

## CORESTA RECOMMENDED METHOD N° 8

### DETERMINATION OF WATER IN THE MAINSTREAM SMOKE OF CIGARETTES BY GAS CHROMATOGRAPHIC ANALYSIS

(August 1991)

#### 1. FIELD OF APPLICATION

The method is applicable to the particulate matter of mainstream cigarette smoke.

#### 2. DEFINITION

Particulate matter is that part of mainstream smoke which is collected by a smoke trap conforming to CORESTA Recommended Method N° 22 or ISO 3308.

#### 3. REFERENCES

*CORESTA Recommended Method N° 5 : 1982*

Determination of carbon monoxide in the mainstream smoke of cigarettes by non-dispersive infrared analysis.

*CORESTA Recommended Method N° 7: 1991*

Determination of nicotine in the mainstream smoke of cigarettes by gas chromatographic analysis.

*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

*ISO 10315: 1991*

Cigarettes - Determination of nicotine in smoke condensates - Gas chromatographic method.

*ISO 3308: 1991*

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

*ISO 4387: 1991*

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

*ISO 8243: 1991*

Cigarettes - Sampling.

#### **4. PRINCIPLE**

Cigarettes are smoked and the particulate matter of mainstream smoke is collected by a standard procedure. The particulate matter 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 cigarette.

#### **5. APPARATUS**

- 5.1.** A standard smoking machine complying with CORESTA Recommended Method N° 22 or ISO 3308 and equipped for smoking.
- 5.2.** A gas chromatograph equipped with a thermal conductivity detector together with a recorder or an integrator. Deactivated stainless steel columns for the gas chromatograph should be 1.5 m to 2 m long and have an internal diameter of about 2mm. A suitable syringe for sample injection or for automatic analysis an autosampler, that is compatible with the gas chromatograph, is required.
- 5.3.** The necessary general laboratory equipment, for the preparation of samples, standards and reagents.

#### **6. REAGENTS**

- 6.1.** Propan-2-ol (analytical grade, maximum water content : 1.0 mg per cm<sup>3</sup>).
- 6.2.** Ethanol (minimum purity 99%).
- 6.3.** Solvent for samples and standards : Propan-2-ol containing 5 cm<sup>3</sup> per litre of ethanol as internal standard.
- 6.4.** Column packing material : Porapak Q, 80 to 100 mesh.
- 6.5.** Carrier gas : helium.
- 6.6.** 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 (usually up to 4 mg per cm<sup>3</sup>) by adding weighed amounts of water to the solvent, described in section 6.3. One of these calibration solutions must be the solvent with no added water (solvent blank).

## 8. PROCEDURES

### 8.1. *Gas chromatography*

Set up and operate the gas chromatograph, recorder or integrator and autosampler (if one is used) according to the manufacturer's instructions.

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

Suitable conditions 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: 2µl

The total analysis time is about 4 minutes.

Condition the system by injecting the solvent prior to use.

### 8.2. *Calibration of the gas chromatograph*

Inject 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 (or height) 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. Use the slope of the linear regression equation.

**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. In addition inject an aliquot of an intermediate standard after every 20 samples. If the value for this solution differs by more than 3% from the original calibration value, repeat the full calibration.

### 8.4. *Smoking and sample preparation*

**8.4.1.** Using CORESTA Recommended Methods N° 22 and 23 or ISO 3308 and 4387, set up the smoking machine, smoke cigarettes and collect the particulate matter.

Extract using the solvent (20 cm<sup>3</sup> or 50 cm<sup>3</sup>) described in section 6.3. The solutions should not be stored in daylight.

**8.4.2.** Due to the absorption of water by smoke traps and solvent, it is necessary to determine a value for the sample blank. Sample blanks are prepared by treating additional smoke traps (usually four per day) in the same manner as that used for smoke collection. They are placed near the smoking machine during smoking and extracted and analysed together with the smoke samples.

The same batch of solvent shall be used for the samples as for the corresponding calibration solutions.

Prepare new calibration solutions (section 7) whenever starting a new batch of solvent (section 6.3).

**Note 1:** If measuring nicotine refer to CORESTA Recommended Method N° 7.

**Note 2:** If measuring carbon monoxide refer to CORESTA Recommended Method N° 5.

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

**8.5.1.** Inject replicate aliquots of the smoke and blank solutions into the gas chromatograph using the conditions described in section 8.1. Record the peak areas (or heights) of water and the internal standard.

Calculate the mean value of the ratio of the peak area (or height) 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 cm<sup>3</sup>. 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 cigarette, for each channel to the nearest 0.01 mg and the average per cigarette to the nearest 0.1 mg.

**Note:** The minimum concentration which can be determined by this method is 0.1 mg per cm<sup>3</sup>.

## **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

A major international collaborative study involving 30 laboratories and 6 samples conducted in 1990 shows that when cigarettes are smoked according to CORESTA Recommended Method N° 23 and the resulting smoke solutions are analysed by this method the following values for repeatability (r) and reproducibility (R) are obtained.

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 Nicotine mg	Repeatability Conditions r	Reproducibility Conditions R
0.083	0.154	0.241
0.153	0.228	0.353
0.338	0.272	0.381
0.962	0.407	0.734
1.595	0.561	0.935
3.187	0.908	1.680

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.

For further details of the interaction of r and R with other factors see CORESTA Report 91/1.

The subject of tolerances due to sampling is dealt with in CORESTA Recommended Method N° 24.

## 11. TEST REPORT

The test report shall give the water content from each cigarette smoked and the method used, and shall include all conditions which may affect the results (*e.g.* atmospheric pressure during smoking). It shall also give all details necessary for the identification of the cigarettes smoked.

## ANNEX

(Informative, this Annex does not form an integral part of the Recommended Method).

1. The described method is a reference method which might be altered for practical purposes or special laboratory conditions in several aspects, especially :
  - 5.2. column tubing material other than deactivated stainless steel such as glass or nickel may be used.
  - 6.2. methanol may be used as the internal standard.
  - 6.4. Porapak QS or Chromosorb 102 may be used as column packing material.
  - 6.5. nitrogen may be used as an alternative carrier gas, if the detector sensitivity is sufficiently high.
  
2. This method can be used in conjunction with the CORESTA Recommended Method N° 7: 1991 - Determination of nicotine in the mainstream smoke of cigarettes by gas chromatographic analysis. This may be done by :
  - (i) including the appropriate level of the internal standard required for the nicotine determination in the solvent described in section 6.3.
  - (ii) injecting an aliquot of the smoke solution into the column for nicotine analysis which is connected to a flame ionisation detector as well as into the water column and detector described in this method. A simultaneous automated analysis of nicotine and water may be achieved by using a splitting system or an autosampler with two injection positions.