



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

**Cigar Smoking Methods Sub-Group**

**CORESTA Recommended Method  
No. 67**

**DETERMINATION OF WATER  
IN THE MAINSTREAM SMOKE  
OF CIGARS BY GAS  
CHROMATOGRAPHIC ANALYSIS**

March 2020



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# CORESTA RECOMMENDED METHOD N° 67

## DETERMINATION OF WATER IN THE MAINSTREAM SMOKE OF CIGARS BY GAS CHROMATOGRAPHIC 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 cigars. These CRMs are applicable to many commercially available products; however, 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 water in the mainstream smoke from cigars, generated and collected using a routine analytical smoking machine, by gas chromatography with thermal conductivity detection.

### 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 Total Particulate Matter, Crude Smoke Condensate, TPM

That portion of the mainstream smoke which is trapped on the glass fibre filter trap, expressed as milligrams per cigar.

#### 3.2 Dry Particulate Matter, Dry Smoke Condensate, DPM

The total particulate matter after deduction of its water content, expressed as milligrams per cigar.

#### 3.3 Nicotine-free Dry Particulate Matter, Nicotine-free Dry Smoke Condensate, NFDPM

The dry particulate matter after deduction of its water and nicotine content, expressed as milligrams per cigar.

### 4. PRINCIPLE

Cigars are conditioned according to CORESTA Recommended Method N° 46. Cigars are then smoked using the procedure found in CORESTA Recommended Method N° 65 and the total particulate matter (TPM) of mainstream smoke is collected on a glass fibre filter trap. The TPM is dissolved in a solvent and the water content of this solution is determined by gas chromatography. Results are expressed as the weight of water delivered per cigar.

### 5. APPARATUS

**5.1** A standard smoking machine complying with CORESTA Recommended Method N° 64 and equipped for smoking.

**5.2** A gas chromatograph equipped with a thermal conductivity detector, autosampler, and a data collection system.

**5.3** A packed or capillary column may be used for analysis. There are several suitable columns.

**5.3.1** Porapak 150 µm (100 mesh) to 190 µm (80 mesh)<sup>[1]</sup> with column dimensions consisting of an internal diameter between 2 mm and 4 mm and length of 1,5 m to 2 m have been shown to provide suitable results.

**5.3.2** Porapak QS and Chromosorb 102<sup>[1]</sup> packed stationary phases with column dimensions consisting of an internal diameter between 2 mm and 4 mm and length of 1,5 m to 2 m have been shown to provide suitable results.

**5.3.3** PoraPLOT Q<sup>[1]</sup> capillary columns with a length between 15 m and 30 m and internal diameter between 320 µm and 530 µm and film thickness between 20 µm and 40 µm have also been shown to provide suitable results.

**5.4** The necessary general laboratory equipment for the preparation of samples, standards and reagents.

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<sup>[1]</sup> This information is given for the convenience of users of this document and does not constitute an endorsement of this product.

## 6. REAGENTS

- 6.1 Propan-2-ol (analytical grade).
- 6.2 Ethanol (minimum purity 99 %).
- 6.3 Solvent for samples and standards: Propan-2-ol containing an appropriate concentration of internal standard (ethanol), normally 5 ml per litre.
- 6.4 Carrier gas: helium.
- 6.5 Distilled water for the preparation of standard solutions.

## 7. STANDARDS

Prepare a series of at least four calibration solutions whose concentrations cover the range expected to be found in the samples by adding weighed amounts of water to the solvent, described in Section 6.3. One of these calibration solutions shall be the solvent with no added water (solvent blank).

## 8. PROCEDURES

### 8.1 *Gas Chromatography*

Set up and operate the gas chromatograph according to the manufacturer's instructions.

Ensure that the peaks for water, internal standard and solvent are well resolved.

Suitable conditions for the packed column with the Porapak stationary phase are:

Column temperature: 170 °C (isothermal)

Injection temperature: 250 °C

Detector temperature: 250 °C

Carrier gas: helium at a flow rate of about 30 cm<sup>3</sup> per minute

Injection volume: 2,0 µl

The total analysis time is about 4 minutes.

Condition the system by injecting the solvent prior to use.

The instrumental conditions shown above are for packed columns using a Porapak stationary phase. Alternative packed and capillary columns have been shown to provide suitable results for the determination of water in TPM. If alternate columns are used, it is necessary to ensure that the water and internal standard peaks are well resolved from the solvent peak and peaks due to other smoke components. Suitable operating conditions will depend on the type of column and injection system used.

### 8.2 *Calibration of the gas chromatograph*

Inject single or replicate aliquots of the standard solutions into the gas chromatograph. Record the peak areas of water and the internal standard.

Calculate the ratio of the water peak to the internal standard peak from the peak area data for each of the calibration solutions including the solvent blank. Plot the graph of the concentrations of added water in accordance with the area ratios or calculate a linear regression equation (concentration of added water in accordance with the area ratios) from these data.

**Note:** Due to the original water content of the solvent, the calibration curve will not pass through the origin.

### 8.3 Calibration check

The full calibration procedure should be carried out daily, ensuring a correlation coefficient of at least 0,99 is obtained. In addition inject an aliquot of an intermediate standard after at least every 20 samples. If the value for this solution differs by more than 10% from the original calibration value, repeat the full calibration. Check the gas chromatograph, and repeat the entire analysis.

### 8.4 Smoking and sample preparation

#### 8.4.1 Using CORESTA Recommended Method N° 64 and CORESTA Recommended Method N° 65 set up the smoking machine, smoke the cigars and collect the particulate matter.

Extract using the solvent (20 ml) described in Section 6.3. The solutions should not be stored in daylight.

The measurement can be done immediately after shaking, or after one night storing (without shaking).

### 8.5 Measurement and calculation of the water content of samples.

#### 8.5.1 Inject single or replicate aliquots of the smoke and blank solutions into the gas chromatograph using the conditions described in Section 8.1. Record the peak areas of water and the internal standard.

Calculate the mean value of the ratio of the peak area of water to that of the internal standard for the replicate injections.

#### 8.5.2 Using the calibration produced in Section 8.2 determine the concentration of water in the smoke and blank solutions in mg per ml. Ensure that the values lie within the range of the standards prepared in Section 7. Determine the mean value of the sample blanks and subtract this from the values obtained for each of the smoke samples.

#### 8.5.3 Calculate the water content and express the test results in milligrams per cigar, for each channel to the nearest 0,01 mg and the average per cigar to the nearest 0,1 mg.

**Note:** The minimum concentration which can be determined by this method is 0,1 mg per ml.

## 9. SPECIAL PRECAUTIONS

Water from the laboratory atmosphere can be adsorbed onto glassware and smoke traps and absorbed by solutions. These factors can produce incorrect and variable results.

To minimise this, the following precautions shall be taken:

- 9.1 Glassware and septa for vials shall be dried and stored under desiccation before use.
- 9.2 The bulk solvent container shall be fitted with a trap to prevent water being absorbed by the solvent.
- 9.3 Flush the dispensing system prior to use by dispensing to waste a minimum of 40 cm<sup>3</sup>.
- 9.4 All solutions shall be kept sealed.
- 9.5 The flasks used for extracting samples should not exceed 150 cm<sup>3</sup> capacity.
- 9.6 The glass fibre filter holders shall be made of a non-hygroscopic and chemically inert material, e.g. high molecular weight polyethylene.

- 9.7 The smoke traps shall be sealed until use and resealed immediately after the completion of smoking.
- 9.8 When determining nicotine and water sequentially, the water determination shall be performed first.
- 9.9 Repeat sampling should be performed with a minimum delay.
- 9.10 Suitable gloves shall be worn when handling smoke traps.

## 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<sup>[2]</sup>.

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 water/cigar) as summarised in the following Table 1:

Product	weight (mg)	diameter (mm)	Water (mg/cigar)	r (mg/cigar)	R (mg/cigar)	r (%)	R (%)
A	2557	8,9	8,77	2,84	5,43	32 %	62 %
B	3287	9,6	4,70	2,81	7,29	60 %	155 %
C	4049	9,6	7,40	3,08	8,39	42 %	113 %
D	2754	10,4	5,61	2,87	4,36	51 %	78 %
E	2774	10,4	4,50	2,19	3,70	49 %	82 %
F	3026	~12	3,61	4,02	4,28	111 %	119 %
G	7721	14-15	8,70	6,49	11,3	75 %	130 %
H	8254	17	24,21	17,6	27,1	73 %	112 %
I	960	7,8	0,86	0,41	0,52	47 %	60 %

These findings suggest that the product variability, inherent to cigar production, is reflected in the variability 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.

<sup>[2]</sup> Cigar Smoking Methods Technical Report CSM-121-0-CTR, 2018 Collaborative Study for CRM65 Update of Repeatability and Reproducibility.

## 11. TEST REPORT

The test report shall give the water content 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.

## 12. BIBLIOGRAPHY

CORESTA Recommended Method N° 46, *Atmosphere for conditioning and testing cigars of all shapes and sizes*

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

CORESTA Recommended Method N° 64, *Routine analytical cigar-smoking machine - specifications, definitions, and standard conditions*

CORESTA Recommended Method N° 67, *Determination of water in the mainstream smoke of cigars by gas chromatographic analysis*

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