



**Cooperation Centre for Scientific Research
Relative to Tobacco**

Cigar Smoking Methods Sub-Group

**CORESTA Recommended Method
No. 68**

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

March 2020



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CORESTA RECOMMENDED METHOD N° 68

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

(March 2020)

0. INTRODUCTION

This CORESTA Recommended Method (CRM) is part of a set of CRMs produced by the Cigar Smoking Methods Sub-Group which describes the machine smoking of cigars:

CRM N° 46 – Atmosphere for Conditioning and Testing Cigars of All Sizes and Shapes;

CRM N° 47 – Cigars – Sampling;

CRM N° 64 – Routine Analytical Cigar-Smoking Machine Specifications, Definitions and Standard Conditions;

CRM N° 65 – Determination of Total and Nicotine-Free Dry Particulate Matter Using a Routine Analytical Cigar-Smoking Machine – Determination of Total Particulate Matter and Preparation for Water and Nicotine Measurements;

CRM N° 66 – Determination of Nicotine in the Mainstream Smoke of Cigars by Gas Chromatographic Analysis;

CRM N° 67 – Determination of Water in the Mainstream Smoke of Cigars by Gas Chromatographic Analysis;

CRM N° 68 – Determination of Carbon Monoxide in the Mainstream Smoke of Cigars by Non-Dispersive Infrared Analysis.

This series of CRMs has been developed for use with machine smoking of cigar products. These CRMs are applicable to many commercially available products; however, some deviations may be necessary to accommodate products not considered during the development of these CRMs.

1. FIELD OF APPLICATION

This method is applicable to the determination of carbon monoxide in the mainstream smoke from cigars, generated and collected using a routine analytical smoking machine, by non-dispersive infrared analysis.

2. NORMATIVE REFERENCES

CORESTA Recommended Method N° 46, *Atmosphere for Conditioning and Testing Cigars of All Sizes and Shapes*

CORESTA Recommended Method N° 64, *Routine Analytical Cigar-Smoking Machine Specifications, Definitions and Standard Conditions*

CORESTA Recommended Method N° 65, *Determination of total and nicotine-free dry particulate matter using a routine analytical cigar-smoking machine – determination of total particulate matter and preparation for water and nicotine measurements*

3. TERMS AND DEFINITIONS

For the purposes of this Recommended Method, the following definitions apply:

- 3.1 **The gas phase of cigar smoke** is that part of the mainstream smoke which passes through a smoke trap conforming to CORESTA Recommended Method N° 64

4. PRINCIPLE

Cigars are conditioned according to CORESTA Recommended Method N° 46 and then smoked on a routine analytical smoking machine in a conditioned atmosphere. The carbon monoxide of the total gas phase of the smoke is determined by non-dispersive infrared analysis.

5. APPARATUS

- 5.1 A standard smoking machine complying with CORESTA Recommended Method N° 64 and equipped for smoking.
- 5.2 A system for the collection of the total gas phase satisfying the following conditions:
- 5.2.1 It shall 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 shall be constructed and maintained so that the carbon monoxide content of the gas phase sample of cigar smoke containing approximately 5 % v/v (4 % - 6 % v/v) of carbon monoxide does not change by more than 0,3 % v/v CO over a period of three hours.
- 5.2.3 Its volume shall be chosen according to the actual smoking conditions and shall be at least equal to the final volume of the gas phase plus clearing puffs but it is recommended that it does not exceed twice that final volume. In practice the collection of the gas phase from cigars requires a volume, that depends on the size of the cigar and the corresponding puff volume (see CRM 65, paragraph 7.5.2). Depending on the carbon monoxide equipment's specifications, a minimum gas phase volume could be required for a measurement. To meet this requirement the collection of the gas phase of more than one cigar, smoked in the same or in different channels, could be needed.
- 5.3 A non-dispersive infrared analyser satisfying the manufacturer's instructions and the following conditions:
- 5.3.1 Range of measurement: An instrument of sufficient range to accommodate expected sample values, which can depend on the number of clearing puffs used to dilute the sample. For undiluted gas samples a range of 0,00 % v/v to 15,00 % v/v has been determined to be sufficient. Alternative ranges are acceptable provided that the performance specification given in paragraph 5.3.2 is equalled or exceeded.
- 5.3.2 Precision: +/- 0,15 % v/v or better
Linearity: +/- 0,15 % v/v or better
Resolution: 0,01 % v/v or better
Repeatability: +/- 0,03 % v/v or better, determined over the normal measurement period and under conditions of constant temperature and pressure.
- 5.3.3 Its response to 10 % (v/v) of carbon dioxide shall not exceed 0,05 % (v/v) as carbon monoxide. Its response to 2 % (v/v) of water vapour shall not exceed 0,05 % (v/v) as carbon monoxide.
- 5.4 A thermometer for measuring ambient temperature to the nearest 0,2 °C.
- 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 2 %, 7 % and 12 % (v/v) of carbon monoxide in nitrogen, or any other set of 3 standards according to the specified range of the CO analyzer. The concentrations shall be guaranteed by the supplier to be better than 2 % (relative).

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 Cigars shall be conditioned according to CORESTA Recommended Method N° 46.

8. PROCEDURES

8.1 *Calibration of the analyser*

8.1.1 After warming up the instrument according to the manufacturer's 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 12 % CO (v/v), or another concentration close to the upper limit of the equipment range, 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 0,3 % CO (v/v) between the observed and expected values, then corrective action on the analyser should be performed.

8.1.5 Establish the relationship between the readings and the actual carbon monoxide concentrations in the standard gases at least weekly. This shall 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 12 % (v/v) carbon monoxide, or another concentration close to the upper limit of the equipment range. If there is a difference greater than 0,3 % 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 shall 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° 64 and smoke the cigars in accordance with CORESTA Recommended Method N° 65.

8.2.3 After the completion of the smoking of each cigar remove the burning coal of the cigar butt and take the necessary clearing puffs - at least one - to assure that all gas phase has been collected and that the CO measurement (% (v/v)) is within the equipment's specified range (see CORESTA Recommended Method N° 65, paragraph 7.6.4). To fulfil this requirement the number of clearing puffs (dilution process) shall be adjusted according to the experience with previous measurements in similar samples. Then remove the cigar butt.

Note: Under certain conditions, for example low total puff count, a sufficient quantity of gas phase smoke may not be collected to allow for CO analysis. There are several possible options to measure CO in these products. 1) Increase the number of clearing puffs 2) Increase the number of cigars smoked per port 3) Collect the gas phase smoke from multiple smoking machine ports into a single bag. The impact of modifying the collection scheme should be evaluated by the testing facility.

The increased number of clearing puffs may be required when smoking small cigars due to the small volume of gas collected during puff collection. This collected volume may be insufficient to account for the dead volume in the smoke machine thus the volume is increased through 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 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 shall be emptied. 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 clearing puffs)

q = number of cigars smoked to produce the measured sample

V = puff volume, ml

t = ambient temperature, °C

p = ambient pressure, kPa

W = average mass of conditioned cigars, g per cigar

9.2 *Calculation of carbon monoxide yield - mass per cigar basis.*

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

9.3 *Calculation of carbon monoxide yield – mass per gram tobacco basis.*

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

10. REPEATABILITY AND REPRODUCIBILITY

Collaborative Studies involving 9 laboratories conducted in 2018 found the following values for repeatability (r) and reproducibility (R) of this method.^[1]

The difference between two single results found on matched cigar 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 cigar 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.

Data analysis gave the estimates (expressed in mg carbon monoxide/cigar) as summarised in the following Table 1:

| Product | weight (mg) | diameter (mm) | CO (mg/cigar) | r (mg/cigar) | R (mg/cigar) | r (%) | R (%) |
|---------|----------------|------------------|------------------|-----------------|-----------------|----------|----------|
| A | 2557 | 8,9 | 82,0 | 16,91 | 25,29 | 20,6 % | 30,9 % |
| B | 3287 | 9,6 | 59,2 | 24,15 | 35,86 | 40,8 % | 60,5 % |
| C | 4049 | 9,6 | 63,7 | 28,82 | 30,98 | 45,3 % | 48,7 % |
| D | 2754 | 10,4 | 105,2 | 18,97 | 33,25 | 18,00 % | 31,6 % |
| E | 2774 | 10,4 | 105,5 | 26,61 | 29,56 | 25,2 % | 28,0 % |
| F | 3026 | ~12 | 117,3 | 36,86 | 42,48 | 31,4 % | 36,2 % |
| G | 7721 | 14-15 | 205,5 | 46,23 | 55,4 | 22,5 % | 27,0 % |
| H | 8254 | 17 | 317,4 | 148,4 | 205,4 | 46,7 % | 64,7 % |
| I | 960 | 7,8 | 9,7 | 2,07 | 2,46 | 21,3 % | 25,4 % |

These findings suggest that the product variability, inherent to cigar production, is reflected in a wide range of smoke yields when cigars are machine-smoked. For the purposes of calculating r and R, one test result (or one “single” result) was defined as the yield obtained from smoking 1 cigar per filter pad for Products B through H.

For products A and I one “single” result was obtained from smoking 2 cigars per filter pad.

The subject of tolerances due to sampling is dealt with in CORESTA Recommended Method N° 47.

11. TEST REPORT

The test report shall give the carbon monoxide yield from each cigar smoked and the method used and shall include all conditions not defined in this Recommended Method that may affect the results (e.g. atmospheric conditions, cigar diameter, puff volume, etc.) as well as any deviations from this Recommended Method. The test report shall also give all details necessary for the identification of the cigars smoked.

^[1] Cigar Smoking Methods Technical Report, 2018 Collaborative Study for CRM65 Update of Repeatability and Reproducibility [CSM-121-0-CTR]

The test report shall include all details required for complete identification of the sample. Where appropriate, record the information in 11.1 to 11.4.

11.1 *Characteristic data about the cigar*

Cigar identification - in the case of a commercial cigar this should include:

- 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) Length of cigar;
- j) Where applicable: length of filter;
- k) Where applicable: length of artificial mouthpiece.

11.2 *Sampling*

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

11.3 *Description of test*

- a) Date of test;
- b) Type of smoking machine used;
- c) Type of analyser used;
- d) Total number of cigars smoked in the entire determination on that cigar type;
- e) Number of cigars smoked into each gas 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.

11.4 *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 cigars to the nearest 0,5 mm;
- c) Average length of the filter (where applicable) to the nearest 0,5 mm;
- c) Average length of the tip (where applicable) to the nearest 0,5 mm;

- d) Butt length to which the cigars 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 cigars at 15 mm from mouth end, to the nearest 0,1 mm (for the determination of the holder construction);
- g) Average diameter of the cigars at 33 mm from mouth end, to the nearest 0,1 mm (for the determination of the Puff Volume);
- h) Average draw resistance of the conditioned cigars;
- i) Average mass (mg per cigar) of the conditioned cigars selected for the smoking operation;
- j) Water content (% mass/mass) of the conditioned cigars;
- k) Average number of puffs per cigar for each channel to the nearest 0,1 puff;
- l) Average number of total puffs taken for each channel/collection device including final clearing puffs to the nearest 0,1 puff;
- m) Observed carbon monoxide concentration (% v/v) for each channel to the nearest 0,01 %;
- n) Carbon monoxide (mg per cigar) for each channel to the nearest 0,1 mg and the average per cigar to the nearest 1 mg;
- o) Carbon monoxide (mg per gram tobacco) for each channel to the nearest 0,1 mg, dividing the Carbon monoxide (in mg/cigar) by the cigar mass (in g per cigar) of the conditioned cigars selected for smoking in each particular channel, and the average per gram tobacco to the nearest 1 mg.

12. BIBLIOGRAPHY

CORESTA Recommended Method N° 47, *Cigars - Sampling*

Cigar Smoking Methods Technical Report, *2018 Collaborative Study for CRM65 Update of Repeatability and Reproducibility* [CSM-121-0-CTR]