Cooperation Centre for Scientific Research Relative to Tobacco

Tobacco and Tobacco Products Analytes Sub-Group

CORESTA Recommended Method No. 88

DETERMINATION OF WATER ACTIVITY OF TOBACCO AND TOBACCO PRODUCTS

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CORESTA RECOMMENDED METHOD Nº 88

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DETERMINATION OF WATER ACTIVITY OF TOBACCO AND TOBACCO PRODUCTS

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DETERMINATION OF WATER ACTIVITY OF TOBACCO AND TOBACCO PRODUCTS

(December 2021)

0. INTRODUCTION

In 2017, the CORESTA Tobacco and Tobacco Products Analytes Sub-Group (TTPA) initiated a proficiency study to evaluate several different measurement principles for the determination of water activity (a_w) of a variety of tobacco products including smokeless tobacco, cigarette filler, and cigar filler [1]. The results from this study indicated that water activity measured using meters equipped with a tunable diode laser (TDL) provided more consistent results for all sample types as compared to meters equipped with capacitance or dewpoint sensors.

In 2018, the TTPA completed a collaborative study for water activity using meters equipped with TDL with the objective to develop a CORESTA Recommended Method (CRM) for the determination of water activity (a_w) of tobacco and tobacco products [2]. Eleven laboratories participated in the study. This study included a range of sample types including a variety of smokeless tobaccos, cigarette filler, and cigar filler. The method specified in this study was shown to be appropriate for the determination of water activity in the aforementioned matrices.

In 2021, the TTPA Sub-group completed a collaborative study for the determination of water activity in nicotine pouches [3]. The intent of the study was to expand the scope of this Recommended Method to also include nicotine pouches. Nicotine pouches do not contain tobacco leaf. Four commercial-like nicotine pouches were included in the study and ten laboratories participated. This Recommended Method has been shown to be fit for the analysis of nicotine pouches.

1. FIELD OF APPLICATION

This method is applicable to the measurement of water activity within a measurement range of 0.250-1.000 a_w of smokeless tobacco (e.g. moist snuff, snus, chewing tobacco, and dry snuff), cigarette filler, ground cigars and nicotine pouches.

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, *Water for analytical laboratory use - specification and test methods*

3. PRINCIPLE

A test portion of tobacco or tobacco product sample is sealed in a measurement chamber equipped with a TDL and an infrared thermometer. The TDL measures the loss of signal strength from the laser to determine the water-vapor pressure of the headspace in equilibrium with the test portion. The infrared thermometer measures the sample temperature to determine the saturated water-vapor pressure. Water activity (a_w) of a sample is the ratio of the partial water-vapor pressure in equilibrium with the test portion analyzed by the TDL to the saturated water-vapor saturation pressure in equilibrium with pure water at the same temperature.

4. APPARATUS

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

4.1 Water Activity meter equipped with tunable diode laser (TDL). Refer to instrument manual for operation.

4.2 Measurement cups, suitable for the apparatus including caps if necessary. Use a new cup preferably with each sample. If cups are reused consult with the instrument manufacturer for proper cleaning procedure.

4.3 Wipes, Activated Carbon, Isopropanol are recommended cleaning materials to clean the meter. Refer to instrument manual for detailed instructions.

5. REAGENTS

All reagents must be of recognized analytical grade.

5.1 Water (1,000 a_w), complying with grade 2 of ISO 3696:1987, or better.

5.2 Water Activity Standards, certified water activity standards covering the range of water activities to be measured; 0,250 a_w to 0,984 a_w is recommended.

5.3 Desiccant, Drierite[1], freshly activated.

6. PROCEDURE

6.1 Sample Preparation

Refer to CORESTA Guide No. 11, Technical Guideline for Sample Handling of Smokeless Tobacco and Smokeless Tobacco Products for sample handling guidelines.

6.1.1 Prior to opening the sample container, allow samples to reach room temperature before analysis (i.e. within 4 °C of the instrument temperature).

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

6.1.3 Portioned smokeless tobacco products and nicotine pouches are analyzed using the whole portion.

[1] This desiccant is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement of this product.

6.1.4 Loose tobacco and non-portioned smokeless tobacco samples that do not require a preparation step are mixed in the sample container before aliquots are removed and placed in a sample cup for analysis.

6.1.5 If a preparation step is needed, the laboratory shall make sure that the preparation and ambient conditions do not cause an increase or decrease in humidity.

6.2 Calibration, verification and adjustment

Note: Allow a 15-minute warm-up period before operating the meter or as specified in the instrument manual.

6.2.1 The TDL calibration is verified using at least two water activity standards bracketing the expected water activity of the samples.

6.2.2 Analyze the water activity standards from lowest to highest (aw) to minimize hysteresis. The standard solutions shall cover the bottom of the sample cup.

Note: Calibration verification shall be performed at the same temperature as sample analysis.

6.2.3 Pour the standard solution into the sample cup and analyze immediately. Record the displayed aw value, reading time and reading temperature.

6.2.4 Water activity standards shall read ± 0,005 aw of the nominal value at 25 °C before the meter is used for sample measurements. Refer to the water activity standard certificate of analysis for the uncertainty value.

Note: If the displayed value for a water activity standard is outside the acceptance range, verify the reading temperature is within 25 °C ±1 °C. Clean the chamber following the instrument manual then perform one reading of activated charcoal before reanalyzing water activity standards to verify the linear offset. If a linear offset has occurred refer to the instrument manual on how to correct for linear offset.

6.3 Sample Analysis

Note: Three individual replicate analyses per sample are recommended.

6.3.1 Mix loose tobacco and non-portioned smokeless tobacco in sample container then remove an aliquot from the sample container using a spatula or forceps; placing sufficient sample in the sample cup to cover the bottom of the cup.

6.3.2 Portioned smokeless tobacco and nicotine pouches: Two to three pouches are typically required to cover the bottom of the sample cup.

Note: Make certain the sample does not surpass the fill line and the rim and outside of the cup are clean.

6.3.3 Analyze immediately. Carefully place the prepared sample cup in the chamber, close the lid, and begin measurement.

6.3.4 Record the displayed a_w value, reading time and reading temperature.

6.3.5 Following the measurement of 9 samples (i.e. 3 samples analyzed in triplicate) or less, verify the calibration using two water activity standards that bracket sample water activity levels. All samples must be bracketed by passing verification standards.

7. REPEATABILITY AND REPRODUCIBILITY

An international collaborative study involving 11 laboratories that used the TDL sensor for

the determination of water activity and tested 10 tobacco and tobacco products was conducted by the CORESTA TTPA Sub-Group in 2018 [2]. Results were analyzed in basic conformance with ISO 5725-2:1994 and ISO/TR 22971:2005. The mean a_w values and the repeatability (r) and reproducibility (R) values are given in Table 1.

In 2021, the TTPA completed a collaborative study, involving 10 laboratories, in order to expand the scope of this Recommended Method to include nicotine pouches [3]. The study included the analysis of four commercial-like nicotine pouches. Results were analyzed in basic conformance with ISO 5725-2:2019. The samples included in this study and the mean values, %r, and %R are shown in Table 1.

Table 1 - Results from the 2018[2] and 2021[3] Collaborative Studies

Product	N*	Mean Water Activity (a_w)	Repeatability		Reproducibility	
			r	% r of mean	R	% R of mean
CRP1.1- Swedish-style snus[2]	11	0,868	0,011	1,30	0,024	2,75
CRP2.1- American-style loose moist snuff[2]	11	0,855	0,007	0,84	0,009	1,05
CRP3.1- American-style dry snuff powder[2]	11	0,404	0,020	4,92	0,050	12,35
CRP4.1- American-style chopped loose-leaf chewing tobacco[2]	11	0,694	0,006	0,93	0,022	3,13
RT1- 1R6F cigarette filler, ground[2]	11	0,565	0,007	1,32	0,017	2,93
American blended menthol cigarette[2]	11	0,574	0,008	1,36	0,027	4,70
RT6- Flavoured cigar filler, ground[2]	11	0,610	0,005	0,88	0,023	3,74
RT8- Unflavoured cigar filler, ground[2]	10	0,662	0,012	1,84	0,018	2,73
STP3- Flavoured American-style loose moist snuff - mint[2]	11	0,876	0,004	0,45	0,009	1,03
STP4- Flavoured American-style loose moist snuff – wintergreen[2]	11	0,837	0,007	0,79	0,011	1,27
NP1 - Nicotine pouch (5 % moisture, 3 mg/portion nicotine)[3]	10	0,390	0,041	10,5%	0,20	50,7%
NP2 - Nicotine pouch (40 % moisture, 11 mg/portion nicotine)[3]	9	0,875	0,012	1,4%	0,02	2,3%
NP3 - Nicotine pouch (15 % moisture, 4 mg/portion nicotine)[3]	8	0,848	0,021	2,4%	0,06	7,2%
NP4 - Nicotine pouch (32 % moisture, 4 mg/portion nicotine)[3]	10	0,939	0,012	1,3%	0,03	3,4%

* The value of 'N' below is the number of the laboratories used to determine the statistics after the removal of outliers

8. TEST REPORT

The test report shall provide the water activity results to precision of three decimal places and shall include all conditions not specified in this Recommended Method which may affect the results. It shall also provide all details necessary for the identification of the sample.

9. BIBLIOGRAPHY

[1] CORESTA Tobacco and Tobacco Products Analytes Sub-Group Technical Report – 2018 Proficiency Study for Water Activity of Tobacco and Tobacco Products, August 2018.

[2] Tobacco and Tobacco Products Analytes Sub-Group Technical Report - 2018 Collaborative Study for the Determination of Water Activity of Tobacco and Tobacco Products, January 2019.

[3] CORESTA Tobacco and Tobacco Products Analytes Sub-Group Technical Report: 2021 Nicotine Pouches Collaborative Study, October 2021.