

CORESTA RECOMMENDED METHOD N° 69

DETERMINATION OF pH OF SMOKELESS TOBACCO PRODUCTS

(June 2010)

0. INTRODUCTION

The CORESTA Sub-Group on Smokeless Tobacco studied various widely-used procedures for the determination of pH in smokeless tobacco products in order to adopt a procedure as the CORESTA Recommended Method. Studies were carried out during 2009 and evaluated sample preparation, extraction, and analysis parameters on nine different types of smokeless products.

1. FIELD OF APPLICATION

This method is applicable to the measurement of the pH in the range 4 – 10 of smokeless tobacco products.

2. REFERENCES

- 2.1 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.*
- 2.2 ISO 3696:1987 Water for analytical laboratory use - specification and test methods.
- 2.3 CORESTA Smokeless Tobacco Sub-Group Report on the 2009 Collaborative Study (in preparation).

3. PRINCIPLE

An aqueous extract of the smokeless 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**, a wrist-action shaker, or a magnetic stirrer.
- 4.3 **Pipette**, 20.0 mL or a calibrated automatic dispenser capable of dispensing 20.0 mL \pm 0.5mL .
- 4.4 **Container**, 50 mL polypropylene container, or equivalent.

5. REAGENTS

All reagents must be of recognized analytical grade and 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 within the calibration range. Calibration is performed in conjunction with the measurements of the samples; the calibration must be completed at the same temperature as the sample measurements.

6.1.2 The electrode 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

6.2.1 Allow the samples to reach room temperature before preparation.

6.2.2 Smokeless tobacco products in the form of plug, twist, loose-leaf or pellets must be ground prior to analysis. In order to obtain a homogeneous sample at least 100 g of sample must be ground.

6.2.3 Smokeless tobacco samples supplied in the form of pouches must be analyzed with their pouch and should be cut into two halves directly into the extraction container.

6.3 Sample Extraction

6.3.1 Weigh, 2.0 ± 0.1 g of sample into a 50 mL container.

6.3.2 Add 20.0 ± 0.5 mL water and shake/stir sample gently for a minimum of five minutes up to thirty minutes.

6.4 pH Measurement of Samples

6.4.1 pH shall be measured within 60 minutes after the shaker/stirrer has been stopped. All pH determinations must be performed with reagents and samples at a room temperature of 20 – 25 °C. Room temperature should not vary more than ± 1 °C during pH determination. Record the temperature.

6.4.2 Measure the pH to a precision of at least two decimal places.

6.4.3 The electrode must be rinsed with water, before and after each measurement.

7. REPEATABILITY AND REPRODUCIBILITY

An international collaborative study involving 12 laboratories who used the CDC method and tested nine smokeless tobacco products was conducted by the CORESTA Smokeless Tobacco Sub-Group in 2009. Results were analyzed according to ISO 5725-2 (1994) to calculate the mean pH values, repeatability (S_r) and reproducibility (S_R) standard deviations, repeatability (r) and reproducibility (R) values as given in Table 1. The value of 'N' below is the number of the laboratories used to determine the statistics after the removal of outliers.

Table 1. Results from the collaborative study.

Sample Type	Mean pH	S_r	S_R	r	R	N
Nasal Snuff	8.07	0.018	0.131	0.050	0.367	11
Loose Snus	8.42	0.038	0.144	0.105	0.404	11
Chewing Tobacco - Twist	5.27	0.014	0.076	0.039	0.214	11
Chewing Tobacco - Flake	8.65	0.021	0.130	0.059	0.364	11
Pellet	7.61	0.029	0.108	0.080	0.302	12
Chewing Tobacco - Loose Leaf	6.00	0.012	0.088	0.033	0.247	11
Moist Snuff	7.77	0.034	0.112	0.095	0.314	12
Moist Snuff	8.30	0.016	0.149	0.046	0.417	10
Pouched Snus	7.41	0.020	0.123	0.057	0.344	10

8. TEST REPORT

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