

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

#### **Heated Tobacco Products Task Force**

# CORESTA Recommended Method No. 100

# DEFINITIONS AND STANDARD CONDITIONS: AEROSOL GENERATION AND COLLECTION FOR CARBON HEATED TOBACCO PRODUCTS

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#### CORESTA RECOMMENDED METHOD Nº 99

Title:

## DEFINITIONS AND STANDARD CONDITIONS: AEROSOL GENERATION AND COLLECTION FOR CARBON HEATED TOBACCO PRODUCTS

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#### CORESTA RECOMMENDED METHOD N° 100

### DEFINITIONS AND STANDARD CONDITIONS: AEROSOL GENERATION AND COLLECTION FOR CARBON HEATED TOBACCO PRODUCTS

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#### 1. INTRODUCTION

This method includes the requirements found necessary for the generation and collection of aerosol from carbon-heated tobacco products (cHTP) for analytical testing purposes. This method is based on the findings reported in the CORESTA Heated Tobacco Products (HTP Task Force Technical Report, 2020: Heated Tobacco Products (HTPs): Standardized Terminology and Recommendations for the Generation and Collection of Emissions<sup>[1]</sup>.

#### 2. FIELD OF APPLICATION

This method: - defines the parameters and specifies the standard conditions for the routine analytical generation and collection of aerosol from cHTPs; - specifies technical requirements for the routine analytical machine for generation and collection of cHTP emissions, termed as "machine" in this document, complying with the standard conditions stated within; - does not specify aerosol trapping nor subsequent sample preparation and analytical methods for analyses of components in the trapped aerosol or the gas phase; - may also be used for products other than defined in 4.15 if a specific testing requirement references this method.

#### 3. NORMATIVE REFERENCES

- **3.1** ISO 20778: Cigarettes Routine analytical cigarette smoking machine Definitions and standard conditions with an intense smoking regime
- **3.2** ISO 3402: *Tobacco and tobacco products Atmosphere for conditioning and testing*
- **3.3** ISO 3308: Routine analytical cigarette-smoking machine Definitions and standard conditions

#### 4. TERMS AND DEFINITIONS

For the purposes of this recommended method the following terms and definitions apply.

#### 4.1 Conditioning atmosphere

Atmosphere in which the carbon Heated Tobacco Products (cHTPs) are kept before being subjected to test.

#### 4.2 Test atmosphere

Atmosphere to which the cHTP is exposed throughout the test.

#### 4.3 Puff number

Number of puffs collected from an cHTP.

#### 4.4 Puff termination

Termination of the connection of the cHTP to the suction mechanism.

#### 4.5 Carbon Heated Tobacco Product (cHTP)

A product containing a tobacco substrate that is heated by smouldering carbon in order to produce a nicotine-containing aerosol.

#### 4.6 Tobacco substrate

Material (substrate) that contains processed tobacco and may contain aerosol generation agents, flavourings, and other ingredients.

#### 4.7 Sample holder

Device for connecting the cHTP to the port of the machine during aerosol generation and collection.

#### 5. STANDARD CONDITIONS

The standard conditions described in clauses 4.1 - 4.5 of ISO 20778: 2018 shall be followed with the substitution of the phrase "mouth end of the CHTP" in place of "butt end of the cigarette".

#### 6. SPECIFICATION OF THE MACHINE

#### 6.1 General

The machine shall comply with the conditions specified in clauses 5.1 - 5.4.7, 5.5 and 5.8 of ISO 20778: 2018 with the following substitutions:

Replace "smoking machine" with "machine"

Replace "cigarette" with "sample"

Replace "butt of the cigarette" with "mouth end of the cHTP"

Replace "cigarette holders" with "sample holders"

Replace "smoke" with "aerosol"

Replace "cigarettes are smoked" with "samples are tested"

Replace "smoking enclosure" with "enclosure around the machine"

Replace "sidestream smoke" with "sidestream emissions".

#### 6.2 Sample holder (see 4.7)

The design of the sample holder is such that it shall connect the sample to the port of the machine in a leak-free manner. It shall be impermeable to air and HTP aerosol.

**NOTE**: For samples with a diameter between 4,5 mm and 9 mm the cigarette/sample holder described in ISO 3308 can be used without the neoprene washer.

#### 7. SAMPLE CONDITIONING AND TESTING

#### 7.1 Sample conditioning and handling

The cHTP samples shall be temperature and humidity conditioned prior to analysis as described in ISO 3402. In summary, this is as follows:

Conditioning atmosphere :

Temperature  $(22 \pm 1)$  °C

Relative Humidity ( $60 \pm 3$ ) %

In case the samples are presented in resealable packaging with user instructions indicating the need to reseal the pack after opening, samples should be conditioned in sealed packs. Samples should be removed from the pack immediately prior to testing. Samples removed from open packs may be kept in sealed containers for a maximum of 4 hours; after this time they should no longer be regarded as suitable for testing and must be discarded. This period may be extended if the testing laboratory is able to demonstrate that there is no influence on yields.

#### Conditioning duration:

In current practice, a duration of 48 hours minimum to 240 hours maximum has been found to be sufficient for loose samples using forced airflow. In all cases, it should be verified that equilibrium has been properly attained as follows:

Equilibrium shall be considered to be attained either:

- a) when the relative variation of the mass of the sample or test pieces is not greater than 0.2 % in 3 h; or,
- b) when the sample or the test pieces, placed in a closed container of volume similar to that of the sample, give rise to a relative humidity in the container equal to that of the conditioning atmosphere.

#### 7.2 Vent blocking

Vent blocking may be applied to cHTPs in order to provide a more intense testing scenario. Vent blocking should not be applied if (a) ventilation holes in the product 'filter' section can not be occluded in normal use or (b) vent blocking compromises the operation of the product (for example, air inlet holes at the carbon heat source end of the product are not 'vents' and must not be occluded in testing).

Where blocking of the filter ventilation holes is required, this may be accomplished by two approaches:

A modified sample holder which fully occludes the ventilation holes.

Sealing of the ventilation holes with tape: a 10 mm to 20 mm wide cellophane tape shall be applied (manually or with an over-tipping machine) around the entire circumference of the cHTP consumable, with the end of the tape not extending beyond the mouth end. If the position of the ventilation holes is not known, it shall be verified that the tape covers them. No wrinkles or air holes shall appear. The tape shall circle the cHTP consumable once with a small overlap.

#### 7.3 Test atmosphere

The test atmosphere shall be controlled to ensure that all the samples are tested under identical conditions with regard to ambient air flow as described in clause 6 of ISO 20778: 2018 with the following substitutions:

Replace "smoking machine" with "machine"

Replace "cigarette" with "sample"

Replace "butt of the cigarette" with "mouth end of the cHTP"

Replace "cigarette holders" with "sample holders"

Replace "smoking run" with "test run"

The temperature and relative humidity of the test atmosphere shall correspond to those specified in ISO 3402:

— Temperature:  $(22 \pm 2)$  °C

— Relative humidity:  $(60 \pm 5)$  %.

**NOTE**: The atmospheric pressure should be within the range 96 kPa  $\pm$  10 kPa. The pressure shall be measured and included in any test report.

#### 7.4 Ignition and initiation of collection

The start of collection should be commenced upon ignition of the carbon tip, and it is recommended that an electrical lighting system is used. If electrical lighters are used they must be preheated prior to puff initiation and the lighter then brought within 1 mm of the carbon heat source.

If user instructions specify a particular ignition method, this should be followed and the deviation recorded on the test report.

#### 7.5 Termination of collection

In order to determine the puff number to be used when testing a cHTP sample, a pre-test must be conducted for each production batch or set of samples.

In this test, the number of puffs during which the heat source emits light (i.e., glows "red" during puffing) is determined for twenty (20) individual cHTP samples. The puff number to be used for testing is calculated as the average result from the twenty individual cHTP samples, rounded to the next higher integer value, with three (3) additional "residual heat" puffs added in order to ensure any aerosol yielded due to residual heat is captured.

In case there is no visible indication of puffing or smouldering from the carbon heat source, puff number may be determined by pre-test, by the following method:

Aerosol collected mass (ACM) must be determined on a puff-by-puff basis for twenty (20) individual cHTP samples. The final puff is defined by ACM yield falling below 10 % of maximum observed ACM/puff for that sample. The puff number to be used for testing is calculated as the average result from the twenty individual cHTP samples, rounded to the next higher integer value.

Alternative methods for determining final puff may be used provided they have been suitably validated to demonstrate extinguishment of the carbon heat source and absence of further aerosol generation.

#### 7.6 Sample failure

If a sample fails to ignite properly, the carbon tip detaches, or other product malfunction occurs during the test the result will be deemed invalid.

#### 8. REPEATABILITY AND REPRODUCIBILITY

An international proficiency study involving 17 laboratories which followed this CORESTA Recommended Method up to the point of aerosol collection and determination of ACM was conducted by the CORESTA HTP Task Force in 2021<sup>[2]</sup>. Results were analyzed in basic conformance with ISO 5725-2:1994 and ISO 13528. The mean ACM values and the repeatability (r) and reproducibility (R) values are given in Table 1.

Table 1. Estimates of test sample mean, standard deviations and repeatability and reproducibility for Aerosol Collected Mass (ACM).

Product	N° of Labs	Mean mg/consumable	r	% r	R	% R	rSD (σ <sub>r</sub> )	RSD (σ <sub>R</sub> )
Sample A	13	42.90	8.105	18.9	10.80	25.2	2.894	3.857

#### 9. **BIBLIOGRAPHY**

- [1] CORESTA Heated Tobacco Products Task Force Technical Report: Heated Tobacco Products (HTPs): Standardized Terminology and Recommendations for the Generation and Collection of Emissions. [HTP-259-CTR]
- [2] CORESTA Heated Tobacco Products Task Force Technical Report Proficiency Study for Propylene Glycol, Glycerin, Nicotine, CO, NO, NOx, ACM, and DML in HTP Aerosol. [HTP-280-CTR]