

CORESTA RECOMMENDED METHOD N° 5

DETERMINATION OF CARBON MONOXIDE IN THE MAINSTREAM SMOKE OF CIGARETTES BY NON-DISPERSIVE INFRARED ANALYSIS

(September 1993)

1. FIELD OF APPLICATION

The method is applicable to the gas phase of the mainstream smoke of cigarettes.

2. DEFINITIONS

- 2.1.** *The gas phase of cigarette smoke* is that part of the mainstream smoke which passes through a smoke trap conforming to CORESTA Method N° 22 (or ISO 3308).
- 2.2.** *A clearing puff* is any puff taken after the cigarette has been extinguished.

3. REFERENCES

CORESTA Recommended Method N° 21 : 1991

Atmosphere for conditioning and testing tobacco and tobacco products.

CORESTA Recommended Method N° 22 : 1991

Routine analytical cigarette-smoking machine specifications, definitions and standard conditions.

CORESTA Recommended Method N° 23 : 1991

Determination of total and nicotine-free dry particulate matter using a routine analytical cigarette-smoking machine - Determination of total particulate matter and preparation for water and nicotine measurements.

CORESTA Recommended Method N° 24 : 1991

Cigarettes - Sampling

CORESTA Recommended Method N° 25 : 1991

Ambient air flow around cigarettes in routine analytical smoking machines : control and monitoring.

ISO 3402 : 1991

Tobacco and tobacco products - Atmosphere for conditioning and testing.

ISO 3308 : 1991

Cigarettes - Routine analytical cigarette-smoking machine - Definition and standard conditions.

ISO 4387 : 1991

Cigarettes - Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine.

ISO 8243 : 1991

Cigarettes - Sampling

4. PRINCIPLE

Cigarettes are smoked according to a standardised procedure. The carbon monoxide of the total gas phase of the smoke is determined by non-dispersive infrared analysis. The yield is reported on a per cigarette basis.

5. APPARATUS

- 5.1.** *A standard smoking machine* with accessories complying with CORESTA Recommended Methods N° 22 and N° 25 (or ISO 3308).
- 5.2.** *A system for the collection of the total gas phase* satisfying the following conditions:
 - 5.2.1.** It must not interfere with the normal performance of the smoking machine and, consequently, with the determination of total particulate matter and nicotine.
 - 5.2.2.** It must be constructed and maintained so that the carbon monoxide content of the gas phase sample of cigarette smoke containing approximately 5% v/v (4-6% v/v) of carbon monoxide does not change by more than 0.2% v/v CO over a period of two hours.
 - 5.2.3.** Its volume must be chosen according to the actual smoking conditions and must be at least equal to the final volume of the gas phase plus clearing puffs but must not exceed twice that volume. In practice the collection of the gas phase from 5 cigarettes requires a volume of 3 l, and the collection of the gas phase from 20 cigarettes requires a volume of 10 l.
- 5.3.** *A non-dispersive infrared analyser* satisfying the following conditions :
 - 5.3.1.** Range of measurement : 0 - 10% v/v of carbon monoxide.
 - 5.3.2.** Precision : 1% of full scale
Linearity : 1% of full scale
Reproducibility : 0.2% of full scale under conditions of constant temperature and pressure.
 - 5.3.3.** Its response to 10% (v/v) of carbon dioxide must not exceed 0.05% (v/v) as carbon monoxide. Its response to 2% (v/v) of water vapour must not exceed 0.05% (v/v) as carbon monoxide.
- 5.4.** *A thermometer* for measuring ambient temperature to the nearest 0.2 K.
- 5.5.** *A barometer* for measuring atmospheric pressure to the nearest 0.1 kPa.

6. STANDARD GASES

At least three standard gas mixtures of accurately known concentrations of carbon monoxide in nitrogen covering the expected range in such a way as to avoid extrapolation of the calibration curve; typically 1%, 3% and 5% (v/v) of carbon monoxide in nitrogen. The concentrations must be guaranteed by the supplier to be better than 2% (relative).

Note : Make up gases other than nitrogen, such as helium, must not be used as the detector response for carbon monoxide will be different.

7. SAMPLING AND SAMPLE PREPARATION

- 7.1. The sampling of cigarettes must be carried out according to CORESTA Recommended Method N° 24 (or ISO 8243).
- 7.2. The sample for smoking must be conditioned according to CORESTA Recommended Method N° 21 (or ISO 3402).

8. PROCEDURE

8.1. *Calibration of the analyser*

- 8.1.1. After warming up the instrument according to the manufacturers recommendations, purge the instrument with air and adjust to read zero.
- 8.1.2. Fill a previously evacuated gas phase collection container with the standard gas of nominally 5% CO (v/v), 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 of the analyser using the system sampling pump. Note the value when a steady reading has been obtained.
- 8.1.3. If necessary, adjust the instrument reading to agree with the certified value of the standard gas.
- 8.1.4. Repeat 8.1.2. for the other standard gases. If there is a difference greater than 0.2% CO (v/v) between the observed and expected values, attention should be given to the analyser linearity.
- 8.1.5. Establish the relationship between the readings and the actual carbon monoxide concentrations in the standard gases at least weekly. This must be linear within the limits reported in section 5. Non linearity indicates that there is an instrument problem.
- 8.1.6. Check the calibration prior to measurement using the standard gas containing about 5% (v/v) carbon monoxide. If there is a difference greater than 0.2% CO (v/v) between observed and expected values repeat the full calibration (8.1.).

8.2. *Smoking and collection of gas phase.*

8.2.1. Setting up the gas phase collection system.

Proceed in accordance with the instructions pertinent to the equipment used. In particular ensure that the collection device is properly evacuated before the start of smoking. There must not be any residual vacuum upstream of the collection device before smoking.

8.2.2. Smoking procedure

Set up the smoking machine in accordance with CORESTA Recommended Method N° 22 (or ISO 3308) and smoke the cigarettes in accordance with CORESTA Recommended Method N° 23 (or ISO 4387).

8.2.3. After the completion of the smoking run remove the cigarette butt and take five clearing puffs.

8.2.4. Record the total number of puffs taken on each channel, *i.e.* smoking puffs plus clearing puffs.

8.3. *Measurement of the carbon monoxide content of the collected gas phase.*

8.3.1. Recheck the calibration of the analyser (see 8.1.6.) and introduce the gas phase into the measuring cell of the analyser under the same conditions of sampling and ambient temperature and pressure and the same gas flow rate as used during calibration. Read the analyser output and determine the observed carbon monoxide concentration.

8.3.2. At the end of each smoking the gas phase collection container must be emptied and flushed with air. The apparatus is then ready for the next smoking starting at 8.2.1.

9. CALCULATIONS

9.1. Let :

C_{obs} = observed carbon monoxide concentration, % (v/v)

N = total number of puffs in the measured sample (including 5 clearing puffs)

q = number of cigarettes smoked to produce the measured sample

V = puff volume, ml

t = ambient temperature, °C

p = ambient pressure, kPa

C = average carbon monoxide concentration in the smoke

$$C = \frac{C_{obs} \times N}{N - 5}$$

9.2. *Calculation of carbon monoxide yield - volume per cigarette basis.*

$$\text{ml carbon monoxide per cigarette} = \frac{C_{obs} \times V \times N \times p \times 273}{q \times 100 \times 101.3 \times (t+273)}$$

This volume is calculated at 101.3 kPa and 0°C.

9.3. *Calculation of carbon monoxide yield - mass per cigarette basis.*

$$\text{mg carbon monoxide per cigarette} = \frac{C_{obs} \times V \times N \times p \times 273 \times 28}{q \times 100 \times 101.3 \times (t+273) \times 22.4}$$

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 Recommended Method, 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. Where appropriate, record the information in 10.1. to 10.4.

10.1. *Characteristic data about the cigarette - cigarette identification*

It should include as appropriate :

- a) Name of manufacturer, country of manufacture;
- b) Product name;
- 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);
- g) Packet number (of that product sampled that day);
- h) Marks on any tax stamp;
- i) Printed smoke yields (if any);
- j) Length of cigarette;
- k) Length of filter;
- l) Length of overwrap.

10.2. *Sampling*

- a) Type of sampling procedure;
- b) Number of cigarettes in laboratory sample;
- c) Date and location of purchase.

10.3. *Description of test*

- a) Date of test;
- b) Type of smoking machine used;
- c) Type of analyser used;
- d) Total number of cigarettes smoked in the entire determination on that cigarette type;
- e) Number of cigarettes smoked into each collection device;
- f) Butt length;
- g) Room temperature (°C) during smoking operation and analysis;
- h) Relative humidity (%) during smoking operation;
- i) Atmospheric pressure (kPa) during smoking operation and analysis.

10.4. 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.

- a) Average length of the cigarettes to the nearest 0.1 mm;
- c) Average length of the filter to the nearest 0.1 mm;
- c) Average length of the overwrap to the nearest 0.1 mm;
- d) Average butt length to which the cigarettes were smoked to the nearest 0.1 mm;
- e) Average length of tobacco portion smoked to the nearest 0.1 mm;
- f) Average diameter of the cigarettes (mm);
- g) Average draw resistance of the conditioned cigarettes;
- h) Average mass (mg per cigarette) of the conditioned cigarettes selected for the smoking operation;
- i) Water content (% mass/mass) of the conditioned cigarettes (ISO 6488);
- j) Average number of puffs per cigarette for each channel to the nearest 0.1 puff;
- k) Average number of total puffs taken for each channel/collection device including final five clearing puffs to the nearest 0.1 puff;
- l) Observed carbon monoxide concentration (% v/v) for each channel to the nearest 0.01% and the average per cigarette to the nearest 0.1%;
- m) Average carbon monoxide concentration in smoke (% v/v) for each channel to the nearest 0.01% and the average per cigarette to the nearest 0.1%;
- n) Carbon monoxide (mg per cigarette or ml per cigarette) for each channel to the nearest 0.1 mg or ml and the average per cigarette to the nearest 1 mg or ml.

11. REPEATABILITY AND REPRODUCIBILITY

An international collaborative study involving 32 laboratories and 4 samples conducted in 1993 shows the following values for repeatability (r) and reproducibility (R) of this method. 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 value (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 value (R) on average not more than once in 20 cases in the normal and correct operation of the method.

Data analysis gave the estimates as summarised in the following table :

Mean yield of carbon monoxide mg	Repeatability conditions r	Reproducibility conditions R
3.45	0.47	1.18
3.56	0.42	1.03
9.89	0.85	2.22
13.80	1.09	3.00

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.