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## Cigarettes – Determination of carbon monoxide in the vapour phase of smoke (NDIR method)

*Cigarettes – Dosage du monoxyde de carbone dans la phase gazeuse de la fumée de cigarette (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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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# Cigarettes — Determination of carbon monoxide in the vapour phase of smoke (NDIR method)

## 1 Scope and field of application

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

## 2 References

ISO 3308, *Cigarettes — Routine analytical cigarette-smoking machine — Definitions, standard conditions and auxiliary equipment.*

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

ISO 4387, *Cigarettes — Determination of total and dry particulate matter by means of a routine analytical cigarette-smoking machine — Glass fibre filter smoke trap method.*

ISO 8243, *Cigarettes — Sampling.*

ISO 8453, *Cigarettes — Determination of total and dry particulate matter by means of a routine analytical cigarette-smoking machine — Electrostatic smoke trap method.*

## 3 Definitions

**3.1 vapour phase:** The portion of smoke which passes the particulate phase trap during smoking according to ISO 4387 or ISO 8453 using a machine conforming to ISO 3308.

**3.2 clearing puff:** Any puff taken after the cigarette has been extinguished.

## 4 Principle

Smoking of cigarettes according to ISO 4387 or ISO 8453. 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, in particular that for gas analysis and:

**5.1 Conditioning enclosure,** maintained accurately in accordance with the conditions specified in ISO 3402, atmosphere A, for conditioning the cigarette sample prior to smoking (see also 8.1).

**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 quantitative collection of 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 subsequent determination of total particulate matter and nicotine.

The impermeability to vapour phase of the gas-collecting device shall be checked with a vapour phase containing 4 to 6 % (V/V) 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 of the vapour phase in the device shall not differ by more than 0,2 % (absolute) from the value measured in the first determination.

When a bag is used as the gas-collecting device, it should be large enough to avoid the final pressure of its contents exceeding the ambient atmospheric pressure. The volume of the bag shall also be no greater than twice the volume of the gas content collected at atmospheric pressure.

**5.4 Non-dispersive infrared (NDIR) analyser,** selective and calibrated for the measurement of carbon monoxide in vapours and gases. The analysers are available from several manufacturers and should have a preferred working range of

0 to 10 % (V/V) CO and a sampling rate of between 0,5 and 2 l/min. It is important to ensure that CO<sub>2</sub> and H<sub>2</sub>O do not affect the response of the instrument by more than 1 % (relative) of the measured CO value.

**5.5 Equipment for the ignition of the cigarettes**, which does not itself produce CO (e.g. hydrogen flame or electric ignition).

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

## 6 Standard gas mixtures

Gas mixtures containing accurately known (to within 2 % V/V relative) concentrations of carbon monoxide in nitrogen for the calibration of the NDIR analyser. The gases should be in cylinders, preferably of aluminium.

The NDIR analyser should be calibrated with at least three standard gas mixtures of accurately known concentrations covering the expected range in such a way as to avoid extrapolation of the calibration curve; typically about 2 %, 4 % and 6 % (V/V) CO in nitrogen would be sufficient.

## 7 Sampling

See ISO 8243.

The laboratory sample shall be taken as agreed between the parties concerned and shall be representative of the population sampled.

## 8 Procedure

### 8.1 Conditioning

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

It is recommended that the testing atmosphere in the laboratory where the smoking is to be carried out be as close as possible to the conditioning atmosphere. If the testing atmosphere is different, place the conditioned final test portion in an airtight container (as small as will just contain the portion) and remove each cigarette from the container just before smoking.

NOTE — Conditioning should be carried out very carefully in order to obtain consistent results.

### 8.2 Calibration of the NDIR analyser

**8.2.1** Adjust the zero of the analyser when the measuring cell has been purged with ambient air.

**8.2.2** Fill a previously evacuated vapour phase collection container with one of the standard gas mixtures, 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, allowing 5 to 10 s for equilibration of pressure of the analyser. Note the reading on the analyser concentration indicator.

**8.2.3** Repeat the procedure as specified in 8.2.2 for the other two standard gas mixtures.

**8.2.4** Plot a calibration curve of the reading obtained on the concentration indicator versus the concentration of the standard gas mixtures used.

**8.2.5** Using the standard gas mixture at about 4 % (V/V) CO, check the calibration curve at least twice a day. If the difference between the readings exceeds 0,2 % (absolute) of the CO concentration given by the calibration curve, prepare a new calibration curve.

### 8.3 Smoking and collection of vapour phase

#### 8.3.1 Preparation of vapour phase collection system

Prepare the system following the instructions pertinent to the unit fitted.

Ensure that the vapour phase collecting device has been completely flushed with ambient air and evacuated before the start of the smoking process.

#### 8.3.2 Smoking procedure

Smoke the cigarettes in accordance with the procedure stated in ISO 4387 or ISO 8453.

#### 8.3.3 Smoking process

**8.3.3.1** Use of fixed or rotating head smoking machine using 44 mm or 55 mm glass fibre filter traps

Zero the puff counters and light the cigarettes. Allow the ash to fall naturally during smoking. When the butt mark has been reached, the burning coal shall be removed from the cigarette. One clearing puff should be taken before removing the butt, to draw the residual smoke from the butt into the filter holder. Insert new cigarettes and repeat the process until the last cigarette has been smoked. When the clearing puff has been taken from the final cigarette, a number of additional clearing puffs shall be taken to ensure that at least 95 % of the vapour phase residing in the smoking machine enters the container. The number of these final clearing puffs depends on the type of equipment and shall be determined by experiment.

Record the total number of puffs (including all clearing puffs) and also the atmospheric pressure and temperature.

NOTE — If the glass fibre filters are to be used for the measurement of the total particulate matter, then only the first of the final series of clearing puffs should be taken with the filters in position, further clearing puffs being taken without the filters.

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**8.3.3.2** Use of rotating head smoking machine using an electrostatic smoke trap or a 92 mm glass fibre filter trap

Zero the puff counters and light the cigarettes. Allow the ash to fall naturally during the smoking process. When the required butt mark has been reached, the burning coal shall be removed from the cigarette. In every case a number of final clearing puffs shall be taken to ensure that at least 95 % of the vapour phase residing in the smoking machine enters the container. At least three clearing puffs shall be taken after all cigarettes have been extinguished.

Record the total number of puffs (including all clearing puffs) and the atmospheric pressure and temperature.

NOTE — The same procedure is also used in the case of simultaneous measurement of CO and total particulate matter.

## 8.4 Measurement of carbon monoxide

Introduce the collected vapour phase (8.3) into the measuring cell of the infrared analyser, allowing 5 to 10 s for equilibrium to be reached. Record the reading and read the observed carbon monoxide from the calibration curve.

## 8.5 Number of determinations

Carry out two determinations as specified in 8.1 to 8.4.

The number of cigarettes of a given type to be smoked shall be in accordance with the provisions of ISO 4387 and ISO 8453.

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## 9 Expression of results

### 9.1 Calculation of carbon monoxide

#### 9.1.1 Calculation of carbon monoxide on volume-per-cigarette basis

The carbon monoxide in the vapour phase, on a volume-per-cigarette basis, is given by the formula

$$\text{ml CO/cigarette} = \frac{C \times V \times N \times p \times 273}{S \times 100 \times 101,3 \times T}$$

where

$C$  is the percentage (V/V) CO observed;

$V$  is the puff volume, in millilitres;

$N$  is the number of puffs in the measured sample (clearing puffs included);

$p$  is the ambient pressure, in kilopascals;

$S$  is the number of cigarettes smoked;

$T$  is the ambient absolute temperature, in kelvins (i.e. Celsius temperature + 273).

Taking as the puff volume  $V = 35$  ml, this expression can be simplified to:

$$\text{ml CO/cigarette} = 0,9432 \times \frac{C \times N \times p}{S \times T}$$

NOTE — Standard conditions should be mentioned in the test report.

#### 9.1.2 Calculation of carbon monoxide on mass-per-cigarette basis

The carbon monoxide in the vapour phase, on a mass-per-cigarette basis, is given by the formula:

$$\text{mg CO/cigarette} = \frac{C \times V \times N \times p \times 273 \times 28}{S \times 100 \times 101,3 \times T \times 22,4}$$

For explanation of symbols see 9.1.1

Taking as the puff volume  $V = 35$  ml, this expression can be simplified to:

$$\text{mg CO/cigarette} = 1,179 \times \frac{C \times N \times p}{S \times T}$$

#### 9.1.3 Calculation of carbon monoxide corrected for number of clearing puffs (only if required)

In place of  $C$  in 9.1.1, the corrected percentage (V/V) CO can be used, calculated as follows:

$$C_{\text{cor}} = \frac{C \times N}{N_L}$$

where

$C_{\text{cor}}$  is the corrected percentage (V/V) CO;

$N_L$  is the number of puffs of the lit cigarettes during the smoking procedure (part puffs included).

### 9.2 Precision

The figures in 9.2.1 and 9.2.2 were derived from the collaborative test results given in the annex.

#### 9.2.1 Repeatability

If the difference between the results of two determinations carried out on the same day by the same operator using the same apparatus and the same laboratory sample exceeds 2,5 mg/cig., then two further determinations shall be carried out.

#### 9.2.2 Reproducibility

The difference between two single values obtained by two analysts in different laboratories, using portions of the same laboratory sample, shall not exceed 5,0 mg/cig. on more than one occasion in twenty.

## 10 Test report

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 result.

The test report shall include all details required for complete identification of the sample.

Where appropriate, record the information listed in 10.1 to 10.4.

### 10.1 Characteristic data about the cigarette

- 10.1.1 Brand name or batch identification.
- 10.1.2 Name of manufacturer; country of manufacture.
- 10.1.3 Length of cigarette.
- 10.1.4 Length of filter.
- 10.1.5 Length of overwrap.

### 10.2 Sampling

- 10.2.1 Type of sampling procedure.
- 10.2.2 Number of cigarettes.
- 10.2.3 Date and location of purchase.

### 10.3 Description of test

- 10.3.1 Date of test.
- 10.3.2 Type of smoking machine used.
- 10.3.3 Number of cigarettes consumed by smoking during a complete analysis.

10.3.4 Number of cigarettes smoked into each vapour phase trap.

10.3.5 Butt length.

10.3.6 Ambient absolute temperature (K) during smoking operation.

10.3.7 Relative humidity (%) during smoking operation.

10.3.8 Ambient pressure (kPa) during smoking operation.

10.3.9 Type of test procedures used.

### 10.4 Test results

10.4.1 Average mass (mg/cig.) of conditioned cigarettes.

10.4.2 Average mass of tobacco (mg/cig.) in conditioned cigarettes.

10.4.3 Moisture content (% by mass) of conditioned tobacco.

10.4.4 Average draw resistance of conditioned cigarettes.

10.4.5 Average diameter of cigarettes.

10.4.6 Average number of puffs per cigarette for each vapour phase trap, to the nearest 0,1 puff.

10.4.7 Carbon monoxide concentration (%) for each vapour phase trap, to the nearest 0,1 %.

10.4.8 Carbon monoxide (mg/cig. or ml/cig.) for each vapour phase trap, to the nearest 1 mg or 1 ml respectively.

10.4.9 Average carbon monoxide (mg/cig. or ml/cig.) for all vapour phase traps, to the nearest 1 mg or 1 ml respectively.

10.4.10 Average mass of burnt tobacco.

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## Annex

## Results of an international collaborative study on five types of cigarettes

(This annex does not form an integral part of the Standard.)

In this study 33 laboratories took part, of which 25 carried out a CO determination. Some laboratories used more than one method of smoking, so that 45 results in all were obtained per cigarette type.

Cigarette type <sup>1)</sup>	Mean dry condensate	Mean nicotine	Mean	CO observed	
				Repeatability <sup>2)</sup> <i>r</i>	Reproducibility <sup>2)</sup> <i>R</i>
A	23,7	1,3	16,408	2,209	4,600
B	17,4	1,3	11,212	1,504	3,337
C	13,6	0,8	15,855	2,111	4,863
D	6,7	0,14	13,878	2,014	4,131
E	4,4	0,37	5,214	0,895	2,556

- 1) A dark tobacco type, without filter
- B virginia type, without filter
- C European type, with filter
- D European type, with filter
- E blended international American type, with ventilated filter

- 2) In accordance with ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

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