Tobacco and Tobacco Products Analytes Sub-Group

Technical Report

2018 Proficiency Study for Water Activity of Tobacco and Tobacco Products

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1. Summary

At the October 2017 CORESTA Tobacco and Tobacco Products Analytes Sub-Group (TTPA) meeting held in Kitzbühel, Austria, the Sub-Group initiated a proficiency study for the determination of water activity (a_w) in smokeless tobacco products, ground tobacco, cigarette filler, and cigar filler. The intent of this study was to assess the capability of the participating laboratories to measure water activity and to support laboratory accreditation. Participating laboratories used water activity meters equipped with one or more of the following sensor types: tunable diode laser (TDL), chilled mirror, or capacitance hygrometer. Water activity measured using a TDL sensor provided consistent results from one lab to another (n=3) for all products. The chilled mirror or capacitance sensors can be affected by volatile constituents (e.g. flavors) found in certain tobacco products. The TTPA Sub-Group recommends that a future collaborative study be conducted that focuses on the use of water activity meters equipped with TDL sensors and that a CRM be drafted.

2. Introduction

The Tobacco and Tobacco Products Analytes Sub-Group (TTPA) conducted a proficiency study for the determination of water activity. The study involved seven laboratories using their in-house analytical procedure. The protocol for this study was distributed in November 2017 and the study was conducted from December 2017 to February 2018. Tabulated data are presented along with z-scores.

2.1 Objective

The participating laboratories were to provide results for the determination of water activity. Each laboratory used their in-house procedure. This study was conducted to support laboratory accreditation by providing an assessment of laboratory capability. Data were collected from the participating laboratories and statistically evaluated in general conformance with the recommendations of ISO 13528:2015.

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, United States
American Snuff Company, United States
Enthalpy Analytical, United States
KT&G Research Institute, South Korea
R.J. Reynolds Tobacco Company, United States
Swedish Match, North America, United States
Swedish Match, Northern Europe, Sweden
Swisher International, United States

3.2 Protocol

Specific details from the protocol are described below:

3.2.1 Sample Shipment

Laboratories were responsible for procuring 1R6F (RT1), RT6, Cigar Filler #1-11/17 and Cigar Filler #2-11/171 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 °C upon receipt. Laboratories were requested to conduct the study in December through February and report data by February 16, 2018. The samples are identified in Table 2.

Table 2: Sample Identification

Product Type
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
1R6F ground filler (Lot/Batch Number RT1) - Unflavoured cigarette filler, ground
Mentholated cigarette - Flavoured American blended cigarette
RT6 - Flavoured cigar Filler, ground
Cigar Filler #1-11/17 - Flavoured cigar filler, ground
Cigar Filler #2-11/17 - Unflavoured cigar filler, ground
MS-W - American-style loose moist snuff - wintergreen
MS-M - American-style loose moist snuff - mint

3.2.2 Within Laboratory Sample Preparation

The laboratories were directed to remove samples from the −20 °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 one 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 ºC 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 their in-house analytical procedure for the determination of water activity. Participating laboratories were requested to submit the type of sensor used to generate results and provide a synopsis of their method. Participating laboratories used water activity meters equipped with one or more of the following sensor types: tunable diode laser (TDL), chilled mirror, or capacitance hygrometer. Synopses reported by the laboratories are provided below:

- Lab 1: Used a tunable diode laser detector to generate data. The calibration and verification of calibration was performed using standard solutions that bracketed samples. Calibration verification was performed after nine determinations. Samples were aliquoted into sample cups with caps placed over the cups before analysis. Samples were analyzed immediately after removing the cap. Samples covered the bottom of the cup and do not fill more than half the cup. The temperature at the time of the reading is recorded as well as the elapsed time for the measurement.
- Lab 2a: Used a tunable diode laser detector to generate data. The calibration and verification of calibration was performed prior to analysis. A homogenous sample is placed in a sample cup. Samples covered the bottom of the cup and do not fill more than half the cup. For pouched samples 2-3 pouches were used to ensure the bottom of the cup is covered.
- Lab 2b: Used a capacitance hygrometer detector to generate data. The calibration and verification of calibration was performed prior to analysis. A homogenous sample is placed in a sample cup. Samples covered the bottom of the cup and do not fill more than half the cup. For pouched samples 2-3 pouches were used to ensure the bottom of the cup is covered.
- Lab 3: Used a capacitance hygrometer detector to generate data. Approximately 3 g of sample was placed in 4 mm diameter × 10 mm depth clear dish. The dish was placed in Rotronics Hygrolab C1 instrument. The measurement time was approximately 5 minutes per sample replicate.
- Lab 4: Used a Dewpoint "Chilled Mirror" detector to generate data. The instrument is allowed to warm up for 15 minutes before verification standards were analyzed using 0,760 NaCl, 0,500 LiCl, 0,250 LiCl, and 1,00 distilled water. The linear offset was verified. A homogenous sample (three replicates) was placed in sample cups to half full and capped until analysis. Volatile samples (MS-W) ran in dewpoint mode gave an error message. Therefore, the volatile samples were analyzed using the meter set to capacitance mode after the linear offset was verified using 0,760 NaCl, 0,500 LiCl, 0,250 LiCl, 0,920 NaCl verification standards.
- Lab 5: Used a tunable diode laser detector to generate data. Calibration verification was performed using two salt standards at 0,500 a_w, 0,760 a_w and 1,000 a_w (UHP water). A check sample is analyzed with each batch. Samples covered the bottom of the cup and do not surpass the cup fill line and were immediately analyzed. Calibration verification is performed using a 0,760 a_w standard after 20 sample measurements.
- Lab 6: Used a tunable diode laser detector to generate data. Calibration verification was performed using 0,250 a_w, 0,760 a_w and 0,984 a_w standards. Two or three verification standards are used to check linear offset and adjusted if necessary. Samples covered the bottom of the cup and fill to half line of the cup. The Aqualab TDL is temperature controlled to 25 °C. After seven measurements, the instrument chamber is cleaned by measuring activated charcoal in a cup.

- Lab 7: Used a tunable diode laser detector to generate data. The instrument is allowed to warm up for at least 15 minutes before four verification standards were analyzed using 0,250, 0,500, 0,760, and 1,00 a_w to verify the linear offset. If standards are out of range then the unit is cleaned and at least two verification standards are analyzed to ensure standards are within specification prior to testing. A homogenous sample is placed in a sample cup ensuring the bottom of the cup is covered. The temperature at the time of the reading is recorded.

All test results were to be reported to a minimum of three decimal places. The study results and the comments were to be sent by e-mail to the study coordinators.

4. Data – Raw

The full data set for the study are provided in Appendix A. Each analysis includes three replicates. The raw data plots are provided in Appendix B.

5. Data – Statistical Analysis

The analysis was carried out in general conformance with ISO 13528. The assigned values and the standard deviation for proficiency assessment for each sample were calculated using Algorithm A, a robust calculation procedure described in ISO 13528, on the laboratory values.

Table 3. Water Activity proficiency test values for each product tested, calculated using Algorithm A.

Product	Assigned Value	Std Dev for Prof. Assess.
1R6F ground filler/RT1	0,562	0,0097
CRP1.1	0,868	0,0057
CRP2.1	0,857	0,0071
CRP3.1	0,433	0,0741
CRP4.1	0,687	0,0112
Cigar Filler #1-11/17	0,597	0,0123
Cigar Filler #2-11/17	0,576	0,0183
Mentholated Cigarette	0,539	0,0351
MS-M, Moist Snuff Mint	0,872	0,0070
MS-W, Moist Snuff Wintergreen	0,829	0,0027
RT6 Flavoured Cigar Filler	0,599	0,0111

Results – Z-Scores

The corresponding z-scores are given in Table 4. As a general rule, the z-scores are individually regarded as reasonable if $-2 < z < 2$ and generally thought to be unacceptable if $|z| > 3$. Laboratories should consider $|z|$ between 2 and 3 as a warning signal. Laboratories should particularly be aware of the pattern of their results. For example a single z-value signal should be looked into, but a pattern of problematic z-scores in the same direction should be taken more seriously still.

Table 4. Z-scores

Product	Laboratory Code							
	1	2a	2b	3	4	5	6	7
CRP1.1	0,50	-0,34	-0,62	-2,15	3,23	-0,03	0,31	0,19
CRP2.1	-1,01	0,11	2,45	-1,20	-0,03	0,03	0,82	-0,22
CRP3.1	-0,05	-0,76	-0,77	0,23	1,12	-0,59	-0,66	1,48
CRP4.1	-0,97	-0,89	-0,68	1,48	0,77	0,07	0,59	-0,36
1R6F ground filler/RT1	-0,10	0,59	-0,31	0,75	5,69	-0,43	-0,93	-1,07
Cigar Filler #1-11/17	-0,18	-0,15	0,25	0,70	5,21	-0,07	-0,55	-1,75
Cigar Filler #2-11/17	0,14	-0,30	-0,59	0,60	2,78	-0,15	0,31	-1,68
MS-M, Moist Snuff Mint	-0,43	-0,20	3,89	-0,72	1,29	-0,71	-0,48	-0,24
MS-W, Moist Snuff Wintergreen	-0,85	-0,72	9,69	0,49	0,37	-1,15	0,49	-0,13
Mentholated Cigarette	-0,63	-0,75	-0,85	0,95	2,46	0,47	-0,07	-0,61
RT6 Flavoured Cigar Filler	-0,21	-0,02	0,15	0,82	6,21	-0,06	-0,91	-1,27

Graphs of the z-scores are given in the following two graphs showing laboratory results organized by product (Figure 1) and sensor type (Figure 2). As the graphs show, there were three different types of sensor used by participating labs (TDL, chilled mirror, or capacitance). Though admittedly there is a small number of participating laboratories, generally speaking the TDL detector gave results that were more consistent from lab-to-lab and also within a particular laboratory.

Figure 1. Z-Scores for Water Activity

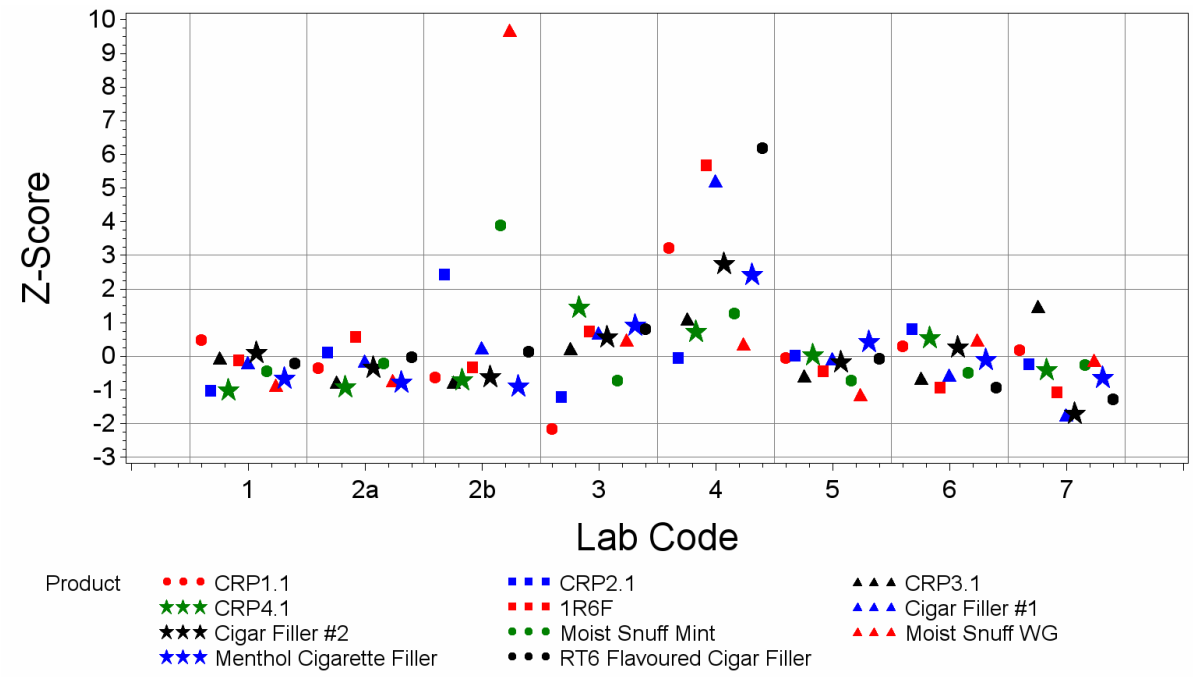
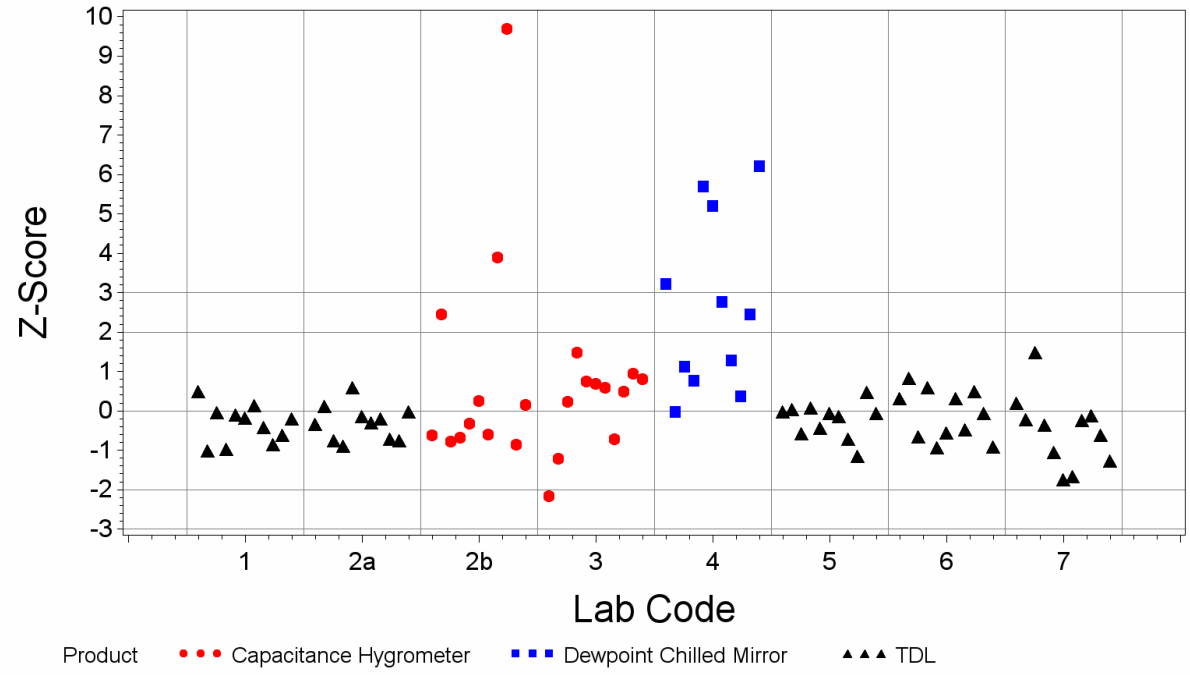


Figure 2. Comparison of Z-Scores Based on Sensor Type



6. Conclusions

Overall, the results from this study samples, except CRP 2.1 and Moist Snuff Mint, indicate a trend in values based on sensor type: TDL<Capacitance<Dewpoint. Water activity measured using a Tunable Diode Laser (TDL) sensor provided consistent results from one lab to another (n=3) 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. The TDL test time is similar to the chilled-mirror dewpoint sensor, since both methods require vapor equilibrium of the sample to the headspace. However, 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 that the chilled-mirror sensor, a longer equilibration time is required and volatiles can be absorbed by the sensor which may alter the calibration or otherwise interfere and degrade the sensor over time. Therefore, there was less consistency when the capacitance hygrometer or dewpoint chilled mirror was used. The TTPA recommends that a future collaborative study be conducted that focuses on the use of water activity meters equipped with TDL sensors and that a CRM be drafted.

APPENDIX A: Full Data Set

Lab Code	Product	a_w
1	1R6F ground filler/RT1	0,5605
1	1R6F ground filler/RT1	0,5617
1	1R6F ground filler/RT1	0,5609
2a	1R6F ground filler/RT1	0,5671
2a	1R6F ground filler/RT1	0,5700
2a	1R6F ground filler/RT1	0,5660
2b	1R6F ground filler/RT1	0,5602
2b	1R6F ground filler/RT1	0,5579
2b	1R6F ground filler/RT1	0,5589
3	1R6F ground filler/RT1	0,5690
3	1R6F ground filler/RT1	0,5690
3	1R6F ground filler/RT1	0,5700
4	1R6F ground filler/RT1	0,6190
4	1R6F ground filler/RT1	0,6160
4	1R6F ground filler/RT1	0,6170
5	1R6F ground filler/RT1	0,5586
5	1R6F ground filler/RT1	0,5572
5	1R6F ground filler/RT1	0,5576
6	1R6F ground filler/RT1	0,5530
6	1R6F ground filler/RT1	0,5520
6	1R6F ground filler/RT1	0,5540
7	1R6F ground filler/RT1	0,5500
7	1R6F ground filler/RT1	0,5520
7	1R6F ground filler/RT1	0,5530
1	CRP1.1	0,8733
1	CRP1.1	0,8732
1	CRP1.1	0,8647
2a	CRP1.1	0,8619
2a	CRP1.1	0,8700
2a	CRP1.1	0,8650
2b	CRP1.1	0,8534
2b	CRP1.1	0,8685
2b	CRP1.1	0,8702
3	CRP1.1	0,8570
3	CRP1.1	0,8550
3	CRP1.1	0,8540

Lab Code	Product	a_w
4	CRP1.1	0,8870
4	CRP1.1	0,8840
4	CRP1.1	0,8870
5	CRP1.1	0,8668
5	CRP1.1	0,8661
5	CRP1.1	0,8693
6	CRP1.1	0,8640
6	CRP1.1	0,8730
6	CRP1.1	0,8710
7	CRP1.1	0,8700
7	CRP1.1	0,8660
7	CRP1.1	0,8700
1	CRP2.1	0,8493
1	CRP2.1	0,8479
1	CRP2.1	0,8519
2a	CRP2.1	0,8595
2a	CRP2.1	0,8587
2a	CRP2.1	0,8548
2b	CRP2.1	0,8798
2b	CRP2.1	0,8728
2b	CRP2.1	0,8701
3	CRP2.1	0,8400
3	CRP2.1	0,8510
3	CRP2.1	0,8540
4	CRP2.1	0,8580
4	CRP2.1	0,8560
4	CRP2.1	0,8560
5	CRP2.1	0,8595
5	CRP2.1	0,8546
5	CRP2.1	0,8572
6	CRP2.1	0,8570
6	CRP2.1	0,8700
6	CRP2.1	0,8610
7	CRP2.1	0,8560
7	CRP2.1	0,8530
7	CRP2.1	0,8570
1	CRP3.1	0,4292
1	CRP3.1	0,4286

Lab Code	Product	a_w
1	CRP3.1	0,4286
2a	CRP3.1	0,3778
2a	CRP3.1	0,3764
2a	CRP3.1	0,3748
2b	CRP3.1	0,3756
2b	CRP3.1	0,3761
2b	CRP3.1	0,3749
3	CRP3.1	0,4530
3	CRP3.1	0,4520
3	CRP3.1	0,4440
4	CRP3.1	0,5140
4	CRP3.1	0,5160
4	CRP3.1	0,5170
5	CRP3.1	0,3903
5	CRP3.1	0,3910
5	CRP3.1	0,3857
6	CRP3.1	0,3860
6	CRP3.1	0,3830
6	CRP3.1	0,3830
7	CRP3.1	0,5400
7	CRP3.1	0,5440
7	CRP3.1	0,5430
1	CRP4.1	0,6767
1	CRP4.1	0,6773
1	CRP4.1	0,6756
2a	CRP4.1	0,6732
2a	CRP4.1	0,6809
2a	CRP4.1	0,6782
2b	CRP4.1	0,6801
2b	CRP4.1	0,6787
2b	CRP4.1	0,6805
3	CRP4.1	0,7030
3	CRP4.1	0,7030
3	CRP4.1	0,7060
4	CRP4.1	0,7060
4	CRP4.1	0,6910
4	CRP4.1	0,6910
5	CRP4.1	0,6867

Lab Code	Product	a_w
5	CRP4.1	0,6891
5	CRP4.1	0,6888
6	CRP4.1	0,6920
6	CRP4.1	0,6940
6	CRP4.1	0,6960
7	CRP4.1	0,6890
7	CRP4.1	0,6770
7	CRP4.1	0,6840
1	Cigar Filler #1-11/17	0,5993
1	Cigar Filler #1-11/17	0,5926
1	Cigar Filler #1-11/17	0,5928
2a	Cigar Filler #1-11/17	0,5917
2a	Cigar Filler #1-11/17	0,5977
2a	Cigar Filler #1-11/17	0,5965
2b	Cigar Filler #1-11/17	0,6017
2b	Cigar Filler #1-11/17	0,6002
2b	Cigar Filler #1-11/17	0,5988
3	Cigar Filler #1-11/17	0,6060
3	Cigar Filler #1-11/17	0,6030
3	Cigar Filler #1-11/17	0,6080
4	Cigar Filler #1-11/17	0,6620
4	Cigar Filler #1-11/17	0,6620
4	Cigar Filler #1-11/17	0,6590
5	Cigar Filler #1-11/17	0,5971
5	Cigar Filler #1-11/17	0,5979
5	Cigar Filler #1-11/17	0,5939
6	Cigar Filler #1-11/17	0,5930
6	Cigar Filler #1-11/17	0,5870
6	Cigar Filler #1-11/17	0,5910
7	Cigar Filler #1-11/17	0,5770
7	Cigar Filler #1-11/17	0,5750
7	Cigar Filler #1-11/17	0,5750
1	Cigar Filler #2-11/17	0,5787
1	Cigar Filler #2-11/17	0,5784
1	Cigar Filler #2-11/17	0,5777
2a	Cigar Filler #2-11/17	0,5766
2a	Cigar Filler #2-11/17	0,5713
2a	Cigar Filler #2-11/17	0,5627

Lab Code	Product	a_w
2b	Cigar Filler #2-11/17	0,5702
2b	Cigar Filler #2-11/17	0,5675
2b	Cigar Filler #2-11/17	0,5570
3	Cigar Filler #2-11/17	0,5900
3	Cigar Filler #2-11/17	0,5860
3	Cigar Filler #2-11/17	0,5840
4	Cigar Filler #2-11/17	0,6280
4	Cigar Filler #2-11/17	0,6250
4	Cigar Filler #2-11/17	0,6270
5	Cigar Filler #2-11/17	0,5747
5	Cigar Filler #2-11/17	0,5721
5	Cigar Filler #2-11/17	0,5721
6	Cigar Filler #2-11/17	0,5830
6	Cigar Filler #2-11/17	0,5790
6	Cigar Filler #2-11/17	0,5820
7	Cigar Filler #2-11/17	0,5440
7	Cigar Filler #2-11/17	0,5440
7	Cigar Filler #2-11/17	0,5470
1	Mentholated Cigarette	0,5154
1	Mentholated Cigarette	0,5218
1	Mentholated Cigarette	0,5145
2a	Mentholated Cigarette	0,5107
2a	Mentholated Cigarette	0,5154
2a	Mentholated Cigarette	0,5123
2b	Mentholated Cigarette	0,5112
2b	Mentholated Cigarette	0,5097
2b	Mentholated Cigarette	0,5068
3	Mentholated Cigarette	0,5730
3	Mentholated Cigarette	0,5710
3	Mentholated Cigarette	0,5740
4	Mentholated Cigarette	0,6280
4	Mentholated Cigarette	0,6250
4	Mentholated Cigarette	0,6240
5	Mentholated Cigarette	0,5564
5	Mentholated Cigarette	0,5575
5	Mentholated Cigarette	0,5535
6	Mentholated Cigarette	0,5370
6	Mentholated Cigarette	0,5340

Lab Code	Product	a_w
6	Mentholated Cigarette	0,5390
7	Mentholated Cigarette	0,5060
7	Mentholated Cigarette	0,5240
7	Mentholated Cigarette	0,5230
1	Moist Snuff-Mint	0,8679
1	Moist Snuff-Mint	0,8696
1	Moist Snuff-Mint	0,8687
2a	Moist Snuff-Mint	0,8695
2a	Moist Snuff-Mint	0,8701
2a	Moist Snuff-Mint	0,8713
2b	Moist Snuff-Mint	0,8965
2b	Moist Snuff-Mint	0,8988
2b	Moist Snuff-Mint	0,9012
3	Moist Snuff-Mint	0,8680
3	Moist Snuff-Mint	0,8670
3	Moist Snuff-Mint	0,8650
4	Moist Snuff-Mint	0,8840
4	Moist Snuff-Mint	0,8790
4	Moist Snuff-Mint	0,8790
5	Moist Snuff-Mint	0,8690
5	Moist Snuff-Mint	0,8671
5	Moist Snuff-Mint	0,8641
6	Moist Snuff-Mint	0,8680
6	Moist Snuff-Mint	0,8680
6	Moist Snuff-Mint	0,8690
7	Moist Snuff-Mint	0,8710
7	Moist Snuff-Mint	0,8740
7	Moist Snuff-Mint	0,8650
1	Moist Snuff-Wintergreen	0,8266
1	Moist Snuff-Wintergreen	0,8259
1	Moist Snuff-Wintergreen	0,8266
2a	Moist Snuff-Wintergreen	0,8253
2a	Moist Snuff-Wintergreen	0,8272
2a	Moist Snuff-Wintergreen	0,8277
2b	Moist Snuff-Wintergreen	0,8517
2b	Moist Snuff-Wintergreen	0,8558
2b	Moist Snuff-Wintergreen	0,8571
3	Moist Snuff-Wintergreen	0,8290

Lab Code	Product	a_w
3	Moist Snuff-Wintergreen	0,8290
3	Moist Snuff-Wintergreen	0,8320
4	Moist Snuff-Wintergreen	0,8310
4	Moist Snuff-Wintergreen	0,8290
4	Moist Snuff-Wintergreen	0,8290
5	Moist Snuff-Wintergreen	0,8253
5	Moist Snuff-Wintergreen	0,8243
5	Moist Snuff-Wintergreen	0,8271
6	Moist Snuff-Wintergreen	0,8310
6	Moist Snuff-Wintergreen	0,8290
6	Moist Snuff-Wintergreen	0,8300
7	Moist Snuff-Wintergreen	0,8280
7	Moist Snuff-Wintergreen	0,8300
7	Moist Snuff-Wintergreen	0,8270
1	RT6 - Flavoured Cigar Filler	0,5979
1	RT6 - Flavoured Cigar Filler	0,5956
1	RT6 - Flavoured Cigar Filler	0,5961
2a	RT6 - Flavoured Cigar Filler	0,5998
2a	RT6 - Flavoured Cigar Filler	0,5988
2a	RT6 - Flavoured Cigar Filler	0,5971
2b	RT6 - Flavoured Cigar Filler	0,6040
2b	RT6 - Flavoured Cigar Filler	0,5980
2b	RT6 - Flavoured Cigar Filler	0,5997
3	RT6 - Flavoured Cigar Filler	0,6080
3	RT6 - Flavoured Cigar Filler	0,6080
3	RT6 - Flavoured Cigar Filler	0,6080
4	RT6 - Flavoured Cigar Filler	0,6710
4	RT6 - Flavoured Cigar Filler	0,6680
4	RT6 - Flavoured Cigar Filler	0,6650
5	RT6 - Flavoured Cigar Filler	0,6047
5	RT6 - Flavoured Cigar Filler	0,5927
5	RT6 - Flavoured Cigar Filler	0,5972
6	RT6 - Flavoured Cigar Filler	0,5740
6	RT6 - Flavoured Cigar Filler	0,5980
6	RT6 - Flavoured Cigar Filler	0,5940
7	RT6 - Flavoured Cigar Filler	0,5840
7	RT6 - Flavoured Cigar Filler	0,5860
7	RT6 - Flavoured Cigar Filler	0,5840

APPENDIX B: Raw Data Plots