



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

**Tobacco and Tobacco Products Analytes
Sub-Group**

**CORESTA Recommended Method
No. 69**

**DETERMINATION OF pH OF
TOBACCO AND TOBACCO
PRODUCTS**

July 2017



CORESTA RECOMMENDED METHOD N° 69

Title:

DETERMINATION OF pH OF TOBACCO AND TOBACCO PRODUCTS

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Document history:

Date of review	Information
June 2010	Version 1
July 2017	Version 2: extension of scope to include 2016 CORESTA Reference Products (CRPs), ground tobacco, cigarette filler and cigar filler

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(July 2017)

0. INTRODUCTION

In 2009, the CORESTA Smokeless Tobacco Sub-Group (STS), now named Tobacco and Tobacco Products Analytes Sub-Group (TTPA), conducted a collaborative study to evaluate several different methodologies for the determination of pH in smokeless tobacco products in order to adopt a procedure as the CORESTA Recommended Method (CRM). This study included nine commercial smokeless tobacco products. Twelve laboratories provided data for the pH determination using the U.S. Centers for Disease Control and Prevention (CDC) method. The results of this collaborative study were the basis for this CRM.

In 2017, this Recommended Method was updated with supporting data from two additional collaborative studies to include the 2016 CORESTA Reference Products (CRPs), ground tobacco, cigarette filler, and cigar filler in order to expand the scope of this CRM. This Recommended Method has been shown to be fit for purpose for the analysis of the aforementioned matrices.

1. FIELD OF APPLICATION

This Recommended Method is applicable for the determination of pH in tobacco and tobacco products that are in the range 4 to 10. Supporting repeatability and reproducibility data are included for ground tobacco, cigarette filler, cigar filler and a wide range of smokeless tobacco products.

2. NORMATIVE REFERENCES

- 2.1. CORESTA Guide N° 11 - Technical Guideline for Sample Handling of Smokeless Tobacco and Smokeless Tobacco Products.
- 2.2. ISO 3696:1987 Water for analytical laboratory use - specification and test methods.

3. PRINCIPLE

An aqueous extract of the tobacco and tobacco product sample is prepared and its pH determined using a pH electrode.

4. APPARATUS

General laboratory apparatus and supplies, and in particular, the following items:

- 4.1 pH meter, see manufacturer's instructions for operation.
- 4.2 Orbital shaker or magnetic stirrer.
- 4.3 Pipette, 20,0 ml or a calibrated automatic dispenser capable of dispensing 20,0 ml \pm 0,5 ml.
- 4.4 Extraction container, 50 ml polypropylene container, or equivalent.

5. REAGENTS

All reagents must be of recognized analytical grade and comply with existing national regulations.

- 5.1 Water, complying with grade 2 of ISO 3696:1987, or better.
- 5.2 Standard pH buffers (4,00, 7,00 and 10,00)

6. PROCEDURE

6.1 Calibration of pH Meter

- 6.1.1 The pH electrode is calibrated using at least two pH buffers (4,00 and 7,00 or 7,00 and 10,00) to produce a two-point calibration that will cover the pH range of the products tested. Calibration is performed in conjunction with the measurements of the samples and must be completed at the same temperature as the sample measurements.
- 6.1.2 The calibration slope must be within 95 % – 105 % before the electrode can be used for sample measurements.
- 6.1.3 The electrode must be rinsed, before and after each measurement, with water.

6.2 Sample Preparation

A homogeneous test portion shall be prepared for each test sample.

- 6.2.1 Allow the samples to reach room temperature before preparation.
- 6.2.2 Tobacco and tobacco products shall be ground unless the samples are homogeneous and have a particle size <4 mm. It is important that the grinding procedure does not generate excessive heat or cause sample degradation. For further information, see CORESTA Guide no. 11. It is recommended to obtain a test sample of at least 100 g.
- 6.2.3 Unless otherwise specified, smokeless tobacco samples supplied in the form of pouches must be analyzed with their pouch and should be cut into two halves and added directly into the extraction container.
- 6.2.4 Cigar filler will typically need to be ground prior to analysis due to the larger particle size. Testing may also involve the analysis of the entire cigar where the wrapper and filler are ground together.

Note: Insufficient equilibration time for samples removed from the freezer has been identified as a source of variability. Samples removed from the freezer should be placed unopened in the refrigerator for approximately 24 hours to ensure water has sufficient time to fully equilibrate throughout the sample. At the time of analysis, samples should be allowed to equilibrate to room temperature before being opened for weighing.

6.3 Measurement of pH

- 6.3.1 All pH determinations must be performed with reagents and samples that are equilibrated to the testing environment. The testing environment shall be between 20 °C and 25 °C and not vary by more than ± 1 °C during pH determination. Record the temperature during testing.
- 6.3.2 Weigh, $2,0 \pm 0,1$ g of sample into a 50 ml container.
- 6.3.3 Add $20,0 \pm 0,5$ ml water and shake/stir sample gently for a minimum of five minutes up to thirty minutes.
- 6.3.4 After the samples are shaken/stirred, proceed to measuring pH. Record the pH of each sample to a precision of at least two decimal places.
- 6.3.5 The electrode must be rinsed with water, before and after each measurement and stored per the manufacturer's instructions.

7. REPEATABILITY AND REPRODUCIBILITY

In 2009, an international collaborative study¹ involving 12 laboratories who used the U.S. Centers for Disease Control and Prevention (CDC) pH method was conducted by the CORESTA Smokeless Tobacco Sub-Group. This study included nine commercial smokeless tobacco products. Results were analyzed according to ISO 5725-2 (1994). The mean values, repeatability (r), and reproducibility (R) are presented in Table 1. The value of 'N' below is the number of the laboratories used to determine the statistics after the removal of outliers.

In 2016, the Smokeless Tobacco Sub-Group conducted a collaborative study² involving 18 laboratories that specified the use of this CRM. This study included the analysis of the four CORESTA Reference Products that were manufactured in 2016. In 2017, the Tobacco and Tobacco Products Analytes Sub-Group conducted a collaborative study³ involving 21 laboratories that specified the use of this CRM. This study included the analysis of ground tobacco, ground cigarette filler, ground cigar filler, ground cigars (wrapper and filler). Results were analyzed in basic conformance with ISO 5725-2:1994 and ISO/TR 22971:2005. The mean values r and R are presented in Table 1. The value of 'N' is the number of the laboratories used to determine the statistics after the removal of outliers.

¹ CORESTA Smokeless Tobacco Sub-Group Technical Report - Working Group 2, 2009 Collaborative Study Report, July 2010 (Updated 2016).

² CORESTA Smokeless Tobacco Sub-Group Technical Report: CORESTA Reference Products 2016 Analysis Collaborative Study, January 2017.

³ CORESTA Tobacco and Tobacco Products Sub-Group Technical Report – 2017 TSNA, pH, and Moisture (Oven Volatiles) Collaborative Study, July 2017.

Table 1 - Results from the 2009¹, 2016² and 2017³ Collaborative Studies

Sample Type	N	Mean pH	Repeatability (r)	Reproducibility (R)
Nasal Snuff ¹	11	8,07	0,050	0,367
Snus - loose ¹	11	8,42	0,105	0,404
Chewing Tobacco - Twist ¹	11	5,27	0,039	0,214
Chewing Tobacco - Flake ¹	11	8,65	0,059	0,364
Pellet ¹	12	7,61	0,080	0,302
Chewing Tobacco - Loose Leaf ¹	11	6,00	0,033	0,247
Moist Snuff ¹	12	7,77	0,095	0,314
Moist Snuff ¹	10	8,30	0,046	0,417
Pouched Snus ¹	10	7,41	0,057	0,344
CRP1.1 - Pouched Snus ²	18	8,30	0,080	0,497
CRP2.1 - Loose Moist Snuff ²	18	7,74	0,047	0,282
CRP3.1 - Loose Dry Snuff Powder ²	18	6,96	0,084	0,399
CRP4.1 - Chewing Tobacco, Loose Leaf ²	18	6,08	0,074	0,283
1R6F - Ground Cigarette Filler ³	21	5,32	0,05	0,24
1R5F - Ground Cigarette Filler ³	19	4,86	0,04	0,18
RTDAC - Dark Air-Cured Ground Tobacco ³	19	5,90	0,07	0,23
RT2 - Flue-Cured Ground Tobacco ³	19	5,01	0,04	0,22
NIST SRM 3222 - Cigarette Cut Filler ³	16	8,71	0,08	0,32
Flavoured Ground Cigar Filler ³	18	5,12	0,03	0,18
Dark Air-Cured Ground Cigar (Wrapper and Filler) ³	18	6,25	0,05	0,27

8. TEST REPORT

The test report shall provide the pH results to a precision of two decimal places. It shall also provide all details necessary for the identification of the sample.

9. BIBLIOGRAPHY

- Federal Register Vol. 74, No. 4, 712-719 (7 January, 2009). Revised Protocol for Analysis of Nicotine, Total Moisture, and pH in Smokeless Tobacco Products.