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

(June 2010)

FOREWORD

The purpose of this Method Nº 65 is to allow comparisons to be made between different types of cigars by using a strictly controlled set of smoking parameters. As such, Method Nº 65 is to be considered as a technical convention, which does not intend to reflect human smoking behaviour.

The smoking technology that is used is similar, but not equal to the technology that is used for other tobacco products such as cigarettes and fine cut smoking articles.

Several Collaborative Studies have demonstrated that repeatability (r) and reproducibility (R) for NFDPM are considerably higher than found for cigarette smoking, reflecting the high product variability inherent to cigars. Future work must pay attention to other, non-product related factors, in order to try to reduce the high tolerances as much as possible.

The CORESTA Cigars Sub-Group has developed a method that provides a credible and useful comparison of the smoke chemistry of cigars of all shapes and sizes, taking into consideration the practical, time consuming work of cigar smoking in laboratories. In order to maintain the same air velocity through the burning cone, the puff volume is modulated as a function of the cigar diameter for cigars with a diameter >12,0 mm.

Cigar diameter is of crucial importance for the determination of the puff volume. The development of more accurate and reliable techniques to determine the diameter for the wide variety of cigars is the number one priority for the future.

Nevertheless the Sub-Group considers this method to be a sound starting point for future developments.

0. INTRODUCTION

Cigars are manufactured in a wide range of sizes and shapes.

Cigars range, in *length*, from approximately 65 mm to 250 mm, and in *weight*, from less than 1 g to over 15 g. In *diameter*, they range from approximately 6 to 25 mm.

In addition, manufactured cigars are far from uniform in shape, as some are nearly round and others are actually conical. Cigars also vary in shaping at the mouth end, with some presenting a straight appearance, whilst others taper gradually to a rounded end.

Another source of variability is the lack of homogeneity of the filler, including variation in particle size and the types of tobacco used, which tends to affect the pressure drop of the cigar, and consequently its smoking characteristics.

Similarly, the binder and wrapper, because not extensively processed, vary in thickness, texture, porosity and combustibility, as any natural substance does. Variations in the veins of the wrapper leaf can affect the seal between the cigar and the smoking machine. Variations in the porosity of the binder and the wrapper also can affect machine-measured yields, as can any air inflow or outflow through the sideseam of the cigar.

Machine smoking of cigars so far has not been standardised, mainly for the reason that a smoking machine, specifically designed to accommodate cigars of all shapes and sizes, was not available.

However CORESTA published, in Bulletin 1975-1, page 33, a *provisional recommendation* on parameters for cigar smoking for cigars of a limited weight range. Since that time, CORESTA's Cigars Sub-Group (also known as ICCSS : *International Committee for Cigar Smoke Study*) adopted these parameters for *all* cigar testing, *i.e.* even for products outside the initially specified weight range.

A major problem in cigar testing in the past has been the lack of a cigar holder that holds and seals cigars of all shapes and sizes well. This work was finalised and provided a breakthrough for the machine smoking of cigars of all shapes and sizes.

In the meantime also two Recommended Methods were developed and approved :

- N° 46 : Atmosphere for conditioning and testing cigars of all sizes and shapes (published in CORESTA Bulletin 1998/1, and
- N° 47 : Cigars – Sampling (published in Bulletin 2000/1).

During the work of the Cigars Sub-Group it also became clear that, apart from a Method for the *Determination of NFDPM for Cigars*, other methods as well were to be developed, *i.e.* :

- a Method for *Routine Analytical Smoking Machine for Cigars*,
- a Method for the *Determination of Nicotine in Mainstream Smoke of Cigars*, and
- a Method for the *Determination of Water in Mainstream Smoke of Cigars*

The Terms of Reference for the Cigars Sub-Group, as stipulated by the Scientific Commission, require "*to provide a credible and useful comparison of the smoke chemistry of cigars of all shapes and sizes*".

In order to cope with this requirement, after several Collaborative Studies, the Sub-Group Cigars recognised the need to adapt the smoking parameters as a function of the size of the cigar to be smoked. From the parameters available (Puff Volume, Puff Duration, Puff Frequency and Butt Length) the *Puff Volume* was considered to be the most logical one to be modulated as a function of the size of the product; specifically the *diameter* of the product was chosen as the basis for modulating the Puff Volume since it allows for a semi-constant airspeed through the cigar irrespective of the diameter of the product.

Puff Volumes have been selected as follows :

Cigar diameter x (mm)	Puff volume y (ml)
≤12,0	20
≥12,1	$y = 0,139 * x^2$

where: y = puff volume, in ml (to the nearest 1 ml)
x = cigar diameter, in mm (to the nearest 0,1 mm)

Inherent to the choice of these Puff Volumes, products with a diameter:

- ≤ 12,0 mm will be smoked with an increasing airspeed for a decreasing diameter
- ≥ 12,1 mm will be smoked with a constant airspeed of 11,8 cm/s.

It must be stated that this method has *limitations*:

- So far a uniform technique for the measurement of cigar diameter has not been developed. It is known that different techniques used give different diameter results, and as a consequence this can lead to different Puff Volumes for one and the same product being used by different laboratories.
- So far the maximum TPM load of the 55 mm filter trap used has not been defined by experiment. In ISO 4387:2000(E) for NFDPM calculation in cigarette smoke inside its section 7.7 it is stated that glass fiber filter pads of 44 mm diameter are capable of retaining up to 150 mg of total particulate matter and pads of 92 mm diameter are capable of retaining 600 mg of TPM. By interpolation it can be calculated that the maximum TPM load of the 55 mm filter trap is 200 mg, if, during smoking, this mass is exceeded for cigars smoked as 2 cigars per filter pad, then these cigars will be smoked at 1 cigar per filter pad. It might well happen that this maximum load in some cases will be exceeded, even when a product is smoked at 1 cigar per filter pad.
- The choice of the Puff Volume on the basis of the cigar diameter leads to a semi-constant airspeed *assuming that the product is cylindrical*. However many cigars on the market are not cylindrical (pressed cigars, conical cigars, torpedo cigars, …).

1. FIELD OF APPLICATION

This Method is applicable to the determination of total particulate matter and for the subsequent determination of nicotine-free dry particulate matter present in the mainstream smoke from cigars, generated and collected using a routine analytical smoking machine.

2. DEFINITIONS

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

2.1. *Total Particulate Matter : Crude Smoke Condensate*
That portion of the mainstream smoke which is trapped in the smoke trap, expressed as milligrams per cigar.

2.2. *Dry Particulate Matter : Dry Smoke Condensate*

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

2.3. *Nicotine-free Dry Particulate Matter : Nicotine-free Dry Smoke Condensate*

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

2.4. *Clearing Puff*

Any puff taken after the cigar has been extinguished, with the same volume as drawn while smoking.

2.5. *Smoking Process*

The use of a smoking machine to smoke cigars from lighting to final puff.

2.6. *Smoking Run*

A specific smoking process to produce such smoke from a sample of cigars as is necessary for the determination of the smoke components.

2.7. *Laboratory Sample*

The sample intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample.

2.8. *Test Sample*

Cigars for test taken at random from the laboratory sample and which are representative of each of the increments making up the laboratory sample.

2.9. *Conditioning Sample*

The cigars selected from the test sample for conditioning prior to tests for particulate matter yield.

2.10. *Conditioned Sample*

Conditioned cigars smoked for particulate matter yield tests

2.11. *Test Portion*

A group of cigars prepared for a single determination and which is a random sample from the test sample or conditioned sample as appropriate.

3. REFERENCES

CORESTA Recommended Method N° 66 : 2005
Determination of nicotine in the mainstream smoke of cigars by gas chromatographic analysis.

CORESTA Recommended Method N° 67 : 2005
Determination of water in the mainstream smoke of cigars by gas chromatographic analysis.

CORESTA Recommended Method N° 46 : 1998
Atmosphere for conditioning and testing cigars of all shapes and sizes.

CORESTA Recommended Method N° 64 : 2005

Routine analytical cigar-smoking machine - Specifications, definitions and standard conditions.

CORESTA Recommended Method N° 47 : 2000
Cigars - Sampling.

CORESTA Recommended Method N° 12 : 1968
Determination of alkaloids in cigarette smoke condensate

CORESTA Recommended Method N° 15 : 1990
Cigarettes – Determination of water in smoke condensates – Karl Fischer method

ISO 4387:2000(E)
Cigarettes – Determination of total and nicotine-free dry particulate matter using routine analytical smoking machine

Informative : ISO 2971 : 1998
Cigarettes and filter rods – Determination of nominal diameter – Method using a laser beam measuring apparatus

Note : this standard is currently being revised, and it is expected that its new title will no longer include the words "laser beam". Instead the words "non-contact optical" will be used.

4. PRINCIPLE

Sampling of the test cigars.

Conditioning of the test cigars.

Smoking of the test cigars on an automatic smoking machine with simultaneous collection of total particulate matter in a glass fibre filter trap.

Gravimetric determination of the mass of total particulate matter so collected.

Extraction of the total particulate matter from the trap for the determination of the water and nicotine contents by gas chromatography.

Note : In laboratories that are not in a position to use the gas-chromatographic methods, the determination of nicotine (as total alkaloids) should be performed by the spectrometric method, and the determination of water in smoke condensate should be performed by the Karl Fischer method. In such cases values obtained for nicotine and water in smoke condensate may be used with the addition of a note made in the expression of the result (see CORESTA Recommended Methods N° 12 and N° 15).

5. APPARATUS

Normal laboratory apparatus and in particular the following items:

5.1. Routine analytical cigar-smoking machine complying with the requirements of CORESTA Recommended Method N° 64 : 2005

5.2. Soap bubble flow meter, graduated at 100 ml with a resolution of 0,1 ml.

5.3. Apparatus for the determination of puff duration and frequency.

5.4. Analytical balance with a resolution of 0,1 mg.

5.5. Draw resistance testing equipment.

5.6. Conditioning enclosure carefully maintained in accordance with the conditions specified in CORESTA Recommended Method N° 46.

5.7. Length-measuring device, suitable for measuring to the nearest 0,5 mm.

5.8. Apparatus for the determination of diameter.

5.9. Sealing device - End caps made from the same non-hygroscopic and chemically inert material as the filter holder.

5.10. Gloves - Cotton or non-talc surgical.

6. SAMPLING

Provide a laboratory sample (see 2.7), by a sampling scheme such as one of those in CORESTA Recommended Method N° 47. This sample will normally contain cigars taken from different parts of the population. Make up the test sample required for the test by randomly selecting cigars from the different parts of the population represented in the laboratory sample.

7. DETERMINATION OF TOTAL PARTICULATE MATTER

In 7.1. and 7.6. below, the following symbols are used:

N is the number of cigars of a given type to be smoked, resulting from sampling at one point in time or from a sub-period sample.

C is a multiplying factor, value greater than 1, to allow for loss due to damage or selection procedures between initial sampling and smoking;

n is the number of replicate determinations of total particulate matter;

q is the number of cigars smoked into the same trap;

P is the total number of packets of cigars available;

Q is the total number of cigars available (laboratory sample, see 2.7).

7.1. *Preparation of the Cigars for Smoking*

If N cigars of a given type are to be smoked, C x N cigars should be prepared from Q for conditioning and butt marking. The multiplier C is usually at least 1,5 to provide extra cigars in case some are damaged and for moisture measurement. For the verification of the repeatability value (r) , C x N shall never be less than 24 (= 1,5 x 16) cigars.

The N cigars to be smoked will be tested in n = N/q determinations if q cigars are smoked into one trap. As far as possible these n determinations should correspond to different test portions of the test sample. Selection of each test portion will depend upon the form of the test sample.

7.1.1. Selection of test portions from a bulk of Q cigars.

If the test sample is in the form of a single bulk, consisting of Q cigars, C x N cigars

should be selected at random so that every cigar has an equal probability of being chosen.

7.1.2. Selection of test portions from P packets.

If the test sample consists of P packets, the selection procedure depends upon the number of cigars in each packet (Q/P) compared with q.

If $Q/P \geq C \times q$, select a test portion by choosing a single packet at random, then randomly select C x q cigars from that packet.

If $Q/P < C \times q$, select the smallest number of packets (k) such that :

$$Q \times k / P \geq C \times q$$

and randomly choose an equal (or as near equal as possible) number of cigars from each packet to form the test portion of C x q cigars.

7.1.3. Duplicate test portions.

Provided that the test sample is sufficiently large ($\geq 2\ C \times N$), it would be prudent to reserve a duplicate set of n test portions. In this event the parallel selection of a test portion and its duplicate would seem sensible. In this case the two selection conditions of 7.1.2 would need to be changed to :

$$Q/P \geq 2\ C \times q \quad \text{and} \quad Q/P < 2\ C \times q.$$

7.2. *Marking the butt length*

7.2.1. Standard butt length.

The standard butt length to which cigars shall be marked shall be the longest of :

- 33 mm, or
- inbuilt filter + 8 mm, or
- artificial mouthpiece + 17 mm

Note : Butt length is defined in CORESTA Recommended Method N° 64 : 2005 as the length of unburnt cigar, including filter and/or mouthpiece, remaining at the moment when smoking is stopped.

7.2.2. Butt Marking

The butt length should be marked on the cigar after conditioning.

It is recommended that two thin lines be drawn using a fine soft tipped marker. The first line at 28 mm to an accuracy of 0,5 mm from the mouth end of the cigar (corresponding to the standard depth of insertion : CORESTA Recommended Method N° 64 : 2005 clause 4.8) and the second at the standard butt length to an accuracy of 0,5 mm from the mouth end for the particular cigar type.

Care should be taken to avoid damaging the cigars during butt marking. Any cigars accidentally torn or punctured during marking, or any found during marking to be defective, shall be discarded and replaced with spare cigars.

7.3. *Selection of Cigars*

If a selection by mass or draw resistance (or any other parameter) is necessary because of the nature of the problem being studied, the selection is not to be considered as a method of reducing the number of cigars to be smoked.

7.4. *Conditioning*

Condition all the test portions in the conditioning atmosphere specified in CORESTA Recommended Method N° 46.

The testing atmosphere in the laboratory where the smoking is to be carried out shall also be in accordance with CORESTA Recommended Method N° 46.

Transfer the test portions to the smoking location in airtight containers (just large enough to contain the portions) unless the smoking location and the conditioning location are adjoining and have identical atmospheres.

7.5. *Preliminary Tests before Smoking*

Determine the following data which may be required in the test report.

7.5.1. Total length of the cigar.

7.5.2. Diameter of the cigar

7.5.2.1 For the determination of the cigar holder construction, measure the diameter of the cigar at 15 mm from the mouth end.

7.5.2.2 For the determination of the Puff Volume, measure the diameter at 33 mm from the mouth end.

7.5.2.3 Diameter measurement

- Definition of "Equivalent Diameter" :

The "equivalent diameter" is defined as :

"The diameter, in mm, as obtained from the direct diameter reading from the instrument, irrespective of the cross section of the cigar"

or

"The diameter, in mm, as obtained from the measurement of the circumference, in mm, divided by π – irrespective of the cross section of the cigar".

- Measurement of "equivalent diameter"

The measurement must be non-destructive.

The measurement is to be done on all cigars of the conditioned sample.

The instrument must be able to measure the equivalent diameter at 15 mm from the mouth end (for the determination of the cigar holder construction) and at 33 mm from the mouth end (for the determination of the puff volume).

Instruments using optical measurement techniques are to be preferred, as defined by ISO 2971 : 1998, measuring the shadow or silhouette of the product, whereby during measurement the cigar is rotating around its length-axis, preventing any eccentric motion of the cigar.

The instrument must be calibrated, following the manufacturer's instructions (see also Annex A and Annex B of ISO 2971 : 1998).

For both measurements, i.e. at 15 mm and 33 mm from the mouthend, the average result is calculated from the conditioned cigars, to the nearest 0,1 mm (see also 8.4).

Note : Instruments using other than optical measurement techniques can be used as well, provided their precision is similar (see also Annex C of ISO 2971 : 1998).

7.5.3. Draw resistance of the cigar.

7.5.4. Average mass of the conditioned cigars selected for the smoking operation (milligrams per cigar).

7.5.5. Water content (% mass/mass) of the conditioned cigars, with reference to the measurement technique.

7.6. *Smoking and Collection of Particulate Matter*

7.6.1. Smoking Plan :

7.6.1.1. For cigars with a diameter ≤ 12.0 mm :

"One single result for TPM, Water, Nicotine and NFDPM" is defined as the average result obtained from smoking 8 cigars, *i.e.* 2 cigars on 4 channels, at 2 cigars per filter pad, except when as consequence of exceeding the maximum TPM load of the filter trap, the cigars have to be smoked at 1 cigar per filter pad, in which case will apply the point 7.6.1.2.

Based on this definition of "one single result" , to obtain one replicate, 8 cigars shall be smoked on 4 channels, at 2 cigars per filter pad.

7.6.1.2. For cigars with a diameter ≥ 12.1 mm :

"One single result for TPM, Water, Nicotine and NFDPM" is defined as the average result obtained from smoking 8 cigars, i.e. 1 cigar on 8 channels, at 1 cigar per filter pad.

Based on this definition of "one single result" , to obtain one replicate, 8 cigars shall be smoked on 8 channels, at 1 cigar per filter pad.

Note : the 8 channels do not necessarily need to be 8 *different* channels, as to allow the use of smoking machines containing less than 8 channels.

7.6.2. Preparation of Smoke Traps and Cigar Holders

For all operations the operator shall prevent contamination from the fingers by wearing gloves of a suitable material.

Insert into their holders filter discs which have been conditioned in the test atmosphere for at least 12 h, and assemble placing the rough side of the filter disc so that it will face the oncoming smoke. After assembly, examine the filter holders to ensure that the discs have been properly fitted.

Following CORESTA Recommended Method N° 64 : 2005, 4.8, select the appropriate bobbin, sleeve and endseal (washer) to compose Cigar Holder Construction Type 1 or Cigar Holder Construction Type 2.

Weigh the assembled smoke traps, including the sealing devices (5.9), to the nearest 0,l mg. Due to absorption of water by smoke traps and solvent, determine a value for the sample blank. Prepare sample blanks by treating additional smoke traps in the same manner as that used for smoke collection.

7.6.3. Setting up the Smoking Machine.

If necessary replace any protective filters on the machine. Switch on and allow to warm up on automatic cycling for at least 20 min.

With the machine warmed up, check that the puff duration and puff frequency on each channel are in accordance with the standard conditions.

7.6.3.1. Measurement of puff duration.

A calibrated timer shall be used to measure the period of time which elapses between the triggering operations which begin and end a puffing action of the smoking machine. The accuracy of the timing device shall be such as to ensure that a 1% error in the puff duration can be detected. The timer should be coupled directly to the triggering circuits.

Note : It is not possible to specify the method of measurement beyond a statement of principle because of the variety of types of suitable timers and smoking machines available.

7.6.3.2. Checking of puff frequency.

Measure the period of time which elapses between the triggering operations which begin successive puffing actions of the smoking machine. This will determine the puff frequency. The calibrated timer used shall be suitable for measuring to the nearest 0,l s. It should, preferably, be coupled directly to the triggering circuits.

7.6.3.3. Setting and Measurement of Puff Volume.

Depending on the diameter of the product to be smoked, set the Puff Volume of the channels of the smoking machine to the required volume, as described in CORESTA Recommended Method N° 64 : 2005, clause 4.3.

The displacement of the bubble in a soap bubble flow meter gives a direct measurement of puff volume and also provides a check for leaks in the system.

A suitable indicator, graduated at 100 ml, shall have a resolution of 0,l ml. It shall be connected through a standard pressure drop device of 1 kPa ± 5 % to the cigar holder of the smoking machine channel under test. Before use on a series of measurements the instrument shall be wetted twice with detergent solution and then allowed to drain for a period of between 30 s and 45 s.

The bubble flow meter shall contain a 15% aqueous solution of a surface active agent. Teepol L® has been found to be satisfactory.

Fit the prepared smoking trap or traps and cigar holders onto the machine. Prepare the soap bubble flow meter by wetting the inside of the tube with the detergent solution to above the top graduation mark. Connect the bubble flow meter to the cigar holder in port 1 and determine the puff volume, adjust if necessary to the volume required ± 0,3 ml.

Repeat for all remaining ports in turn.

Replicate determinations shall be made until the necessary precision of measurement is obtained. If the number of replicates exceeds three then continue until the correct precision is obtained but replace the pad before smoking, reweigh the smoke trap and recheck the puff volume with the new pad in place.

Measure the temperature and relative humidity of the air surrounding the smoking machine and note the atmospheric pressure.

7.6.4. Procedure for Smoking Run.

Insert the conditioned cigars into the cigar holders so that the butt-end of the cigar is sealed airtight by the endseal. Avoid any leaks or deformations. Any cigars found to have obvious defects, or which have been damaged during insertion, shall be discarded and replaced with spare conditioned cigars.

Ensure that the cigars are positioned correctly so that the angle formed by the longitudinal axis of the cigar and the horizontal plane shall be as small as possible; it shall not exceed 10° if the centre of the butt end is lower than the centre of the other end and 5° if the centre of the butt end is higher than the centre of the other end. The axis of the cigars shall coincide with the axis of the ports.

When a puff termination device is used, adjust the position of each cigar so that when the burning coal reaches the butt mark, the puff termination device is activated. If the burning through of 100 % cotton, (48 ± 4) tex thread is used to terminate smoking at the butt mark, the cotton shall just touch the cigars at the butt mark, without modifying the cigar positioning.

When, due to the shape of the cigar, a puff termination device can not be used, smoking shall be terminated manually when the coal reaches the butt mark. (See CORESTA Recommended Method N° 64 : 2005, 5.3.7).

When, due to the shape of the cigar, the automated lighting of the smoking machine can not be used, a hand held electrical lighter or conventional gas lighter may be used.

Zero the puff counters and light each cigar at the beginning of its first puff. Should it be necessary to light a cigar more times, or when relighting during the smoking run is required, a hand held electrical lighter or conventional gas lighter may be used at the next puff(s). When each butt mark has been reached, remove the burning coal by scissors and note the final reading of the puff counters. Then take at least one clearing puff, preferably immediately after the finishing of the smoking of the cigar. After the smoking process is complete leave the cigar butt in place for at least 30 s to enable deposition of any residual smoke in the trap.

Notes :

1. Avoid disturbance of the smoking by artificial removal of ash. Allow ash to fall naturally into the ash tray.
2. Rotating the filter holder is allowed when the cigar is burning unevenly, up to the decision of the experienced operator. After rotation of the filter holder, the support bar can not be repositioned, and therefore will lose its function.

If required, new cigars shall be inserted immediately and the smoking process repeated until the predetermined number of cigars, according to the smoking plan, has been smoked into the smoke trap. Begin the determination of total particulate matter as described in 7.7 immediately.

7.7. *Determination of Total Particulate Matter*

Wearing gloves remove the smoke traps from the smoking machine. Where necessary, remove the cigar holder from the smoke trap.

Cover the front and back apertures of the trap with the sealing devices (5.9). It is recommended that the removal of the holder be conducted with the smoke trap held with

its cigar - facing side downwards to avoid any possible contaminants from the cigar holder reaching the filter disc.

Immediately after smoking weigh the smoke traps, including the sealing devices, to the nearest 0,1 mg.

Check the back of each filter disc to ensure that there are no brown stains indicating overloading or pad damage. Discard any disc showing such stains or damage. (55 mm glass fibre filter pads are capable of retaining a limited amount of total particulate matter (TPM). By interpolation it can be calculated that the maximum TPM load of the 55 mm filter trap is 200 mg. If, after smoking, brown staining at the back of the filter is found, the disc should be discarded.)

7.8. *Calculation of Total Particulate Matter*

The mean mass per cigar of total particulate matter, T, for each channel, expressed in milligrams per cigar, is given by the equation:

$$T = (m_1 - m_0) / q$$

where :

m_0 is the mass of the smoke trap before smoking (in milligrams)

m_1 is the mass of the smoke trap after smoking (in milligrams)

q is the number of cigars smoked into the trap.

7.9. *Treatment of Total Particulate Matter*

7.9.1. Extraction procedure.

Wearing gloves, remove the sealing devices from the smoke trap, open it and remove the filter disc with forceps. Fold it twice, total particulate matter inwards, being careful to handle only the edge with forceps and gloved fingers. Place the folded disc in a dry conical flask (maximum 150 ml for 55 mm discs). Pipette solvent [propan-2-ol containing the internal standards for both nicotine and water determinations] into the flask [20 ml for 55 mm discs]. [CORESTA Recommended Method N° 66 : 2005 and CORESTA Recommended Method N° 67 : 2005].

Wipe the inner surface of the filter holder front with two separate quarters of an unused conditioned filter disc and add these to the flask. Stopper the flask immediately and shake gently on an electric shaker for at least 20 minutes, ensuring that the disc does not disintegrate. The shaking time should be adjusted to ensure full extraction of the nicotine and water in the particulate matter.

Follow the same procedure with each of the blank smoke traps used for the determination of water.

7.9.2. Determination of Water and Dry Particulate Matter

Water in the supernatant solution in each flask is determined according to CORESTA Recommended Method N° 67 : 2005.

The dry particulate matter, D, is calculated for each trap from the equation:

$$D = T - W$$

where :

D is the dry particulate matter, in milligrams per cigar;

T is the total particulate matter, in milligrams per cigar;

W is the water content in the total particulate matter, in milligrams per cigar.

7.9.3. Determination of Nicotine and Nicotine-free Dry Particulate Matter

Nicotine in the supernatant solution in each flask is determined according to CORESTA Recommended Method N° 66 : 2005.

The nicotine-free dry particulate matter, G, is calculated for each trap from the equation:

$$G = D - H_{nic}$$

where

G is the nicotine-free dry particulate matter, in milligrams per cigar;

D is the dry particulate matter, in milligrams per cigar;

H_{nic} is the nicotine in the total particulate matter, in milligrams per cigar.

8. 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 8.1 to 8.4.

8.1. *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.

8.2. *Sampling*

- Type of sampling procedure.
- Number of cigars in laboratory sample.
- Date and location of purchase.

8.3. *Description of Test*

Date of test.

- Type of smoking machine used.
- Type of smoke trap used.
- Type of instrument used for diameter measurement
- Total number of cigars smoked in the entire determination on that cigar type.
- Number of cigars smoked into each smoke trap.
- Butt length.
- Room temperature (°C) during smoking operation.
- Relative humidity (%) during smoking operation.
- Atmospheric pressure (kPa) during smoking operation.

8.4. *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.

- Average length of the cigars to the nearest 0,5 mm.
- Average length of the filter (where applicable) to the nearest 0,5 mm.
- Average length of the tip (where applicable) to the nearest 0,5 mm.
- Butt length to which cigars were smoked to the nearest 0,5 mm.
- Average length of tobacco portion smoked to the nearest 0,5 mm.
- Average diameter of the cigars at 15 mm from mouth end, to the nearest 0,1 mm (for the determination of the holder construction).
- Average diameter of the cigars at 33 mm from mouth end, to the nearest 0,1 mm (for the determination of the Puff Volume).
- The Puff Volume, corresponding with the diameter at 33 mm from the mouth end, to the nearest 1 ml.
- Average draw resistance of the conditioned cigars.
- Average mass (milligrams per cigar) of the conditioned cigars selected for the smoking operation.
- Water content (% mass/mass) of the conditioned cigars.
- Average number of puffs per cigar for each channel to the nearest 0,1 puff.
- Total particulate matter (TPM, in milligrams per cigar) for each channel to the nearest 0,1 mg and the average per cigar to the nearest 1 mg.
- Dry particulate matter (DPM, in milligrams per cigar) for each channel to the nearest 0,1 mg and the average per cigar to the nearest 1 mg.
- Nicotine-free dry particulate matter (NFDPM, in milligrams per cigar) for each

channel to the nearest 0,1 mg and a range of two values expressed in mg per cigar to the nearest 1 mg/cigar where the minimum is the average minus two times the standard deviation and the maximum is the average plus two times the standard deviation.

- Nicotine-free dry particulate matter (NFDPM, in mg per g) for each channel to the nearest 0,1 mg/g, dividing the NFDPM (in mg per cigar) by the cigar mass (in g per cigar) of the conditioned cigars selected for the smoking in each particular channel and a range of two values expressed in mg per g to the nearest 1 mg/g where the minimum is the average minus two times the standard deviation and the maximum is the average plus two times the standard deviation.

9. REPEATABILITY AND REPRODUCIBILITY

Collaborative Studies involving between 10 and 15 laboratories conducted in 2003/2004 found the following values for repeatability (r) and reproducibility (R) of this method.

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/cigar) as summarised in the following table 1:

Product	weight g	diameter mm	length mm	NFDPM mg/cigar	r mg/cigar	R mg/cigar	r %	R %
O	0,95	8,5	75,0	22,9	2,4	6,3	10	28
P	1,36	9,0	87,7	36,7	3,3	9,7	9	26
Q	1,60	9,1	99,0	47,0	4,6	13,2	10	28
K	2,11	11,1	88,0	40,7	3,3	17,2	8	42
M	3,25	14,6	95,0	55,0	6,1	17,1	11	31
R	5,10	13,7	129,5	79,1	9,5	27,6	12	35
N	6,60	15,5	127,0	66,8	8,9	28,7	13	43
S	9,60	17,0	164,0	77,8	16,9	48,0	22	62
T	11,27	17,5	162,0	88,8	24,8	79,1	28	89
C1	14,28	18,3	174,0	106,0	24,0	69,1	23	65

Data analysis gave the estimates (expressed in mg/g) as summarised in the following table 2:

Product	weight g	diameter mm	length mm	NFDPM mg/g	r mg/g	R mg/g	r %	R %
O	0,95	8,5	75,0	24,3	2,9	7,4	12	31
P	1,36	9,0	87,7	28,5	2,6	8,1	9	28
Q	1,60	9,1	99,0	31,0	3,2	9,5	10	31
K	2,11	11,1	88,0	20,4	1,9	8,6	9	42
M	3,25	14,6	95,0	17,3	2,2	5,6	13	32
R	5,10	13,7	129,5	16,9	2,2	6,8	13	40
N	6,60	15,5	127,0	11,2	2,3	6,1	21	55
S	9,60	17,0	164,0	8,7	1,7	3,8	20	44
T	11,27	17,5	162,0	8,8	2,6	5,2	30	59
C1	14,28	18,3	174,0	8,3	1,8	4,1	21	50

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 mean yield obtained from smoking 8 cigars, *i.e.* : 2 cigars on 4 channels, at two cigars per filter pad for Product O, P and Q.

For Products K, M, R, N, S, T and C1 one "single" result equally was obtained from smoking 8 cigars, but at 1 cigar per filter pad.

The subject of tolerances due to sampling is dealt with in CORESTA Recommended Method N° 47 : 2000.

(NFDPM = nicotine-free dry particulate matter)