



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

**Technical Report**

**2018 Collaborative Study for the  
Determination of Water Activity of  
Tobacco and Tobacco Products**

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## 1. Summary

At the April 2018 CORESTA Tobacco and Tobacco Products Analytes Sub-Group (TTPA) meeting held in Guildford, United Kingdom the Sub-Group initiated a collaborative study for the determination of water activity ( $a_w$ ) in smokeless tobacco products, cigarette filler, and cigar filler. The intent of this study was to develop a CORESTA Recommended Method (CRM) for the determination of water activity ( $a_w$ ) of tobacco and tobacco products using a tunable diode laser (TDL). Water activity measured using a TDL sensor provided consistent results from one lab to another (n=11 labs) for all products and the method employed in the collaborative study is suitable for a CRM.

## 2. Introduction

In 2017, the TTPA initiated a proficiency study for the determination of water activity in tobacco products (published August 2018).<sup>[1]</sup> Since this was a proficiency study, laboratories used their in-house methods. Participants in this initial proficiency study used water activity meters equipped with one of three types of sensors (TDL, capacitance, or chilled-mirror dewpoint). The results of the proficiency study demonstrated that water activity measured using a TDL provided more consistent results than the capacitance or chilled-mirror dewpoint sensors. The TDL sensor is selective for water in the presence of other volatiles by measuring water vapor spectroscopically. The chilled-mirror dewpoint sensor cannot determine an accurate dewpoint temperature for samples containing chemicals that condense on the mirror and interfere with water condensation measurements. Although the capacitance hygrometer sensor can measure water activity for samples with volatiles better than the chilled-mirror dewpoint sensor, volatiles can be absorbed by the sensor which may alter the calibration or otherwise interfere and degrade the sensor over time. For these reasons, the TTPA recommended that a CRM be developed for the determination of water activity and that this CRM specify the use of a water activity meter equipped with a TDL sensor.

The focus of this report is a collaborative study for the determination of water activity in tobacco products where the protocol specified the use of a water activity meter equipped with a TDL sensor. The study involved 11 laboratories and specified the use of a draft recommended method for the determination of water activity in a variety of tobacco products. The protocol for this study was distributed in June 2018 and the study was conducted in July 2018 through August 2018.

### 2.1 Objective

The participating laboratories were to provide results for the determination of water activity. Each laboratory used the provided draft recommended method. This study was conducted to develop a CORESTA Recommended Method (CRM) for the determination of water activity ( $a_w$ ) of tobacco and tobacco products using a TDL. Data were collected from the participating laboratories and statistically evaluated in general conformance with ISO 5725-2:1994 and ISO/TR 22971:2005. Repeatability (r) and reproducibility (R) values are presented.

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<sup>[1]</sup> CORESTA Tobacco and Tobacco Products Analytes Sub-Group Technical Report – 2018 Proficiency Study for Water Activity of Tobacco and Tobacco Products – August 2018

### 3. Organization

#### 3.1 Participants

A list of the participating laboratories is provided in Table 1. The laboratories are listed in alphabetical order. Laboratory codes were assigned to each laboratory and do not correspond to the order shown in the table below.

**Table 1: List of Participating Laboratories**

Participating Laboratories
Altria Client Services LLC (Analytical Sciences), United States
Altria Client Services LLC (ELPSS), United States
American Snuff Company, United States
Enthalpy Analytical, Richmond, United States
Labstat International ULC, Canada
R.J. Reynolds Tobacco Company, United States
Sweden Match (Owensboro), United States
Swedish Match, (Analytical Science, Stockholm), Sweden
Swedish Match, (Gothenburg), Sweden
Swedish Match, (Kungälv), Sweden
University of Kentucky, United States

#### 3.2 Protocol

The study protocol is provided in Appendix A and the draft CRM is provided in Appendix B. Specific details from the protocol are described below:

##### 3.2.1 Sample Shipment

Laboratories were responsible for procuring RT1, RT6, and RT8 from University of Kentucky, CRPs from the North Carolina State University (NCSU) Tobacco Analytical Services Lab, the mentholated cigarette from Altria Client Services LLC, and two loose moist snuff samples from American Snuff Company. Laboratories were requested to store the samples at approximately  $-20^{\circ}\text{C}$  upon receipt. Laboratories were requested to conduct the study in July through August and report data by August 16, 2018. The samples are identified in Table 2.

**Table 2: Sample Identification**

Sample Identification	Description
CRP1.1	Swedish-style snus
CRP2.1	American-style loose moist snuff
CRP3.1	American-style dry snuff powder
CRP4.1	American-style chopped loose-leaf chewing tobacco
RT1	1R6F cigarette filler, ground
Mentholated cigarette	American blended menthol cigarette
RT6	Flavoured cigar filler, ground
RT8	Unflavoured cigar filler, ground
STP3	Flavoured American-style loose moist snuff - mint
STP4	Flavoured American-style loose moist snuff - wintergreen

### **3.2.2 Within Laboratory Sample Preparation**

The laboratories were directed to remove samples from the  $-20\text{ }^{\circ}\text{C}$  freezer and place the unopened samples in a refrigerator for a minimum of 24 hours to ensure water was fully equilibrated. Samples could then be removed from the refrigerator for a minimum of 1 hour prior to opening for analysis. Once samples were opened, the samples could be stored in a tightly sealed container and stored at approximately  $4\text{ }^{\circ}\text{C}$  for up to one week. Special handling requirements are described below:

- CRP1.1: Water activity shall be measured on the whole portion; including paper and tobacco.

Note 2-3 pouches were required to cover the bottom of the sample cup.

- Mentholated Cigarette: Immediately prior to analysis, open three packs of cigarettes, remove the filler and place the filler in a tightly sealed bottle and mix well. This process should be completed without delay to avoid volatile losses. This composite sample should be discarded after one week.
- All other samples may be analyzed by removing aliquots directly from the container after mixing with a spatula.

### **3.2.3 Sample Analysis and Data Reporting**

The participating laboratories were instructed to conduct triplicate analyses (individual tobacco weighing) using the provided draft recommended method for the determination of water activity. Participating laboratories were requested to submit any significant deviations from the draft recommended method to generate results. Participating laboratories used water activity meters equipped with a TDL. Only one protocol deviation was reported by the laboratories, it is identified below. Since it was a minor deviation, no data were excluded from the study.

For Lab 8 a minor deviation was reported for the mentholated cigarette sample where the participant selected three cigarettes from one pack of cigarettes for the three replicate analyses (one cigarette per replicate). However, the protocol specified that three packs of cigarettes be opened to prepare a composite sample from which the three replicates would be taken. Although the lab did not follow the protocol for the mentholated cigarette sample, their results were consistent with the results obtained from the remaining study participants. Therefore, this was considered a minor deviation and the results were included in the study.

All test results were to be reported to a minimum of three decimal places.

## **4. Data – Raw**

The full data set for the study is provided in Appendix C. Each analysis includes three replicates. The raw data plots are provided in Appendix D.

## **5. Data – Statistical Analysis**

The statistical analysis was conducted in basic conformance with ISO 5725-2:1994 and ISO/TR 22971:2005. A summary of the results from outlier detection and the calculated results for repeatability ( $r$ ) and reproducibility ( $R$ ) are given below in sections 5.1 and 5.2, respectively. Raw data plots that include all replicates, without removal of outliers, are shown in Appendix D.

## 5.1 Exclusion of Outliers

Procedures outlined in ISO 5725-2:1994 and ISO/TR 22971:2005 were generally used for the exclusion of outliers. An adaptation of Levene's Test<sup>[2]</sup> was used for eliminating laboratories with overly large repeatability standard deviations and Grubbs' Test was used to eliminate laboratories with outlying mean values.

ISO 5725(2) also recommends the use of Mandel's h and k plots. Mandel's h statistic is the same as the statistic used in Grubbs' Test. Similarly, Mandel's k statistic, associated with within lab standard deviation, is statistically equivalent to the c-value calculated in Cochran's Test ( $k = \sqrt{n_{labs}c}$ ). However, the critical values associated with Mandel's h and k statistics do not make allowance for multiple testing and can therefore, give a false impression of statistical significance. Thus, Mandel's h and k statistics do not add fundamentally new information and may lead to incorrect conclusions. For those reasons, we do not include Mandel's h and k plots.

There was only a single outlier in data set as shown in Table 3.

**Table 3: Outliers**

Product	Analyte	Levene's Outlier Lab	Grubbs' Outlier Lab
RT8 - Unflavoured cigar filler, ground	a <sub>w</sub>	–	3

The (–) symbol indicates an outlier was not detected.

## 5.2 Calculation of Repeatability (r) and Reproducibility (R)

After removal of outlying data based on numerical data consistency methods (Grubbs' Test and Levene's Test), the final repeatability and reproducibility (r & R) results were calculated. The r & R results are shown in Table 4. The r & R results reflect both laboratory variability and product consistency.

**Table 4: Repeatability (r) and Reproducibility (R) Limits**

Product	N° of Labs*	Mean Water Activity (a <sub>w</sub> )	Repeatability		Reproducibility	
			r	% r of mean	R	% R of mean
CRP1.1 - Swedish-style snus	11	0,868	0,011	1,30 %	0,024	2,75 %
CRP2.1 - American-style loose moist snuff	11	0,855	0,007	0,84 %	0,009	1,05 %
CRP3.1 - American-style dry snuff powder	11	0,404	0,020	4,92 %	0,050	12,35 %
CRP4.1 - American-style chopped loose-leaf chewing tobacco	11	0,694	0,006	0,93 %	0,022	3,13 %
RT1 - 1R6F cigarette filler, ground	11	0,565	0,007	1,32 %	0,017	2,93 %
American blended menthol cigarette	11	0,574	0,008	1,36 %	0,027	4,70 %
RT6 - Flavoured cigar filler, ground	11	0,610	0,005	0,88 %	0,023	3,74 %
RT8 - Unflavoured cigar filler, ground	10	0,662	0,012	1,84 %	0,018	2,73 %
STP3 - Flavoured American-style loose moist snuff – mint	11	0,876	0,004	0,45 %	0,009	1,03 %
STP4 - Flavoured American-style loose moist snuff – wintergreen	11	0,837	0,007	0,79 %	0,011	1,27 %

\* "N" is the number of laboratory data sets reported as values and after removal of outliers.

<sup>[2]</sup> The approach is discussed in detail by Michael Morton in "Within-Laboratory Variance Outlier Detection: An Alternative to Cochran's Test" in *Beitrag zur Tabakforschung International*, Vol 27, No. 7, pp 135-144.

## **6. Data Interpretations**

Water activity measured using a TDL sensor provided consistent results from one lab to another (n=11 labs) for all products. The TDL sensor is selective for water in the presence of other volatiles by measuring water vapor spectroscopically at a specific wavelength tuned to the absorption band of water and the results demonstrate excellent repeatability and reproducibility values.

## **7. Recommendations**

The TTPA recommends that the results from this collaborative study be used to finalize the draft recommended method specified for use in this study (Appendix B) and that the method be published as a CRM.

## **APPENDIX A: Protocol**



### **CORESTA TOBACCO and TOBACCO PRODUCTS ANALYTES SUB-GROUP**

Project Title: Collaborative study for Water Activity of Tobacco and Tobacco Products

Type of Document: Collaborative Study Protocol

Date: May 23, 2018

Written by: Karl Wagner and Anthony Brown - Study Coordinators

Confidentiality Notice: All data submitted by participating laboratories will be coded and kept confidential.



## 1. Introduction

At the CORESTA Tobacco and Tobacco Products Analytes Sub-Group (TTPA) meeting held on April 17<sup>th</sup>, 2018 in Guildford, it was decided that a collaborative study for water activity ( $a_w$ ) would be conducted during June-August of 2018.

## 2. Objective

The objective of this study is to develop a CORESTA Recommended Method (CRM) for the determination of water activity ( $a_w$ ) of tobacco and tobacco products using meters equipped with a tuneable diode laser (TDL). This study will include a variety of styles of smokeless tobacco products, cigarette filler, and cigar filler. The final output will be a technical report that will include repeatability (r) and reproducibility (R) values and a CRM. The results will be presented at the fall TTPA meeting.

## 3. Time schedule

**Table 1: Study timeline**

Date	Activity
June 12, 2018	Distribute the study protocol and data reporting sheet
June 19, 2018	Additional Laboratories state their intention to participate
June 19, 2018	American Snuff Co. and Altria distribute study samples. Laboratories order remaining samples from North Carolina State University and the University of Kentucky
July 1- August 15, 2018	Laboratories to conduct the study
August 16, 2018	Laboratories submit results by this date
October 22, 2018	Discuss results at Fall 2018 TTPA meeting

## 4. Participating Laboratories:

The laboratories listed in Table 2 have kindly agreed to take part in the study. Other laboratories are encouraged to participate and should notify Anthony Brown [REDACTED], Karl Wagner [REDACTED], and John Bunch [REDACTED] of their interest to participate.

**Table 2: Participating Laboratories**

Participating Laboratories
Altria Client Services LLC, United States (2 sites)
American Snuff Company, United States
Enthalpy Analytical, Richmond, United States
Eurofins, Sweden
Labstat International ULC, Canada
R.J. Reynolds Tobacco Company, United States
Swedish Match, United States
Swedish Match, Sweden (3 sites)
University of Kentucky, United States

## 5. Samples

Order the samples from the suppliers listed in Table 3. The CRPs continue to be stored at -20 °C and distributed by the North Carolina State University (NCSU) Tobacco Analytical Services Lab under the direction of Dr. Ramsey Lewis and Karen Andres. American Snuff Co. will provide two flavoured smokeless tobacco products and Altria will provide a mentholated cigarette. Unless analyzed within one week, all test samples should be placed in moisture barrier bags and stored at -20 °C prior to analysis.

**All remaining samples should be retained in sealed containers at -20 °C as they may be required for a reanalysis.**

**Measure samples immediately after opening the storage container. If samples are left out in ambient lab conditions for more than 5 minutes they may adsorb or desorb moisture and give a false value. Samples must be stored in a manner adequate to protect from atmospheric changes at all times, e.g. sealed containers with lids, zip locked Mylar bags, original packaging, etc. It is best practice to aliquot from the storage container and reseal the container immediately to preserve the product in its packaged state.**

Table 3: Samples

Sample Name	Description	Supplier	Quantity to Request
CRP1.1	Swedish-style Snus	NCSU	10 cans
CRP2.1	American-style loose moist snuff	NCSU	10 cans
CRP3.1	American-style dry snuff powder	NCSU	10 cans
CRP4.1	American-style chopped loose-leaf chewing tobacco	NCSU	10 cans
RT1	1R6F ground filler	UKY	2 bottles
Mentholated cigarette	Flavoured American blended cigarette	Altria	1 carton
RT6	Flavoured Cigar Filler, ground	UKY	2 bottles
RT8	Unflavoured Cigar Filler, ground	UKY	2 bottles
STP3	Flavoured American-style loose moist snuff-mint	American Snuff Co.	3 cans
STP4	Flavoured American-style loose moist snuff-wintergreen	American Snuff Co.	3 cans

1. **Note:** The 2016 CRPs must be ordered from the NC State University: <https://strp.wordpress.ncsu.edu/ordering/> Please contact Karen Andres ([karen\\_andres@ncsu.edu](mailto:karen_andres@ncsu.edu)) with questions regarding ordering and shipping.

2. **Note:** The RT1 -1R6F Ground Filler, RT6 -Flavoured Cigar Ground Filler, and RT8 - Unflavoured Cigar Ground Filler must be ordered from the University of Kentucky: <https://ctrp.uky.edu/> Do not use filler that has been removed from cigarettes.

## 6. Analysis

### 6.1 Analytes:

Each participating laboratory should report water activity for each sample.

## 6.2 Methods:

Each participating laboratory should follow the supplied draft CRM for the determination of water activity using a meter equipped with a tunable diode laser (TDL) sensor.

**Since the purpose of this study is to develop a CRM, data generated with other methods or other types of sensors other than TDL will not be included in this study.**

**6.3 Replicates:** Conduct three (3) independent replicate analyses for each sample. The replicates should be determined from independent tobacco aliquots from the same bottle or can.

**6.4 Sample equilibration:** Samples held at -20 °C shall be placed unopened in a refrigerator for a minimum of 24 hours to ensure water has fully equilibrated within the product. Samples shall be removed from the refrigerator a minimum of 2 hours prior to opening for analysis. The samples should not be opened during the time the samples are equilibrating to ambient temperature. Once samples are equilibrated to ambient temperature, the samples may be stored at approximately 4 °C for up to one week if the analyses will not be conducted immediately.

## 6.5 Sample Handling Requirements:

- CRP1.1: Water activity shall be measured on the whole portion; including paper and tobacco.
- Mentholated Cigarette: Immediately prior to analysis, open three packs of cigarettes, remove the filler and place the filler in a tightly seal bottle and mix well. This process should be completed without delay to avoid volatile losses. This composite sample should be discarded after one week.
- All other samples may be used as is without further grinding.

## 6.6 Data Reporting:

The provided data report spreadsheet with the results of analysis and synopsis of the method shall be sent by email to the study coordinators Anthony Brown ( ) and Karl Wagner ( ).

Report the data and provide a one paragraph method synopsis in the supplied data reporting sheet:

- Data shall be reported to three decimal places.

## 7. Statistical Analysis

A statistical analysis in general conformance with ISO 5725-2:1994 and ISO/TR 22971:2005 will be conducted. Repeatability ( $r$ ) and reproducibility ( $R$ ) values will be reported.

## 8. Presentation of the Results

The results were presented for discussion at the fall 2018 TTPA meeting.

# **APPENDIX B: Draft Recommended Method**

## **CORESTA RECOMMENDED METHOD N° XX**

### **DETERMINATION OF WATER ACTIVITY OF TOBACCO AND TOBACCO PRODUCTS**

*(June 2018)*

#### **1. INTRODUCTION**

In 2017, the CORESTA Tobacco and Tobacco Products Analytes Sub-Group (TTPA) conducted a proficiency study to evaluate several different measurement principles for the determination of water activity ( $a_w$ ) of a variety of smokeless tobacco products, cigarette filler, and cigar filler. 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 capacitance or dewpoint sensors. For this reason, the TTPA decided to initiate 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 using a tunable diode laser.

#### **2. 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 and ground cigars.

#### **3. NORMATIVE REFERENCES**

- 3.1** CORESTA Guide N° 11 - Technical Guideline for Sample Handling of Smokeless Tobacco and Smokeless Tobacco Products.
- 3.2** ISO 3696:1987 *Water for analytical laboratory use - specification and test methods*.
- 3.3** CORESTA Tobacco and Tobacco Products Analytes Sub-Group Technical Report, date TBD Proficiency Study for Water Activity of Tobacco and Tobacco Products, date TBD.

#### **4. PRINCIPLE**

A test portion of tobacco or tobacco product sample is sealed in a measurement chamber with a tunable diode laser (TDL) and an infrared thermometer. The tunable diode laser (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.

## 5. APPARATUS

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

- 5.1 Water Activity meter equipped with tunable diode laser (TDL). Refer to instrument manual for operation.
- 5.2 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.
- 5.3 Wipes, Activated Carbon, Isopropanol are recommended cleaning materials to clean the meter. Refer to instrument manual for detailed instructions.

## 6. REAGENTS

All reagents must be of recognized analytical grade.

- 6.1 Water (1.000  $a_w$ ), complying with grade 2 of ISO 3696:1987, or better.
- 6.2 Water Activity Standards, certified water activity standards cover the range of water activities measured; 0.250  $a_w$  to 0.984  $a_w$  is recommended.
- 6.3 Desiccant, Drierite<sup>[3]</sup>, freshly activated.

## 7. PROCEDURE

### 7.1 Sample Preparation

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

- 7.1.1 Prior to opening the sample container, allow samples to reach room temperature before analysis (i.e. within 4 °C of the instrument temperature).
- 7.1.2 A homogeneous test portion shall be prepared for each test sample.
- 7.1.3 Pouched tobacco products are analyzed using the whole portion.
- 7.1.3 Loose tobacco and non-pouched 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.
- 7.1.4 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.

### 7.2 Calibration, verification and adjustment

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<sup>[3]</sup> The following 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.

**Note:** Allow a 15 minute warm-up period before operating the meter. Refer to the instrument manual to determine the appropriate warm period.

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

**7.2.2** 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.

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

**7.2.4** Water activity standards shall read  $\pm 0.005$  aw of the nominal value at 25 oC 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 oC  $\pm 1$  oC. 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.

### **7.3 Sample Analysis**

**Note:** Three individual replicate analyses per sample are recommended.

**7.3.1** Mix loose tobacco and non-pouched 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.

**7.3.2** Pouched Tobacco: 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.

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

**7.3.4** Record the displayed aw value, reading time and reading temperature.

**7.3.5** 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.

## 8. REPEATABILITY AND REPRODUCIBILITY

An international collaborative study involving X laboratories that used the TDL sensor for the determination of water activity and tested Y tobacco and tobacco products was conducted by the CORESTA TTPA Sub-Group in 2018<sup>[4]</sup>. Results were analyzed according to ISO 5725-2 (1994). The mean  $a_w$  values and the repeatability (r) and reproducibility (R) values are given in Table 1.

**Table 1. Results from the 2018 collaborative study.**

Product	N° of Labs*	Mean Water Activity ( $a_w$ )	Repeatability		Reproducibility	
			r	% r of mean	R	% R of mean

\* “N” is the number of laboratory data sets reported as values and after removal of outliers.

## 9. TEST REPORT

The test report shall provide the water activity results to precision of three decimal places. It shall also provide all details necessary for the identification.

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<sup>[4]</sup> Reference to be provided for the 2018 collaborative study report.

## APPENDIX C: Full Data Set

Lab Code	Rep	Product	A <sub>w</sub>
1	1	CRP1.1- Swedish-style snus	0,863
1	2	CRP1.1- Swedish-style snus	0,869
1	3	CRP1.1- Swedish-style snus	0,869
2	1	CRP1.1- Swedish-style snus	0,890
2	2	CRP1.1- Swedish-style snus	0,881
2	3	CRP1.1- Swedish-style snus	0,885
3	1	CRP1.1- Swedish-style snus	0,872
3	2	CRP1.1- Swedish-style snus	0,870
3	3	CRP1.1- Swedish-style snus	0,869
4	1	CRP1.1- Swedish-style snus	0,876
4	2	CRP1.1- Swedish-style snus	0,874
4	3	CRP1.1- Swedish-style snus	0,878
5	1	CRP1.1- Swedish-style snus	0,860
5	2	CRP1.1- Swedish-style snus	0,865
5	3	CRP1.1- Swedish-style snus	0,873
6	1	CRP1.1- Swedish-style snus	0,857
6	2	CRP1.1- Swedish-style snus	0,851
6	3	CRP1.1- Swedish-style snus	0,854
7	1	CRP1.1- Swedish-style snus	0,865
7	2	CRP1.1- Swedish-style snus	0,868
7	3	CRP1.1- Swedish-style snus	0,873
8	1	CRP1.1- Swedish-style snus	0,864
8	2	CRP1.1- Swedish-style snus	0,869
8	3	CRP1.1- Swedish-style snus	0,867
9	1	CRP1.1- Swedish-style snus	0,863
9	2	CRP1.1- Swedish-style snus	0,857
9	3	CRP1.1- Swedish-style snus	0,865
10	1	CRP1.1- Swedish-style snus	0,871
10	2	CRP1.1- Swedish-style snus	0,862
10	3	CRP1.1- Swedish-style snus	0,866
11	1	CRP1.1- Swedish-style snus	0,867
11	2	CRP1.1- Swedish-style snus	0,865
11	3	CRP1.1- Swedish-style snus	0,875
1	1	CRP2.1- American-style loose moist snuff	0,858
1	2	CRP2.1- American-style loose moist snuff	0,855



Lab Code	Rep	Product	A <sub>w</sub>
1	3	CRP2.1- American-style loose moist snuff	0,853
2	1	CRP2.1- American-style loose moist snuff	0,859
2	2	CRP2.1- American-style loose moist snuff	0,860
2	3	CRP2.1- American-style loose moist snuff	0,856
3	1	CRP2.1- American-style loose moist snuff	0,861
3	2	CRP2.1- American-style loose moist snuff	0,858
3	3	CRP2.1- American-style loose moist snuff	0,849
4	1	CRP2.1- American-style loose moist snuff	0,853
4	2	CRP2.1- American-style loose moist snuff	0,855
4	3	CRP2.1- American-style loose moist snuff	0,855
5	1	CRP2.1- American-style loose moist snuff	0,852
5	2	CRP2.1- American-style loose moist snuff	0,853
5	3	CRP2.1- American-style loose moist snuff	0,856
6	1	CRP2.1- American-style loose moist snuff	0,850
6	2	CRP2.1- American-style loose moist snuff	0,852
6	3	CRP2.1- American-style loose moist snuff	0,854
7	1	CRP2.1- American-style loose moist snuff	0,856
7	2	CRP2.1- American-style loose moist snuff	0,853
7	3	CRP2.1- American-style loose moist snuff	0,851
8	1	CRP2.1- American-style loose moist snuff	0,855
8	2	CRP2.1- American-style loose moist snuff	0,856
8	3	CRP2.1- American-style loose moist snuff	0,853
9	1	CRP2.1- American-style loose moist snuff	0,860
9	2	CRP2.1- American-style loose moist snuff	0,860
9	3	CRP2.1- American-style loose moist snuff	0,861
10	1	CRP2.1- American-style loose moist snuff	0,854
10	2	CRP2.1- American-style loose moist snuff	0,853
10	3	CRP2.1- American-style loose moist snuff	0,854
11	1	CRP2.1- American-style loose moist snuff	0,859
11	2	CRP2.1- American-style loose moist snuff	0,857
11	3	CRP2.1- American-style loose moist snuff	0,855
1	1	CRP3.1- American-style dry snuff powder	0,396
1	2	CRP3.1- American-style dry snuff powder	0,395
1	3	CRP3.1- American-style dry snuff powder	0,391
2	1	CRP3.1- American-style dry snuff powder	0,427
2	2	CRP3.1- American-style dry snuff powder	0,425
2	3	CRP3.1- American-style dry snuff powder	0,426

Lab Code	Rep	Product	A <sub>w</sub>
3	1	CRP3.1- American-style dry snuff powder	0,403
3	2	CRP3.1- American-style dry snuff powder	0,407
3	3	CRP3.1- American-style dry snuff powder	0,398
4	1	CRP3.1- American-style dry snuff powder	0,372
4	2	CRP3.1- American-style dry snuff powder	0,381
4	3	CRP3.1- American-style dry snuff powder	0,381
5	1	CRP3.1- American-style dry snuff powder	0,421
5	2	CRP3.1- American-style dry snuff powder	0,382
5	3	CRP3.1- American-style dry snuff powder	0,418
6	1	CRP3.1- American-style dry snuff powder	0,394
6	2	CRP3.1- American-style dry snuff powder	0,392
6	3	CRP3.1- American-style dry snuff powder	0,389
7	1	CRP3.1- American-style dry snuff powder	0,381
7	2	CRP3.1- American-style dry snuff powder	0,384
7	3	CRP3.1- American-style dry snuff powder	0,385
8	1	CRP3.1- American-style dry snuff powder	0,414
8	2	CRP3.1- American-style dry snuff powder	0,420
8	3	CRP3.1- American-style dry snuff powder	0,421
9	1	CRP3.1- American-style dry snuff powder	0,423
9	2	CRP3.1- American-style dry snuff powder	0,425
9	3	CRP3.1- American-style dry snuff powder	0,425
10	1	CRP3.1- American-style dry snuff powder	0,422
10	2	CRP3.1- American-style dry snuff powder	0,423
10	3	CRP3.1- American-style dry snuff powder	0,423
11	1	CRP3.1- American-style dry snuff powder	0,399
11	2	CRP3.1- American-style dry snuff powder	0,400
11	3	CRP3.1- American-style dry snuff powder	0,398
1	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,687
1	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,689
1	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,686
2	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,692
2	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,694
2	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,694
3	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,692
3	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,693
3	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,691
4	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,700

Lab Code	Rep	Product	A <sub>w</sub>
4	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,700
4	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,699
5	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,706
5	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,695
5	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,696
6	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,695
6	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,691
6	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,690
7	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,701
7	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,704
7	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,703
8	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,685
8	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,686
8	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,687
9	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,710
9	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,707
9	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,706
10	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,684
10	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,686
10	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,685
11	1	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,688
11	2	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,687
11	3	CRP4.1- American-style chopped loose-leaf chewing tobacco	0,686
1	1	RT1- 1R6F cigarette filler, ground	0,566
1	2	RT1- 1R6F cigarette filler, ground	0,564
1	3	RT1- 1R6F cigarette filler, ground	0,561
2	1	RT1- 1R6F cigarette filler, ground	0,559
2	2	RT1- 1R6F cigarette filler, ground	0,557
2	3	RT1- 1R6F cigarette filler, ground	0,564
3	1	RT1- 1R6F cigarette filler, ground	0,575
3	2	RT1- 1R6F cigarette filler, ground	0,578
3	3	RT1- 1R6F cigarette filler, ground	0,577
4	1	RT1- 1R6F cigarette filler, ground	0,559
4	2	RT1- 1R6F cigarette filler, ground	0,555
4	3	RT1- 1R6F cigarette filler, ground	0,557
5	1	RT1- 1R6F cigarette filler, ground	0,563
5	2	RT1- 1R6F cigarette filler, ground	0,562

Lab Code	Rep	Product	A <sub>w</sub>
5	3	RT1- 1R6F cigarette filler, ground	0,561
6	1	RT1- 1R6F cigarette filler, ground	0,559
6	2	RT1- 1R6F cigarette filler, ground	0,566
6	3	RT1- 1R6F cigarette filler, ground	0,564
7	1	RT1- 1R6F cigarette filler, ground	0,565
7	2	RT1- 1R6F cigarette filler, ground	0,563
7	3	RT1- 1R6F cigarette filler, ground	0,566
8	1	RT1- 1R6F cigarette filler, ground	0,573
8	2	RT1- 1R6F cigarette filler, ground	0,571
8	3	RT1- 1R6F cigarette filler, ground	0,572
9	1	RT1- 1R6F cigarette filler, ground	0,569
9	2	RT1- 1R6F cigarette filler, ground	0,564
9	3	RT1- 1R6F cigarette filler, ground	0,568
10	1	RT1- 1R6F cigarette filler, ground	0,573
10	2	RT1- 1R6F cigarette filler, ground	0,565
10	3	RT1- 1R6F cigarette filler, ground	0,566
11	1	RT1- 1R6F cigarette filler, ground	0,561
11	2	RT1- 1R6F cigarette filler, ground	0,563
11	3	RT1- 1R6F cigarette filler, ground	0,567
1	1	American blended menthol cigarette	0,574
1	2	American blended menthol cigarette	0,572
1	3	American blended menthol cigarette	0,579
2	1	American blended menthol cigarette	0,576
2	2	American blended menthol cigarette	0,577
2	3	American blended menthol cigarette	0,577
3	1	American blended menthol cigarette	0,589
3	2	American blended menthol cigarette	0,589
3	3	American blended menthol cigarette	0,587
4	1	American blended menthol cigarette	0,566
4	2	American blended menthol cigarette	0,563
4	3	American blended menthol cigarette	0,563
5	1	American blended menthol cigarette	0,581
5	2	American blended menthol cigarette	0,573
5	3	American blended menthol cigarette	0,579
6	1	American blended menthol cigarette	0,565
6	2	American blended menthol cigarette	0,562
6	3	American blended menthol cigarette	0,567

Lab Code	Rep	Product	A <sub>w</sub>
7	1	American blended menthol cigarette	0,567
7	2	American blended menthol cigarette	0,561
7	3	American blended menthol cigarette	0,562
8	1	American blended menthol cigarette	0,577
8	2	American blended menthol cigarette	0,584
8	3	American blended menthol cigarette	0,584
9	1	American blended menthol cigarette	0,587
9	2	American blended menthol cigarette	0,586
9	3	American blended menthol cigarette	0,585
10	1	American blended menthol cigarette	0,574
10	2	American blended menthol cigarette	0,571
10	3	American blended menthol cigarette	0,571
11	1	American blended menthol cigarette	0,558
11	2	American blended menthol cigarette	0,565
11	3	American blended menthol cigarette	0,563
1	1	RT6- Flavoured cigar filler, ground	0,616
1	2	RT6- Flavoured cigar filler, ground	0,617
1	3	RT6- Flavoured cigar filler, ground	0,616
2	1	RT6- Flavoured cigar filler, ground	0,618
2	2	RT6- Flavoured cigar filler, ground	0,613
2	3	RT6- Flavoured cigar filler, ground	0,615
3	1	RT6- Flavoured cigar filler, ground	0,590
3	2	RT6- Flavoured cigar filler, ground	0,591
3	3	RT6- Flavoured cigar filler, ground	0,592
4	1	RT6- Flavoured cigar filler, ground	0,603
4	2	RT6- Flavoured cigar filler, ground	0,603
4	3	RT6- Flavoured cigar filler, ground	0,604
5	1	RT6- Flavoured cigar filler, ground	0,609
5	2	RT6- Flavoured cigar filler, ground	0,609
5	3	RT6- Flavoured cigar filler, ground	0,613
6	1	RT6- Flavoured cigar filler, ground	0,603
6	2	RT6- Flavoured cigar filler, ground	0,605
6	3	RT6- Flavoured cigar filler, ground	0,604
7	1	RT6- Flavoured cigar filler, ground	0,617
7	2	RT6- Flavoured cigar filler, ground	0,617
7	3	RT6- Flavoured cigar filler, ground	0,615
8	1	RT6- Flavoured cigar filler, ground	0,609

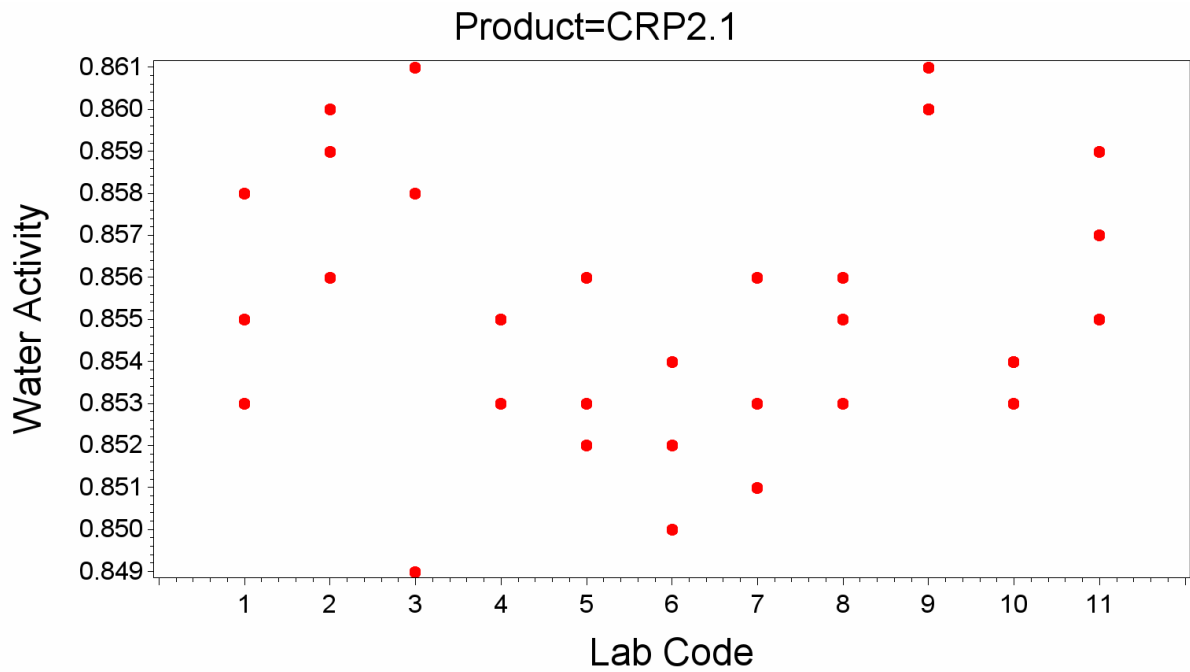
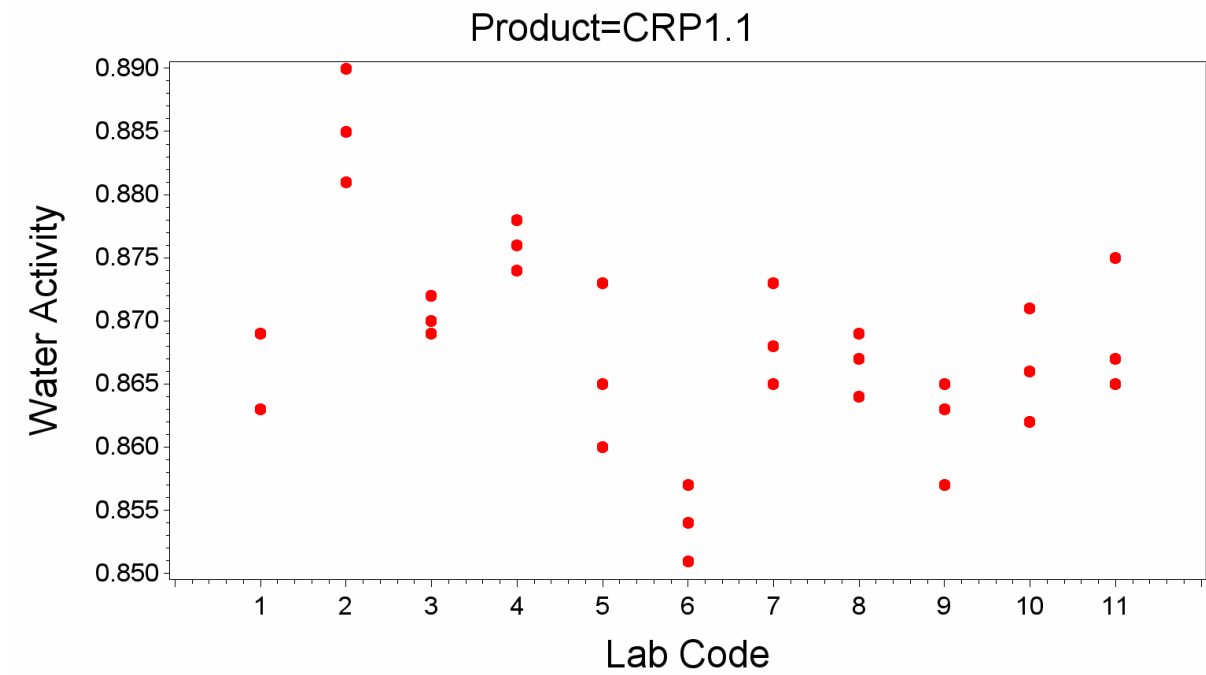
Lab Code	Rep	Product	A <sub>w</sub>
8	2	RT6- Flavoured cigar filler, ground	0,613
8	3	RT6- Flavoured cigar filler, ground	0,611
9	1	RT6- Flavoured cigar filler, ground	0,614
9	2	RT6- Flavoured cigar filler, ground	0,615
9	3	RT6- Flavoured cigar filler, ground	0,617
10	1	RT6- Flavoured cigar filler, ground	0,609
10	2	RT6- Flavoured cigar filler, ground	0,611
10	3	RT6- Flavoured cigar filler, ground	0,612
11	1	RT6- Flavoured cigar filler, ground	0,613
11	2	RT6- Flavoured cigar filler, ground	0,620
11	3	RT6- Flavoured cigar filler, ground	0,620
1	1	RT8- Unflavoured cigar filler, ground	0,668
1	2	RT8- Unflavoured cigar filler, ground	0,670
1	3	RT8- Unflavoured cigar filler, ground	0,665
2	1	RT8- Unflavoured cigar filler, ground	0,669
2	2	RT8- Unflavoured cigar filler, ground	0,667
2	3	RT8- Unflavoured cigar filler, ground	0,667
3	1	RT8- Unflavoured cigar filler, ground	0,609
3	2	RT8- Unflavoured cigar filler, ground	0,606
3	3	RT8- Unflavoured cigar filler, ground	0,607
4	1	RT8- Unflavoured cigar filler, ground	0,655
4	2	RT8- Unflavoured cigar filler, ground	0,650
4	3	RT8- Unflavoured cigar filler, ground	0,651
5	1	RT8- Unflavoured cigar filler, ground	0,673
5	2	RT8- Unflavoured cigar filler, ground	0,665
5	3	RT8- Unflavoured cigar filler, ground	0,660
6	1	RT8- Unflavoured cigar filler, ground	0,656
6	2	RT8- Unflavoured cigar filler, ground	0,657
6	3	RT8- Unflavoured cigar filler, ground	0,656
7	1	RT8- Unflavoured cigar filler, ground	0,660
7	2	RT8- Unflavoured cigar filler, ground	0,667
7	3	RT8- Unflavoured cigar filler, ground	0,667
8	1	RT8- Unflavoured cigar filler, ground	0,663
8	2	RT8- Unflavoured cigar filler, ground	0,658
8	3	RT8- Unflavoured cigar filler, ground	0,661
9	1	RT8- Unflavoured cigar filler, ground	0,666
9	2	RT8- Unflavoured cigar filler, ground	0,665

Lab Code	Rep	Product	A <sub>w</sub>
9	3	RT8- Unflavoured cigar filler, ground	0,659
10	1	RT8- Unflavoured cigar filler, ground	0,662
10	2	RT8- Unflavoured cigar filler, ground	0,665
10	3	RT8- Unflavoured cigar filler, ground	0,658
11	1	RT8- Unflavoured cigar filler, ground	0,646
11	2	RT8- Unflavoured cigar filler, ground	0,657
11	3	RT8- Unflavoured cigar filler, ground	0,664
1	1	STP3 - Flavoured American-style loose moist snuff – mint	0,876
1	2	STP3 - Flavoured American-style loose moist snuff – mint	0,875
1	3	STP3 - Flavoured American-style loose moist snuff – mint	0,876
2	1	STP3 - Flavoured American-style loose moist snuff – mint	0,881
2	2	STP3 - Flavoured American-style loose moist snuff – mint	0,881
2	3	STP3 - Flavoured American-style loose moist snuff – mint	0,883
3	1	STP3 - Flavoured American-style loose moist snuff – mint	0,875
3	2	STP3 - Flavoured American-style loose moist snuff – mint	0,878
3	3	STP3 - Flavoured American-style loose moist snuff – mint	0,874
4	1	STP3 - Flavoured American-style loose moist snuff – mint	0,879
4	2	STP3 - Flavoured American-style loose moist snuff – mint	0,876
4	3	STP3 - Flavoured American-style loose moist snuff – mint	0,877
5	1	STP3 - Flavoured American-style loose moist snuff – mint	0,873
5	2	STP3 - Flavoured American-style loose moist snuff – mint	0,874
5	3	STP3 - Flavoured American-style loose moist snuff – mint	0,876
6	1	STP3 - Flavoured American-style loose moist snuff – mint	0,871
6	2	STP3 - Flavoured American-style loose moist snuff – mint	0,873
6	3	STP3 - Flavoured American-style loose moist snuff – mint	0,873
7	1	STP3 - Flavoured American-style loose moist snuff – mint	0,879
7	2	STP3 - Flavoured American-style loose moist snuff – mint	0,879
7	3	STP3 - Flavoured American-style loose moist snuff – mint	0,879
8	1	STP3 - Flavoured American-style loose moist snuff – mint	0,874
8	2	STP3 - Flavoured American-style loose moist snuff – mint	0,875
8	3	STP3 - Flavoured American-style loose moist snuff – mint	0,872
9	1	STP3 - Flavoured American-style loose moist snuff – mint	0,882
9	2	STP3 - Flavoured American-style loose moist snuff – mint	0,881
9	3	STP3 - Flavoured American-style loose moist snuff – mint	0,880
10	1	STP3 - Flavoured American-style loose moist snuff – mint	0,874
10	2	STP3 - Flavoured American-style loose moist snuff – mint	0,875
10	3	STP3 - Flavoured American-style loose moist snuff – mint	0,875

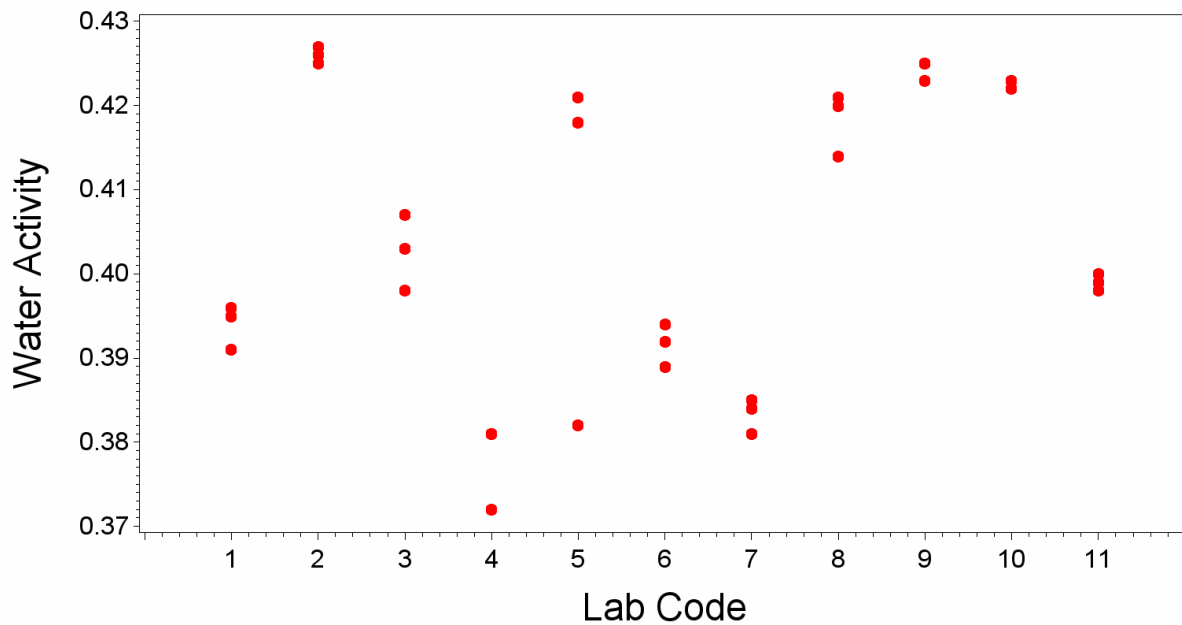
Lab Code	Rep	Product	A <sub>w</sub>
11	1	STP3 - Flavoured American-style loose moist snuff – mint	0,875
11	2	STP3 - Flavoured American-style loose moist snuff – mint	0,878
11	3	STP3 - Flavoured American-style loose moist snuff – mint	0,873
1	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,837
1	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,837
1	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,833
2	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,845
2	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,845
2	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,845
3	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,837
3	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,835
3	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,832
4	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,836
4	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,839
4	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,838
5	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,835
5	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,840
5	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,839
6	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,836
6	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,837
6	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,830
7	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,845
7	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,836
7	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,840
8	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,835
8	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,836
8	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,832
9	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,837
9	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,838
9	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,837
10	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,837
10	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,836
10	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,836
11	1	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,834
11	2	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,834
11	3	STP4 - Flavoured American-style loose moist snuff – wintergreen	0,834



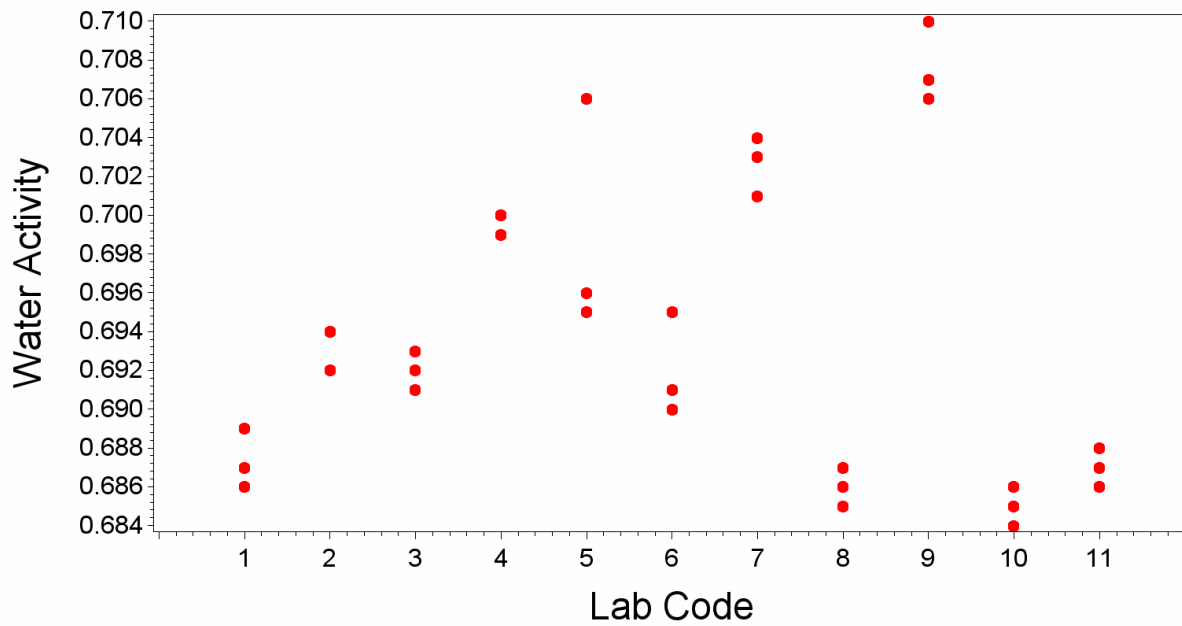
## APPENDIX D: Raw Data Plots



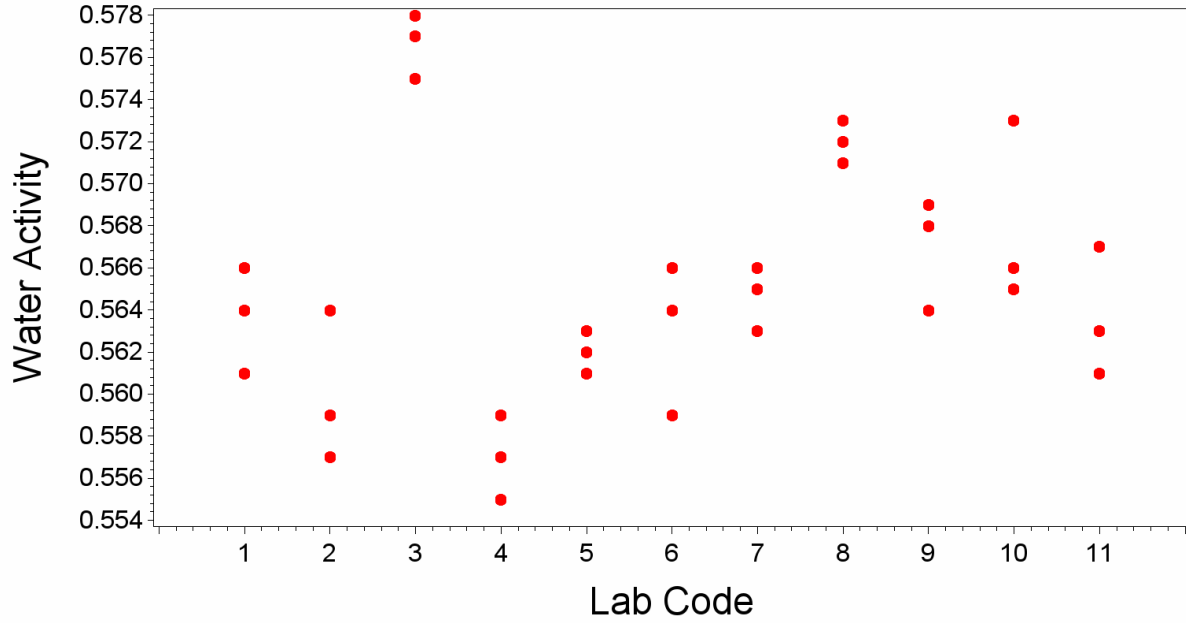
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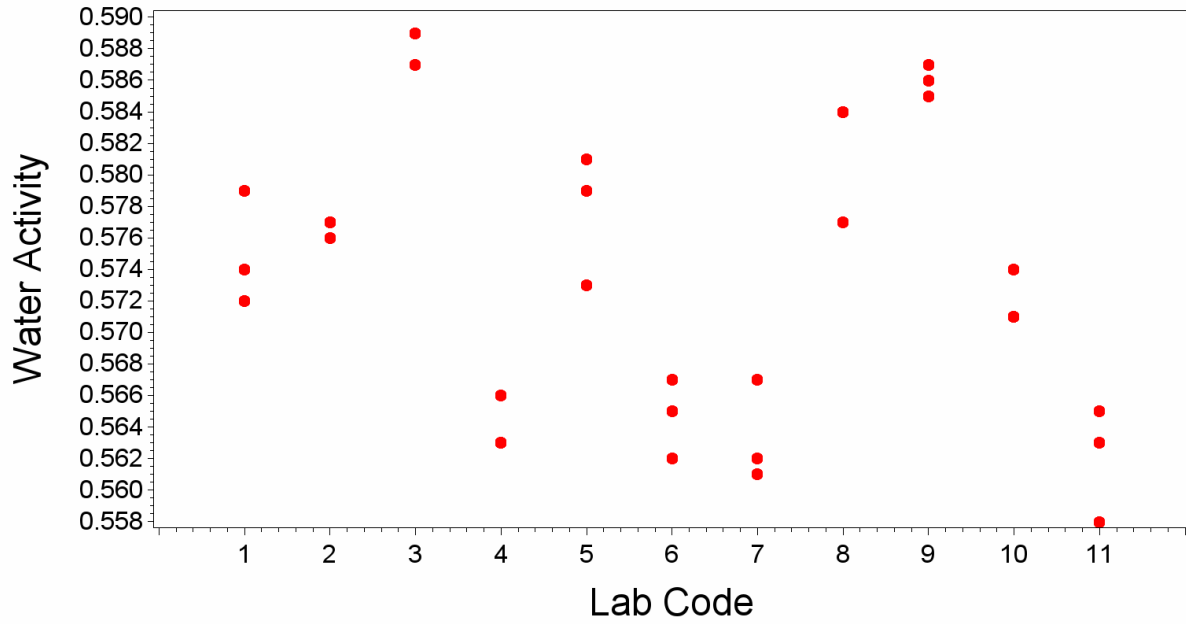
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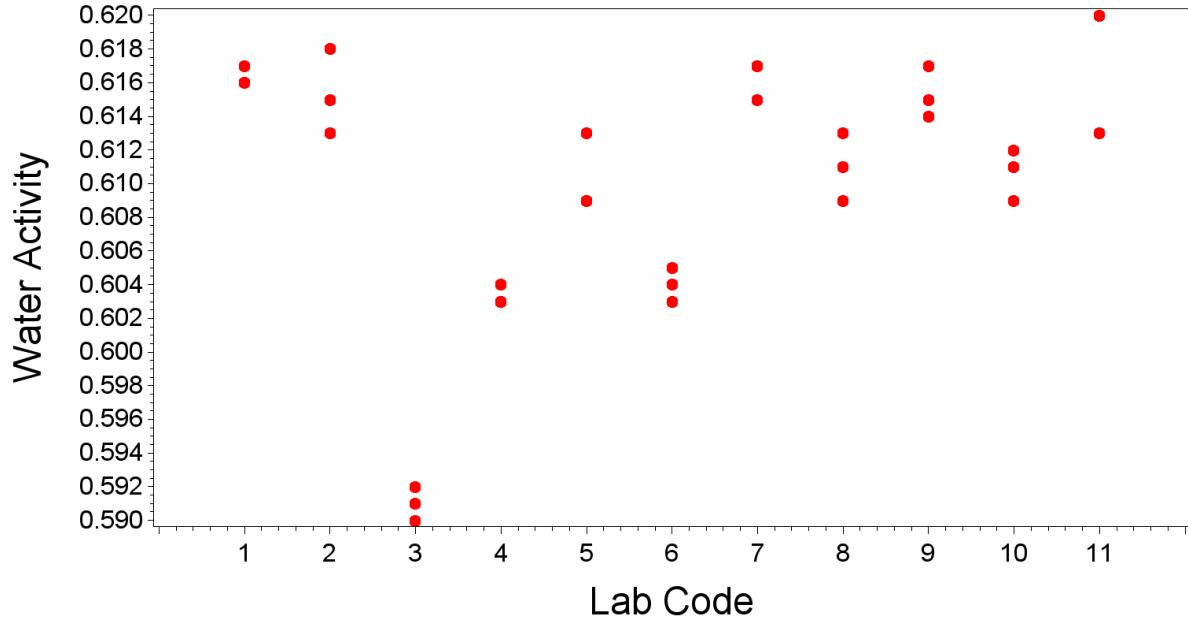
Product=RT1



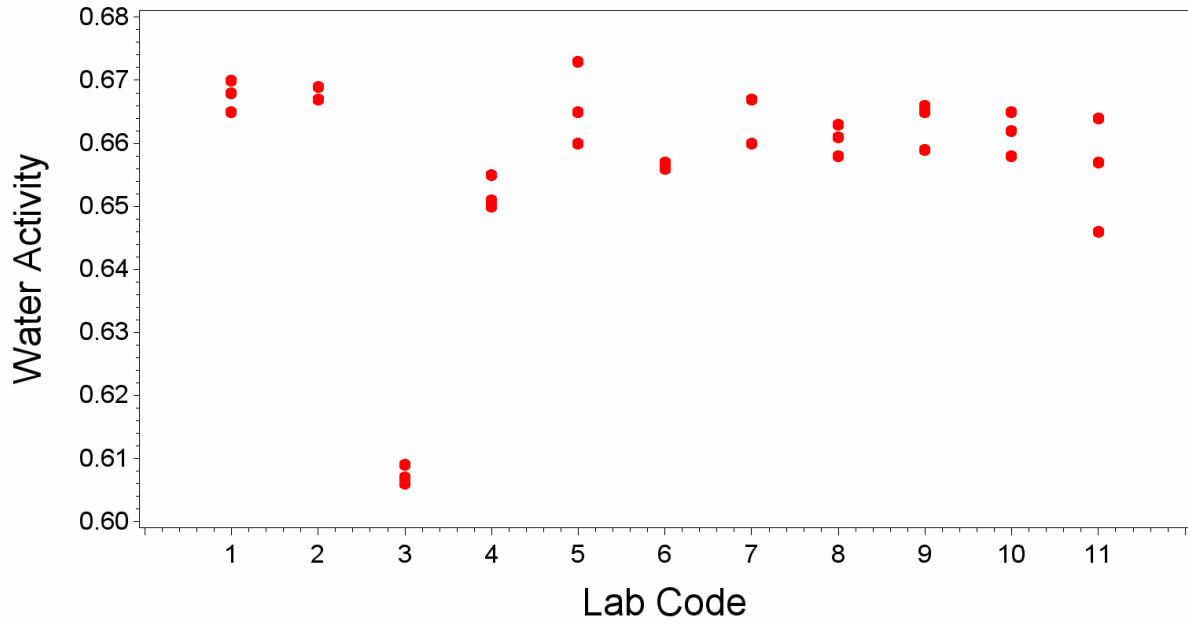
Product=Mentholated cigarette



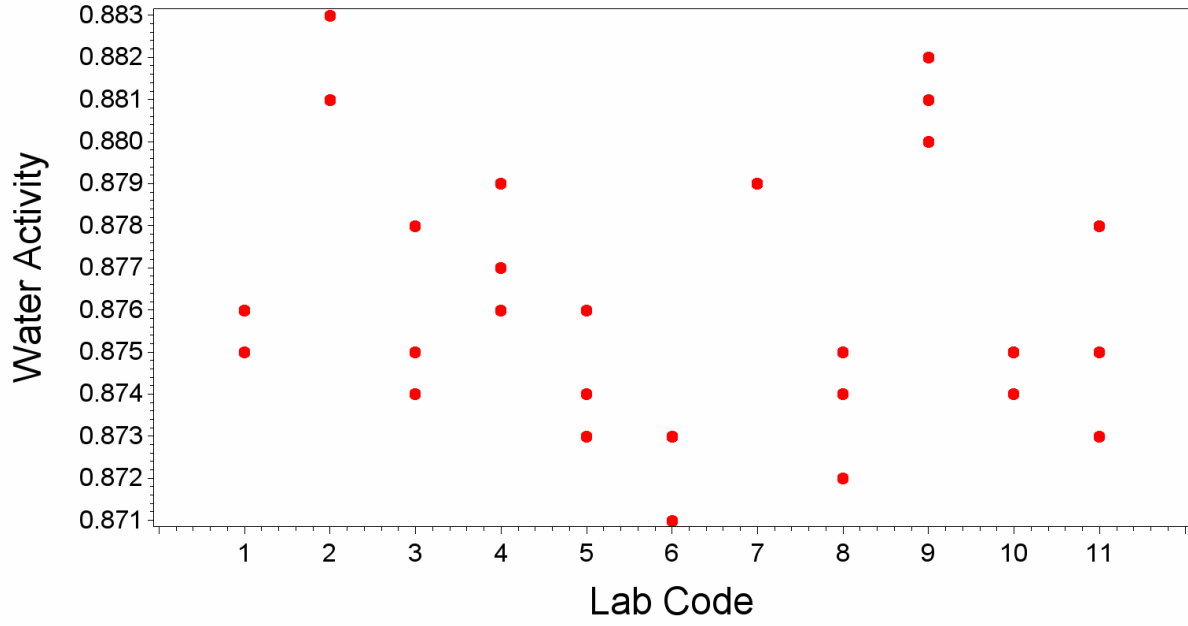
Product=RT6



Product=RT8



Product=STP3



Product=STP4

