5 % of CO in nitrogen are appropriate.

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 Fill a previously evacuated vapour-phase collection container with the standard gas mixture of a volume fraction of about 5 % CO, 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 without adjusting the analyser readings. 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.

Where the standard gas mixture is a volume fraction of certified 1,00 % CO, the maximum allowed range for the observed value is 0,80 % to 1,20 %.

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 standard gas containing a volume fraction of about 5 % carbon monoxide. 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

Smoke the cigarettes in accordance with ISO 20779.

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 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.

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

- 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:

$V = 55$ 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 \(2\)](#):

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

where

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 when cigarettes are smoked in accordance with Health Canada Method T-115, which recommends an identical smoking regime as ISO 20779 does, and the resulting vapour phase smoke are analysed by this method, the following values for the repeatability limits (r) and the reproducibility limits (R) are obtained.

The difference between two single results found on matched cigarette samples by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit (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 differ by more than the reproducibility limit (R) on average not more than one in 20 cases in the normal and correct operation of the method.

The test results were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in [Table 1](#).

Table 1 — Estimates given by data analysis

Mean value m_{cig} mg per cigarette	Repeatability limit r mg per cigarette	Reproducibility limit R mg per cigarette
19,71	1,20	4,21
20,18	1,13	3,18
22,25	1,38	3,54
22,89	1,42	3,57
26,21	1,22	3,32
26,97	1,33	4,68
27,02	1,74	4,37
28,53	1,97	7,79
28,98	1,34	4,35
33,54	2,07	5,04

For the purpose of calculating r and R , one test result from a rotary machine was the mean of two runs smoking 10 test articles each and from a linear machine it was the mean of seven ports/channels, smoking three test articles per port/channel.

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

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 cigarette and cigarette identification

All necessary details to describe the sample fully such as:

- a) name of the manufacturer;
- b) country of the manufacture;
- c) product name;
- d) packet identifier (of that product sampled that day);
- e) marks on any tax stamp;
- f) printed smoke yields (if any);
- g) length of cigarette;
- h) length of filter;
- i) length of overwrap.

10.3 Sampling

All necessary details to describe the sampling fully such as:

- a) type of sampling procedure;
- b) number of cigarettes in laboratory sample;
- c) date of sampling;
- d) place of purchase or sampling;
- e) kind of sampling point;
- f) sampling point (e.g. address of retail outlet or machine number).

NOTE Sampling information as provided in ISO 8243.

10.4 Description of test

All necessary details to describe the test fully such as:

- a) a reference to this document, i.e. ISO 22947:2019;