
**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*



Reference number
ISO 8454:2007(E)

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Foreword

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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.

ISO 8454 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 8454:1995), which has been technically revised.

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

1 Scope

This International Standard specifies a method for the determination of carbon monoxide (CO) in the vapour phase of cigarette smoke.

2 Normative references

The following referenced documents are indispensable for the application 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.

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, complying with the requirements of ISO 3308.

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 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 % 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 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 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. 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 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

7.3.2.1 Smoke the cigarettes in accordance with the procedure stated in ISO 4387.

7.3.2.2 For linear smoking machines: after completion of smoking each of the first four cigarettes, remove the cigarette butt and take one clearing puff for each trap. After completion of the smoking of all five cigarettes five clearing puffs shall be taken.

7.3.2.3 For rotary smoking machines: after completion of the smoking run, remove the cigarette butts and take five clearing puffs.

7.3.2.4 Record the total number of puffs taken on each channel, 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 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 7.3.2.1.

8 Expression of results

8.1 Calculation of the average volume of carbon monoxide per cigarette

The average volume of carbon monoxide per cigarette is given by Equation (1):

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

where

- V_{as} is the average 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:

$V = 35$ 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 is given by Equation (2):

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

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

An international collaborative study [1] conducted in 2003, involving 58 laboratories and eight samples (seven commercial brands and the CORESTA monitor test piece CM4), gave the following values for this method.

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 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

Values in milligrams per cigarette

Mean value m_{cig}	Repeatability limit r	Reproducibility limit R
4,12	0,77	1,11
6,79	0,94	1,49
8,31	0,99	1,75
9,55	0,93	1,71
10,99	1,36	2,25
11,45	1,45	2,13
11,85	1,39	2,36
13,23	1,46	2,31

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.

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 International Standard 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 manufacturer;
- b) country of manufacture;
- c) product 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 that product sampled that day);
- i) marks on any tax stamp;
- j) printed smoke yields (if any);

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- k) length of cigarette;
- l) length of filter;
- m) 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 and location of purchase or sampling at manufacturers' premises.

10.4 Description of test

All necessary details to describe the test fully such as:

- a) reference to this International Standard, i.e. ISO 8454:2007;
- b) date of test;
- c) type of smoking machine used;
- d) type of analyser used;
- e) total number of cigarettes smoked in the entire determination on that cigarette type;
- f) number of cigarettes smoked into each collection device;
- g) butt length;
- h) room temperature ($^{\circ}\text{C}$) during smoking operation and analysis;
- i) relative humidity (%) during smoking operation;
- j) atmospheric pressure (kPa) during smoking operation and analysis.

10.5 Test results

The expression of the laboratory data depends on the purpose for which the data are required, and the level of laboratory precision. Confidence limits shall be calculated and expressed on the basis of the laboratory data before any rounding has taken place:

- average length of the cigarettes, average length of the filters, average length of the overwrap, average butt length to which the cigarettes were smoked, average length of tobacco portion smoked, all to the nearest 0,1 mm;
- average diameter of the cigarettes, in millimetres;
- average draw resistance of the conditioned cigarettes;
- average mass, in milligrams per cigarette, of the conditioned cigarettes selected for the smoking operation;
- average number of puffs per cigarette for each channel, to the nearest 0,1 puff;
- average number of total puffs taken for each channel/collection device, including final five clearing puffs, to the nearest 0,1 puff;
- observed carbon monoxide concentration, expressed as a percentage by volume, for each channel, to the nearest 0,01 %, and the average per cigarette, to the nearest 0,1 %;
- amount of carbon monoxide determined, in milligrams per cigarette for each channel, to the nearest 0,1 mg, and the average per cigarette, to the nearest 1 mg.

Bibliography

- [1] CORESTA Report, *CORESTA study for the determination of repeatability and reproducibility of the measurement of nicotine-free particulate matter, nicotine and CO in smoke using the ISO smoking methods*; October 2003
- [2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*
- [3] 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*

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