

Cooperation Centre for Scientific Research Relative to Tobacco

Cigar Smoking Methods Sub-Group

CORESTA Recommended Method No. 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

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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

(August 2019)

0. INTRODUCTION

Cigars are manufactured in a wide range of sizes and shapes and may be fully or partially machine-made or handmade. Cigars range in length from approximately 65 mm to over 250 mm and in weight from less than 1 g to over 15 g. Diameters typically vary from 6 mm to over 25 mm. Cigar filler may vary in particle size and tobacco types used. This may impact pressure drop and smoking characteristics. Binders and wrappers may be constructed of natural leaf or of reconstituted tobacco sheet materials. Natural leaf binders and wrappers may vary extensively among samples of a given product for thickness, porosity, etc.

Due to the wide design characteristics for this product category, the Cigar Smoking Methods Sub-Group recognised the need to adapt smoking parameters as a function of the size of the cigar to be smoked. From the parameters available (puff volume, puff duration, puff period 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. These parameters are not intended to reflect human smoking patterns but are simply meant to provide a means for meaningful analytical testing

Thus a series of CORESTA Recommended Methods (CRMs) have been developed for use with machine smoking of cigar products. These CRMs are applicable to many commercially available products. Same level of deviations may be necessary in order to accommodate products not available or not considered at the time of development of these CRMs.

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

- At the time this method was published, a standardized method for the measurement of cigar diameter has not been developed. Non-cylindrical cigars present a challenge when estimating diameter.
- Available filter traps may not be of sufficient capacity for smoking an entire cigar on a single trap for larger cigars.
- The current method is not validated for handmade cigars. Collaborative studies intended to document repeatability and reproducibility have not included such products. This very diversified product category includes many formats, shapes and sizes which cannot be accommodated for testing applying this method.

• It is not possible to establish repeatability and reproducibility (r&R) values across the full range of product designs due to the diversity in product styles, shape, and size. Thus, researchers testing products outside of the total particulate matter (TPM) loading and/or design characteristics included in this method should be mindful of this limitation in information.

No machine smoking regime can represent all human smoking behaviour:

- It is recommended that cigars also be tested under conditions of a different intensity of machine smoking than those specified in this Recommended Method;
- Machine smoking testing is useful to characterize cigar emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;
- Smoke emission data from machine measurements may be used as inputs for product hazard
 assessment, but they are not intended to be nor are they valid as measures of human
 exposure or risks. Communicating differences between products in machine measurements
 as differences in exposure or risk is a misuse of testing using ISO standards and this
 CORESTA Recommended Method.

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. NORMATIVE REFERENCES

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

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

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

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

CORESTA Recommended Method N° 47, Cigars - Sampling

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 in the smoke 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 nicotine content, expressed as milligrams per cigar.

3.4 Smoking Process

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

3.5 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.

3.6 Laboratory Sample

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

3.7 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.

3.8 Conditioning Sample

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

3.9 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.

4. PRINCIPLE

The test cigars are sampled then conditioned. The test cigars are smoked on a routine analytical smoking machine in a conditioned atmosphere with collection of total particulate matter on a glass fibre filter trap. The mass of the total particulate matter so collected is determined gravimetrically. The total particulate matter is extracted from the trap for determination of the water and nicotine contents, typically, by gas chromatography.

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.
- **5.2** Soap bubble flow meter, graduated in the range of volumes to be measured with a resolution of 0,2 ml.
- **5.3** Apparatus for the determination of puff duration and frequency.

5.4 Analytical balance suitable for measuring to the nearest 0,1 mg.

The weighing of filter pad holders may be affected by static electricity, necessitating the use of an antistatic device.

- **5.5** Conditioning enclosure maintained under the conditions specified in CORESTA Recommended Method N° 46.
- **5.6** Length-measuring device, suitable for measuring to the nearest 0,5 mm.
- **5.7** Apparatus for the determination of diameter where non-contact optical is the preferred technology.
- **5.8** Smoke Trap Sealing device End caps made from a non-hygroscopic and chemically inert material.
- **5.9** Gloves Cotton or non-talc laboratory gloves.

6. SAMPLING

A laboratory sample (3.6) shall be taken by a sampling scheme such as one of those given in CORESTA Recommended Method N° 47. This sample will normally contain cigars taken from different parts of the population. Make up the test sample (3.7) 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 3.7).

7.1 Preparation of the Cigars for Smoking

If N cigars of a given type are to be smoked, $C \times N$ 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 optional tests that may be.

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

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

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

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

7.1.3 Duplicate test portions

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

$$Q/P \ge 2 C \times q$$
 and $Q/P < 2 C \times q$

7.2 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.

7.3 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.

7.4 Marking the cigar product

7.4.1 Standard butt length

The standard butt length to which cigars shall be marked shall be the greatest of the following three lengths:

- 33 mm after cutting if necessary, or
- filter $+ 8 \, \text{mm}$, or
- artificial mouthpiece + 17 mm

For "V-cut" cigars, the length of the "V" is included in the butt length.

Cigars with a closed mouth end shall be cut straight at 5 mm from the mouth end. The length of the removed part is not included in the butt length.

7.4.2 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 should be drawn at the insertion depth (corresponding to the typical insertion depth: CORESTA Recommended Method N° 64) and the second line should be drawn at the standard butt length to an accuracy of 0,5 mm from the mouth end for the particular cigar type as calculated under 7.4.1.

Care should be taken to avoid damaging the cigars during 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.

7.5 Preliminary Tests before Smoking

Determine physical data, some of which is required for correct configuration of the smoking machine and all of which is required for subsequent inclusion in the test report (see also 8.4).

7.5.1 Cigar diameter data

Cigar diameter data is required to correctly configure the smoking machine and for inclusion in the test report (see also 8.4).

NOTE: Diameter measurement will typically employ a non-contact optical measurement technique such as defined in ISO 2971. Alternative measurement techniques may be used if they deliver the required precision and a note is made in the test report.

- **7.5.1.1** For the determination of the cigar holder component dimensions, measure the diameter of each conditioned cigar at 15 mm from the mouth end. The average result shall be calculated and reported to the nearest 0,1 mm. Typically, this average value will be used to determine the holder dimensions for all test articles to be measured. Additional judgement may be needed if the range in diameter for the test set is greater than 1 mm 2 mm.
- **7.5.1.2** For the determination of the puff volume, measure the diameter of each conditioned cigar at 33 mm from the mouth end. The average result shall be calculated and reported to the nearest 0,1 mm.

7.5.2 Additional physical data

Additional physical data may be determined for the test report. Measurements shall be made using a method appropriate to the stated precision. The measurement technique shall be referenced where this is required for the test report (see also 8.4).

- Average length of the cigar to the nearest 0,5 mm
- Average length of the filter (where applicable) to the nearest 0,5 mm
- Average length of the artificial mouthpiece (where applicable) to the nearest 0,5 mm

- Average draw resistance of the conditioned cigars to the nearest 10 kPa (1 mmWG)
- Average mass of the conditioned cigars selected for the smoking operation (milligrams per cigar to the nearest milligram)
- Water content (as a mass fraction) of the conditioned cigars to the nearest 1 %, with reference to the measurement technique

7.6 Smoking and Collection of Particulate Matter

7.6.1 For cigars with a diameter \leq 12,0 mm

"One single result for TPM, Water, Nicotine and NFDPM" is defined as the result obtained from smoking 1 cigar or the average result obtained from smoking 2 cigars.

7.6.2 For cigars with a diameter > 12,0 mm

"One single result for TPM, Water, Nicotine and NFDPM" is defined as the result obtained from smoking 1 cigar.

NOTE: Overloading the capacity of the glass fiber filter should be avoided.

7.6.3 Smoking Plan:

Choose a smoking plan. The plan shall show the number of cigars to be smoked into each trap (q) and the number in the conditioning sample $(C \times N)$.

- **7.6.3.1** Typically, for cigars with a diameter ≤ 12,0 mm, 1 or 2 cigars per trap are smoked, except when as a consequence of exceeding the maximum TPM load of the smoke trap. When possible, the size of the smoke trap is chosen to allow for 1 or 2 cigars per trap without overload.
- **7.6.3.2** Typically, for cigars with a diameter > 12,0 mm, 1 cigar per trap is smoked. When possible, the size of the smoke trap is chosen to allow for 1 cigar per trap without overload. In cases where this is not possible, segmented puff collection may be required; use of multiple traps may be needed.

NOTE: Collaborative studies used to establish r&R values have defined one replicate collection as 8 cigars regardless of the number of cigars/traps required for smoking.

7.6.4 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 the smoke traps 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, select the appropriate Cigar Holder.

Weigh the assembled smoke traps to the nearest 0,1 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.

7.6.5 Setting up the smoking machine

If necessary replace any protective filters on the machine. Switch on the smoke machine and allow it to warm up according to manufacturer requirements.

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

7.6.5 Measurement of puff duration

A certified 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.

7.6.6 Checking of puff period

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

7.6.7 Setting and measurement of puff volume

Set the puff volume as required according to CORESTA Recommended Method N° 64.

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 shall have a resolution of 0,2 ml. It shall be connected through a standard pressure drop device of 1 kPa \pm 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.

It is recommended to use the detergent solution as specified by the supplier of the soap bubble flow meter in the corresponding manual.

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 ± 0.6 ml.

NOTE: The puff volume tolerance is determined based on resolution of the measuring device multiplied by a factor of 3. For this reason, it may be appropriate to use tolerance that is less than \pm 0,3 ml for lower puff volumes.

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 necessary 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.

7.6.8 Procedure for Smoking Run

Insert the conditioned cigars into the cigar holders so that the butt-end of the cigar is sealed airtight. 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 axes of the cigars shall coincide with the axes of the ports and ensure that the cigar is held within -10° to $+5^{\circ}$ of the cigar holder and smoke trap axis.

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 cigar at the butt mark, without modifying the cigar positioning.

When, due to the shape of the cigar, a puff termination device cannot be used, smoking shall be terminated manually when the coal reaches the butt mark.

When, due to the shape of the cigar, the automated lighting of the smoking machine cannot be used, a hand held electrical lighter or conventional gas lighter or torch may be used. A hand held electrical lighter is preferred.

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 or torch may be used at the next puff(s). A hand held electrical lighter is preferred. When each butt mark has been reached, remove the burning coal 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.

NOTE: When practical, avoid disturbance of the smoking by artificial removal of ash. Allow ash to fall naturally into the ash tray. In some cases, the amount of ash may be so large as to cause extinguishing of the cigar if left in place. In this case, it is not practical to leave the ash undisturbed. Laboratories should determine if ash removal is required and if so, ash removal should be done in a consistent manner and documented in the study report. It may also be necessary to remove the ash when relighting the cigar is necessary.

The cigar holder may be rotated when the cigar is burning unevenly.

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

7.7 Determination of Total Particulate Matter

Remove the cigar from the cigar holder followed by removing the cigar holder and smoke trap from the smoke machine while wearing gloves. Where necessary, remove the cigar holder from the smoke trap. 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.

Cover the front and back apertures of the trap with the sealing devices (5.8).

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.

7.8 Calculation of Total Particulate Matter

The mean mass per cigar of total particulate matter, m_{TPM} , for each channel, expressed in milligrams per cigar, is given by the equation

$$m_{\rm TPM} = \frac{(m_1 - m_0)}{q}$$

where:

m₀ is the mass of the smoke trap before smoking (in milligrams)

m₁ 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 for example). Pipette solvent (propan-2-ol containing the internal standards for both nicotine and water determinations) into the flask (20 ml for 55 mm discs) (See CORESTA Recommended Method N° 66 and CORESTA Recommended Method N° 67).

NOTE: The extraction volume can be adjusted if larger filter pads are used (e.g. 92 mm pad and 50 ml extraction solution).

Each trap should be extracted separately. 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 solution in each flask is determined according to CORESTA Recommended Method N° 67.

The dry particulate matter, $m_{\rm DPM}$, is calculated for each trap from the equation:

$$m_{\rm DPM} = m_{\rm TPM} - m_{\rm W}$$

where:

 m_{TPM} is the total particulate matter, in milligrams per cigar;

 $m_{\rm W}$ is the water content in the total particulate matter, in milligrams per cigar.

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).

7.9.3 Determination of Nicotine and Nicotine-free Dry Particulate Matter

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

The nicotine-free dry particulate matter, m_{NFDPM} , is calculated for each trap from the equation:

$$m_{\text{NFDPM}} = m_{\text{DPM}} - m_{\text{N}}$$

where:

 $m_{\rm DPM}$ is the dry particulate matter, in milligrams per cigar;

 $m_{\rm N}$ 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

- a) type of sampling procedure
- b) number of cigars in laboratory sample
- c) date and location of purchase

8.3 Description of Test

- a) date of test
- b) type of smoking machine used
- c) type and size of smoke trap used
- d) type of instrument used for diameter measurement
- e) total number of cigars smoked in the entire determination on that cigar type
- f) number of cigars smoked into each smoke trap
- g) butt length
- h) room temperature (°C) during smoking operation
- i) relative humidity (%) during smoking operation
- j) 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.

- a) average length of the cigars to the nearest 0,5 mm
- b) average length of the filter (where applicable) to the nearest 0,5 m
- c) average length of the tip (where applicable) to the nearest 0,5 mm
- d) butt length to which cigars were smoked to the nearest 0,5 mm
- e) average length of tobacco portion smoked to the nearest 0,5 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) the puff volume, corresponding with the diameter at 33 mm from the mouth end, to the nearest 1 ml
- i) average draw resistance of the conditioned cigars, if determined
- j) average mass (milligrams per cigar) of the conditioned cigars selected for the smoking operation
- k) water content (% mass/mass) of the conditioned cigars, if determined
- 1) average number of puffs per cigar to the nearest 0,1 puff
- m) 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
- n) nicotine-free dry particulate matter (NFDPM), in milligrams per cigar, for each channel to the nearest 0,1 mg and the average per cigar to the nearest 1 mg
- o) number of relights required and technique for relighting
- p) deviations and/or anomalies of note

9. REPEATABILITY AND REPRODUCIBILITY

Collaborative Studies involving 8 or 9 laboratories conducted in 2018 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 NFDPM/cigar) as summarised in the following Table 1:

Product	weight mg	diameter mm	NFDPM mg/cigar	r mg/cigar	R mg/cigar	r %	R %
А	2557	8,9	60,5	12,76	17,17	21,10 %	28,40 %
В	3287	9,6	49,1	13,74	34,49	28,00 %	70,30 %
С	4049	9,6	57,1	18,6	29,69	32,50 %	52,00 %
D	2754	10,4	62,5	14,75	17,89	23,60 %	28,60 %
Е	2774	10,4	60,9	11,45	16,3	18,80 %	26,80 %
F	3026	~12	53,9	23,15	23,99	42,90 %	44,50 %
G	7721	14-15	70,7	23,15	38,02	32,70 %	53,80 %
Н	8254	17	92,1	52,5	69,64	57,00 %	75,60 %
I	960	7,8	12,3	2,02	3,38	16,40 %	27,40 %

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 equally 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.

10. BIBLIOGRAPHY

- [1] CORESTA Recommended Method N° 12, Determination of alkaloids in cigarette smoke condensate
- [2] CORESTA Recommended Method N° 15, Cigarettes Determination of water in smoke condensates Karl Fischer method
- [3] ISO 2971, Cigarettes and filter rods Determination of nominal diameter Method using a non-contact optical measuring apparatus