Smoke Analysis Sub-Group
(Routine Analytical Chemistry Sub-Group)

Technical Report

2019 Initial Collaborative Study for the Determination of Very Low Nicotine in Total Particulate Matter from the Mainstream Smoke

November 2021

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1. Summary

At the October 2019 CORESTA Routine Analytical Chemistry Sub-Group (RAC) meeting held in Hamburg, the Sub-Group initiated a collaborative study to verify the feasibility of expanding the calibration range of ISO 10315:2021 Cigarettes – Determination of nicotine in total particulate matter from the mainstream smoke – Gas-chromatographic method. The intent of this study was to provide an assessment of laboratory capability to determine very low nicotine (VLN) in total particulate matter from mainstream smoke generated under ISO 3308 smoking conditions. The International Standard ISO 10315:2021 was developed by ISO/TC126 and includes r and R values from CORESTA Recommended Method (CRM) No 7 - Determination of Nicotine in Mainstream Smoke of Cigarettes by Gas Chromatographic Analysis published in 1991. The results of the study demonstrate that, with modification to the calibration range, ISO 10315 is suitable for the determination of very low nicotine in total particulate matter from mainstream cigarette smoke. This important project confirms ISO 10315 is relevant for emerging regulations to reduce nicotine for combusted tobacco products[1,2].

2. Introduction

In 1990, the CORESTA Task Force on the Review of Smoking Methods coordinated a major international study for the determination of NFDPM and nicotine in total particulate matter from mainstream smoke. The study involved 30 laboratories and included six samples covering a range for TPM (1 mg/cig -17 mg/cig) and nicotine (0,09 mg/cig - 1,41 mg/cig) smoked under ISO 3308 (non-intense) smoke regime. The results established a set of CORESTA reference methods for machine smoking of cigarettes. In 1991, CORESTA Recommended Method No 7 - Determination of Nicotine in Mainstream Smoke of Cigarettes by Gas Chromatographic Analysis was published and made available to the International Standards Organization[3]. In 2000, ISO/TC 126 developed International Standard ISO 10315 based on CRM No 7.

In late 2019, RAC initiated a collaborative study to verify the feasibility of lowering the calibration range for the method according to ISO 10315 to analyse very low nicotine smoke yields. Since commercially made cigarettes with reduced nicotine levels were not available, two ventilated cigarettes were provided for this study with expected nicotine yields in smoke (0,02-0,05 mg/cig) in the range of reported nicotine yields for SPECTRUM® research cigarettes and VLN™ cigarettes produced by 22nd Century[4,5]. A traditional nicotine content reference cigarette and a monitor test piece (t.p.) were included to verify that changes in the method required to quantitate very low nicotine content in total particulate matter did not negatively affect the analysis of nicotine in mainstream smoke from traditional nicotine content cigarettes. The protocol for this study was distributed in November 2019 and the study was conducted in December 2019 through February 2020. Data were collected from the participating laboratories and statistically evaluated in basic conformance with the recommendations of ISO 5725-2:1994 and ISO/TR 22971:2005

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[1] World Health Organization (2015) Advisory Note: Global Nicotine Reduction Strategy - ISBN 978 92 4 150932 9, WHO Study Group on Tobacco Product Regulation (TobReg), (https://www.who.int/tobacco/publications/prod_regulation/nicotine-reduction/en/).

[2] Tobacco Product Standard for Nicotine Level of Combusted Cigarettes, 83 Federal Register (March 16, 2018)

[3] Task Force on the Review of Smoking Methods Technical Report: CORESTA Report 1991-1, September 2019

[4] Ding Y.S., Richter P., Hearn B., et al. Chemical Characterization of Mainstream Smoke from SPECTRUM Variable Nicotine Research Cigarettes; Tob. Regul. Sci. 2017;3(1):81-94.

[5] Carmines E., Gillman G., Comparison of the Yield of Very Low Nicotine Content Cigarettes to the Top 100 United States Brand Styles; Beiträge zur Tabakforschung International 28 (2019), 253-266.

2.1 Objective

The objective of this study was to verify that the modifications to ISO 10315 calibration range are fit for purpose to determine nicotine in mainstream smoke yields, 0,02 to 1,5 (mg/cig or t.p.), in a variety of cigarettes and provide an assessment of inter-laboratory capability.

3. Organization

3.1 Participants

Thirteen laboratories participated in the study using linear and/or rotary smoking machines. Nine participants provided data using a linear smoke machine, five provided data using a rotary smoke machine, and included one laboratory that provided data with both for a total of 14 data sets. A list of the participating laboratories is provided in Table 1. The laboratories are listed in alphabetical order. The numerical laboratory codes used in this report do not correspond to the same order as shown in the table below.

Table 1: List of Participating Laboratories

Participants
Altria Client Services LLC, United States
British American Tobacco, Germany
C.I.T. Montepaz S.A., Uruguay
China National Tobacco Corporation (CNTC) Beijing Cigarette Factory, China
China National Tobacco Quality Supervision and Test Centre (CNTQTC), China
Enthalpy Analytical Richmond, United States
Inciensa, Costa Rica
ITC Limited, India
ITG Brands LLC, United States
Japan Tobacco Inc., Japan
Labstat International ULC, Canada
Liggett Group, United States
Philip Morris International, Poland
RJ Reynolds tobacco Company, United States
Reemtsma/Imperial Tobacco, Germany

3.2 Protocol

The Protocol is provided in Appendix A and specific details from the protocol are described below.

3.2.1 Study Samples

Laboratories were responsible for procuring study cigarettes University of Kentucky Reference Cigarette 1R6F and CORESTA Monitor 9 test piece. Study cigarettes, Dunhill and Rothmans were provided by British American Tobacco. Laboratories were requested to store the samples at ambient conditions if they were analysed within 2 weeks or in a refrigerator at approximately 4 °C prior to conditioning if the analyses would be conducted greater than two weeks from receipt. The study was to be conducted from November 2019 through January 2020. Laboratories were requested to submit data by January 31, 2020. The final data, including re-checks, were received by March 2020. The samples are identified in Table 2.

Table 2: Sample Identification

Sample Name	Description	Mean TPM Yields (mg/cig or mg/t.p.)[1]
Dunhill	Dunhill cigarette	0,2
Rothmans	Rothmans cigarette	0,7
1R6F	American blended reference cigarette, unflavored	10,1
CM9	CORESTA Approved Monitor No. 9	17,2

[1] Mean TPM (total particulate matter) yields in this study

3.2.2 Analysis

Participants were requested to conduct three independent smoke runs using linear and/or rotary smoke machines. For linear smoking, a test result was defined as the average of four independent replicates per sample per smoke run. A replicate was defined as five cigarettes per port or one 44 mm Cambridge filter pad. For rotary smoking, a replicate was defined as 20 cigarettes per smoke run. Participants were to follow the ISO standards listed below for smoking and analysis.

- ISO 3402, Tobacco and tobacco products – Atmosphere for conditioning and testing
- ISO 3308, Routine analytical cigarette-smoking machine. Definitions and standard conditions
- ISO 4387, Cigarettes – Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine
- ISO 10315, Cigarettes – Determination of nicotine in total particulate matter from the mainstream smoke – Gas-chromatographic method

Participants were asked to create a composite of five packs per ISO 8243, condition samples per ISO 3402 prior to smoking.

3.2.3 Sample Analysis and Data Reporting

Participating laboratories were requested to perform replicate analyses for each sample and use the current version of ISO 10315 with the method modifications stated in the protocol and summarized below.

- ISO 10315: Cigarettes – Determination of nicotine in total particulate matter from the mainstream smoke – Gas-chromatographic method
    - The calibration range was expanded from 0,02 mg/mL (0,08 mg/cig) to 0,002 mg/mL (0,008 mg/cig). The modification is a factor of 10 lower. Participants were instructed to prepare at least five nicotine standard solutions and use a linear regression equation with 1/x and 1/x2, x being concentration.

- Acceptance criteria for R2 >0,990 and % relative error ≤ 15 % for calibration level 1 (0,002 mg/mL) and ≤ 10 % relative error for calibration levels ≥ 0,005 mg/mL.
- Participants were instructed to report Nicotine (mg/cig) to three decimal places.

3.2.4 Deviations

Participating laboratories were requested to document any deviations from International Standard ISO 10315 protocol and any event that may have influenced a test result and submit the deviations with their results. As stated in the protocol, data submitted with significant deviations from the applicable International Standard would be excluded from the study. There were no deviations reported by the laboratories.

4. Study Data

The raw data set, without removal of outliers, is provided in Appendices B. Each linear analysis includes three test results (a test result consisting of 20 cigarettes). The type of smoking machine and the number of cigarettes smoked per replicate for each participating laboratory are shown in the table below:

Table 3: Smoking Machine Type and Number of Cigarettes per Port for each Participating Laboratory

Laboratory code	Smoking Machine Type	# of Cigarettes per Port
1	Linear	5
1	Rotary	20
2	Rotary	20
3	Linear	5
4	Rotary	20
5	Linear	5
6	Linear	5
7	Linear	5
8	Rotary	20
10	Linear	5
11	Rotary	20
12	Linear	5
13	Linear	5
14	Linear	5

5. Statistical Analysis

The statistical analysis was conducted in basic conformance with ISO 5725-2:1994 and ISO/TR 22971:2005. 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 C.

5.1 Exclusion of Outliers

An adaptation of Levene's Test[6] was used for eliminating laboratories with overly large repeatability standard deviations and Grubbs' Test was used to eliminate laboratories with outlying mean values. In general, Levene's Test tends to identify fewer outliers than Cochran's Test and in some instances may result in a larger estimated r value than if Cochran's Test was used. There is not a consistent effect on the estimated R value.

In this instance, there were no labs identified as outliers.

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. 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 as typically employed may lead to incorrect conclusions. For those reasons, we do not include Mandel's h and k plots.

5.2 Calculation of Repeatability and Reproducibility

After removal of outlying data based on numerical data consistency methods discussed above (Grubbs' Test and Levene's Test), the final repeatability and reproducibility (r & R) results were calculated and are shown in Tables 4-5. The r & R results reflect both laboratory variability and product consistency.

Table 4: Repeatability (r) and Reproducibility (R) Limits for Nicotine (mg/cig or mg/t.p.) (all machines) Determined Using a Linear Regression Equation with 1/x Weighting

Product	No. of Labs *	Mean (mg/cig or t.p.)	Repeatability		Reproducibility	
			r	r (%)	R	R (%)
Dunhill	14	0,025	0,0072	28,9 %	0,0155	62,3 %
Rothmans	14	0,059	0,0127	21,5 %	0,0210	35,7 %
1R6F	13	0,717	0,0534	7,4 %	0,1315	18,4 %
CM9	13	1,50	0,088	5,9 %	0,2643	17,6 %

* The number of laboratory data sets after removal of outliers.

Table 5: Repeatability (r) and Reproducibility (R) Limits for Nicotine (mg/cig or mg/t.p.) (all machines) determined using a linear regression equation with $1/x^2$ weighting

Product	No. of Labs *	Mean (mg/cig or t.p.)	Repeatability		Reproducibility	
			r	r (%)	R	R (%)
Dunhill	14	0,025	0,0073	29,3 %	0,0154	62,1 %
Rothmans	14	0,059	0,0132	22,2 %	0,0204	34,3 %
1R6F	13	0,729	0,0555	7,6 %	0,1248	17,1 %
CM9	13	1,52	0,0902	5,9 %	0,2407	15,8 %

* The number of laboratory data sets after removal of outliers.

[6] The approach is discussed in detail by Michael Morton in "Within-Laboratory Variance Outlier Detection: An Alternative to Cochran's Test" in Beiträge zur Tabakforschung International, Vol 27 No. 7, pp135-144.

6. Data Interpretation

For each of the products tested, results for mean and r and R in Table 4 using linear regression with weighting 1/x are in good agreement with results in Table 5 with a weighting $1/x^2$. The variability of the results for CM9 and 1R6F in this study are comparable on both TPM and nicotine to previous reported studies[7,8,9]. For Dunhill and Rothmans, TPM and nicotine levels are 93 % to 98 % lower than values for CM9 and 1R6F. However, the variability of results for the Dunhill and Rothmans are less than several results in the 1990 study that formed the basis for this International Standard[9,10]. From the raw data graphs in Appendix C, there is generally little difference between calibrating using 1/x and $1/x^2$ weighting. A possible exception being Lab 13 where the difference is larger, approximately 9 % for CM9 and 1R6F.

In Figures 1-2 each laboratory's average (n=3 test results) nicotine result (mg/cig or mg/t.p.) for 1/x and $1/x^2$ weightings per product are shown. Overall, results for CM9, 1R6F and Rothmans samples trend $1/x^2 > 1/x$.

Figure 1. Each Labs CM9 and 1R6F Average Test Result (n=3).

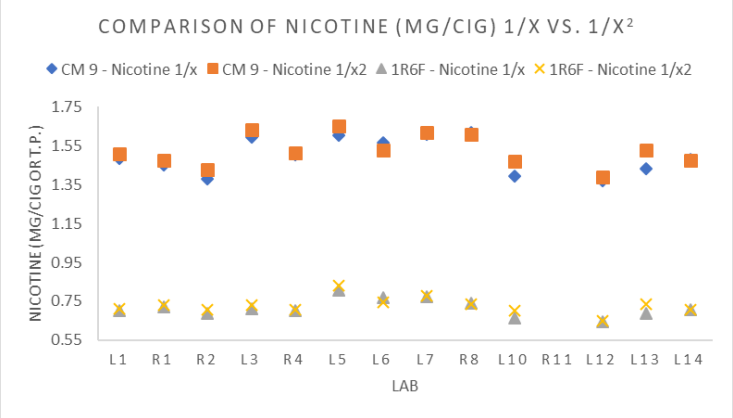
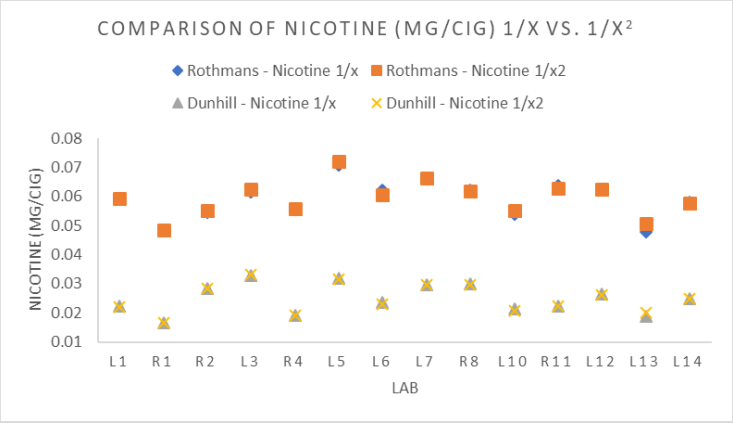


Figure 2. Each Labs Rothmans and Dunhill Average Test Result (n=3).



[7] CORESTA Routine Analytical Chemistry Sub-Group Technical Report: 2018 Collaborative Study of CORESTA Monitor 8 (CM8) and 9 (CM9), February 2019

[8] Center for Tobacco Reference Products. Certificate of Analysis 1R6F Certified Reference Cigarette. Vol. 2016-002CTRP. Lexington (KY): University of Kentucky; 2016.

[9] Task Force on the Review of Smoking Methods Technical Report: CORESTA Report 1991-1, September 2019

[10] International Standard ISO 10315 Cigarettes — Determination of nicotine in total particulate matter from the mainstream smoke — Gas-chromatographic method

For Rothmans the difference between ($1/x^2$-$1/x$) weighted results is proportionally smaller relative to CM9 and 1R6F. For Dunhill, laboratory results generally showed a different trend ($1/x > 1/x^2$) but the difference is less than one microgram of nicotine per cigarette.

The calibration range for ISO 10315 was lowered by a factor of 10 to bracket very low nicotine content in total particulate matter samples. The study protocol did not require any additional modifications to the International Standard. Laboratories were requested cover a range of nicotine from 0.002 to 1.0 mg/mL for at least five calibration standard levels, use $1/x$ and $1/x^2$ weightings to reduce the error at the low end of the calibration range, provide nicotine concentrations per calibration standard levels, %RCR (i.e. percent error) for each level, signal to noise ratios (S/N) for the two lowest standards. These values and information for GC inlet conditions and columns are provided in Appendix D.

In examining %RCR, it looked like there was a small improvement in %RCR at the low end of the calibration range using $1/x^2$ weighting, but it also appeared to negatively impact the accuracy at the high end of the calibration range. Generally, the %RCR for lowest calibration level 0,002 mg is ≤15 % RCR and ≤10 % RCR for the remaining calibration levels (0,005 to 1,0 mg/mL). In Table 6 each laboratory's linear regression equation correlation coefficient, slope, y-intercept for each weighting are shown. Generally, the values for slope (m) are greater and values for y-intercepts (b) are less in linear regression equations with 1/x weighting than $1/x^2$ weighting.

Table 6. Summary of linear regression equations with weightings 1/x and $1/x^2$

Lab	Smoke Machine	1/x			$1/x^2$		
		R^2	m	b	R^2	m	b
L1	Linear	0,99989	3,040	-0,00049	0,99976	2,994	-0,00002
R1	Rotary Day 1	0,99990	3,041	-0,00049	0,99976	2,995	-0,00002
R1	Rotary Day 2	0,99989	3,042	-0,00065	0,99976	2,998	-0,00021
R2	Rotary	0,99995	1,991	-0,00147	0,99786	1,924	-0,00079
L3	Linear	0,99923	0,450	0,00096	0,9984	0,462	0,00071
R4	Rotary	0,99997	2,490	-0,00017	0,99991	2,472	-0,00007
L5	Linear	0,99957	2,480	-0,00002	0,99866	2,403	0,00096
L6	Linear	0,99994	0,876	NA	0,99958	0,900	NA
L7	Linear	0,99882	0,831	-0,00071	0,99932	0,825	-0,00052
R8	Rotary Day 1	0,99991	7,529	-0,00404	0,99897	7,336	-0,00207
R8	Rotary Day 2	0,99969	7,576	-0,00376	0,99902	7,360	-0,00156
L10	Linear	0,9989	2,320	-0,00242	0,9944	2,190	-0,00137
R11	Rotary Day 1&2	0,99993	0,056	-0,00253	0,99990	0,054	-0,00193
L12	Linear	0,99997	3,770	-0,00090	0,99986	3,720	-0,00037
L13	Linear	0,9983	3,255	-0,00160	0,9926	2,968	-0,00030
L14	Linear	0,99997	0,928	0,00054	0,99968	0,931	0,00047

The general trend for slope (m) values where $1/x > 1/x^2$ provides a rationale for the trend in nicotine results for CM9, 1R6F ($1/x^2 > 1/x$). For Lab 13, the average % difference in slope (m) between weightings is approximately 9 % which agrees with the average % difference in nicotine results between the weightings for CM9 and 1R6F. Table 7 shows each product average percent difference in nicotine results (mg/cig or mg/t.p.) divided by average percent difference in slope (m) between the two weightings ($1/x^2$-$1/x$) per laboratory. The reason for normalizing by the slope (m) is to allow for a simple mode for comparison.

Table 7. Comparison of Average % Difference in Nicotine results (mg/cig or t.p.) Normalized to the Average % Difference in Slope (m) $1x^2$ -1/x

Lab	Smoke Machine	CM9	1R6F	Rothmans	Dunhill
1	Linear	0,97	0,94	0,30	0,84
1	Rotary	0,98	0,91	0,45	0,00
2	Rotary	0,98	0,97	0,36	0,00
3	Linear	0,99	0,97	0,55	0,41
4	Rotary	0,68	0,63	0,52	0,00
5	Linear	0,99	0,96	0,37	0,25
6	Linear	1,00	1,00	1,00	1,00
7	Linear	0,98	0,95	0,41	0,40
8	Rotary	0,20	0,20	0,23	0,30
10	Linear	0,93	0,91	0,26	0,65
11	Rotary	NA	NA	0,32	0,00
12	Linear	0,97	0,94	0,32	0,64
13	Linear	0,99	0,98	0,69	0,25
14	Linear	1,05	1,19	3,74	0,00

Generally, for CM9 and 1R6F there is a good correlation between the change in slope values for the two weightings and the change in nicotine results. However, for Rothmans and Dunhill, the change in very low nicotine in smoke yields ($1/x^2$-$1/x$) may not be fully explained by the difference in slope (m) values between the two weightings. It seems reasonable that the y-intercept (b) value plays a greater role in the nicotine result as the test portion's response ratio (y) decreases.

7. Recommendations

In 2019, RAC conducted a collaborative study for the determination of very low nicotine in total particulate matter. The International Standard listed below was originally developed for the analysis of nicotine in total particulate matter from the mainstream smoke generated under ISO 3308 (non-intense) conditions.

- ISO 10315: 2021 *Cigarettes – Determination of nicotine in total particulate matter from the mainstream smoke – Gas-chromatographic method*

In this study the calibration range was expanded 10-fold, from 0,02 mg/mL to 0,002 mg/mL (0,08 mg/cig to 0.008 mg/cig), to bracket very low nicotine content in total particulate matter. In examining %RCR, it looked like there was a small improvement in %RCR at the low end of the calibration range using $1/x^2$ weighting, but it also appeared to negatively impact the accuracy at the high end of the calibration range. However, it does not appear that the extension of the calibration range negatively impacts the analysis of nicotine in mainstream smoke from conventional cigarettes smoked under non-intense conditions.

The two weighting functions produced similar reproducibility statistics and compared reasonably to previous studies. There was generally very little difference between the results using either of the two calibration weightings. Overall, there was no compelling reason to choose $1/x^2$ weighting over 1/x weighting. These observations support the RAC's recommendation that 1/x weighting function is used when modifications to ISO 10315 calibration range are made to expand the method scope to include VLN and conventional particulate matter from the mainstream smoke.

Acknowledgments

The authors appreciated the support of Dr. Guy Jaccard, Dr. Jana Jeffrey and British American Tobacco for distributing samples to participants in the study. Additional gratitude to the 13 participants that provided data.

APPENDIX A: Study Protocol

CORESTA Routine Analytical Chemistry Sub-Group

Project Title: Initial Study for the Determination of Very Low Nicotine (VLN) in Smoke Condensates

Type of Document: Collaborative Study Protocol

Date: October 10, 2019

Written by: Anthony Brown

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

1. Introduction

At the CORESTA Routine Analytical Chemistry (RAC) Subgroup meeting held on October 5th, 2019 in Hamburg, the group proposed an initial collaborative study to verify the feasibility of expanding the calibration range of ISO 10315: *Cigarettes – Determination of nicotine in smoke condensates – Gas-chromatographic method* to expand the scope to include very low nicotine (VLN) content in smoke condensates. This ISO method was developed and includes r and R values from CORESTA Recommended Method (CRM) No 7 published in 1991.

2. Objective

The objective of this collaborative study is to verify the feasibility to expand the scope of ISO 10315 beyond traditional nicotine content in smoke condensates to include very low nicotine (VLN) content in smoke condensates. This will necessitate modifications to the following ISO method:

Since VLN cigarettes are not available, this study will include two highly ventilated cigarettes and traditional nicotine content reference cigarettes and test pieces to provide a range of very low nicotine to traditional nicotine content in smoke condensates.

The final output will be a presentation of the results at the Spring 2020 RAC Sub-Group meeting, and one technical report, including repeatability (r) and reproducibility (R) values and recommendations for future work.

Note: Since the purpose of this study is to verify the feasibility of expanding the calibration range of ISO 10315 method to include VLN smoke condensates and calculate new r&R values, it is imperative that participants follow the specified normative references provided below and follow the method modifications to ISO 10315 that are detailed in Appendix 1 of this protocol.

3. Normative References

ISO Standard	Title
ISO 8243	Cigarettes – Sampling
ISO 3402	Tobacco and tobacco products – Atmosphere for conditioning and testing
ISO 3308	Routine analytical cigarette-smoking machine – Definitions and standard conditions
ISO 4387	Cigarettes – Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine
ISO 10315	Cigarettes – Determination of nicotine in smoke condensates – Gas-chromatographic method

4. Time schedule

Table 1: Study timeline

Date	Activity
October 21, 2019	Distribute the study protocol and data reporting sheet
October 23, 2019	Laboratories submit shipping information for BAT provided commercial cigarettes
November 2019	Order 1R6F reference and CM9 monitor cigarettes
November 2019	BAT will distribute two commercial cigarettes
November 2019-January 2020	Laboratories conduct the study.
January 31, 2020	Laboratories submit results by this date
April 23, 2020	Discuss results at spring RAC SG meeting

5. 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, Hiromoto Yamazaki and Thomas Schmidt of their interest to participate.

Table 2: Participating Laboratories

Participating Laboratories
Altria Client Services LLC, United States
British American Tobacco, Germany
C.I.T. Montepaz S.A., Uruguay
China National Tobacco Corporation (CNTC) Beijing Cigarette Factory, China
China National Tobacco Quality Supervision and Test Centre (CNTQTC), China
Enthalpy Analytical Richmond, United States
Inciensa, Costa Rica
ITC Limited, India
ITG Brands LLC, United States
Labstat International ULC, Canada
Liggett Group, United States
Philip Morris International, Poland
RJ Reynolds tobacco Company, United States
Reemtsma/Imperial Tobacco, Germany
Japan Tobacco Inc., Japan

6. Samples

Order the samples from the suppliers listed in Table 3. The approximate nicotine yield is provided for each sample in order to show the range for the various samples. Ordering information is shown below:

- 1R6F cigarette samples: Ordering information can be found at the University of Kentucky website for reference products. https://ktrdc.ca.uky.edu/Reference-Products
- CORESTA Monitor No. 9: Monitor test pieces are available in units of 5000 test pieces: Ordering information can be obtained through:

Borgwaldt KC GmbH

Tel: +49 40 85 31 380
Fax: +49 40 850 56 00

e-mail: BKC@borgwaldt.com

Cerulean

Tel: +44 1908 23 38 33

Fax: +44 1908 23 53 33

e-mail: info@cerulean.com

- BAT commercial cigarette samples: These samples will be distributed by British American Tobacco in November.

Note: Upon receipt samples should be stored at ambient conditions if they will be analyzed within 2 weeks. Samples held longer than 2-weeks prior to analysis should be stored in the refrigerator at approximately 4 °C. All remaining samples should be retained in sealed containers at −20 °C as they may be used for future collaborative studies for other analytes.

Table 3: Samples

Approximate Nicotine per cigarette (mg/cig)	Sample Name	Description	Provider	Quantity to Request
0,01	Dunhill	Dunhill cigarette	British American Tobacco	Carton
0,05	Rothmans	Rothmans cigarette	British American Tobacco	Carton
0,721[11]	1R6F	American blended reference cigarette, unflavored	University of Kentucky	Case
1,480[12]	CM9	CORESTA Approved Monitor No. 9	Borgwaldt KC GmbH or Cerulean UK	Case

[11] Certificate of Analysis – 1R6F Certified Reference Cigarette, Certificate Number 2017-002CTRP, Center for Tobacco Reference Products, University of Kentucky, August 24, 2017

[12] Routine Analytical Chemistry Sub-Group – CORESTA Technical Report [RAC-187-CTR], 2018 Collaborative Study of CORESTA Monitor 8 (CM8) and 9 (CM9) for the Determination of Test Piece Weight, TPM, Water, Nicotine, NFDPM, Carbon Monoxide and Puff Count Obtained under Mainstream 'ISO' and 'Intense' Smoking Regimes, February 2019.

7. Smoke Methods

7.1. General Guidance

As a general guide, experiments conducted under the smoking regime provided in Table 4 shall be conducted according to the ISO standards provided in Section 3. Please note any deviations and any event that may have influenced a test result in the reporting spreadsheet. Exceptions to this guiding principle include the number of cigarettes smoked per sample and the butt length for smoking CM9 test pieces and 1R6F cigarettes. Participants shall use specified butt lengths for CM9 monitor and 1R6F reference cigarettes provided below in section 6.1.3. Specifications for ISO 3308 smoke regime are summarized in Table 4.

Table 4: Smoking Regime Specifications

Smoking Regime	Puff Volume (mL)	Puff Interval* (s)	Puff Duration (s)	Vent Blocking (%)
ISO 3308	35 ± 0.3	60 ± 0.5	2 ± 0.02	0

*** Note: Puff Interval is time in seconds from the start of one puff to the start of the next puff.**

7.1.1 Test Item Storage

Upon receipt samples should be stored at ambient conditions if they will be analysed within 2 weeks. Samples held longer than 2-weeks prior to analysis should be stored in the refrigerator at approximately 4 °C prior to conditioning.

7.1.2 Sampling

A composite of five packs should be prepared for conditioning according to ISO 8243.

7.1.3 Butt Marking

CM9 test pieces shall be butt marked to a fixed butt length of 33 mm. 1R6F reference cigarettes shall be butt marked to a fixed butt length of 35 mm. BAT provided study samples, Dunhill and Rothmans cigarettes, shall be butt marked as per ISO 4387.

7.1.4 Conditioning

All samples shall be conditioned as per ISO 3402 prior to smoking.

7.1.5 Replicate Required per Smoke Run

Note: Replicates shall be obtained under repeatability conditions i.e. same instrument and same operator.

- Linear Smoking: Conduct four (4) independent replicate analyses for each sample type per smoke run.
- Rotary Smoking: Conduct one (1) independent replicate analysis for each sample type per smoke run.

7.1.6 Smoking Machine

Participants should confirm the type of smoking machine being used (linear or rotary) and report the model and manufacturer. The temperature and relative humidity at the time the smoking is conducted should be recorded.

Where laboratories have more than one type of smoking machine (i.e. linear and rotary variants) they are encouraged to generate results separately using both linear and rotary smoking machines.

7.1.7 Smoking Plans

The smoking plans will incorporate products listed in Table 3, shown as M1 for CM9, M2 for 1R6F, B1for Dunhill, and B2 for Rothmans cigarette samples. These plans are designed to smoke each sample type in all ports for the ISO 3308 puffing regime, for linear and rotary smoking in a (1) day.

Specifically, for rotary smoking due to the required smoke runs, participants may allow for two days of smoking. However, three independent replicates for each sample type shall be conducted in a (1) day.

If possible, each participating laboratory should use only one operator (for each smoking machine type) throughout the course of the study.

Note: The side seam of the test pieces should be positioned at random and not in a fixed position since it has been observed that some laboratory staff habitually place cigarettes in a particular orientation.

Note: Special precautions should be taken in placing Dunhill and Rothmans cigarettes next to traditional nicotine delivery cigarettes due to possible nicotine contamination from side stream smoke.

- Linear machine smoking: A test result is defined as the mean yield obtained from smoking 20 test pieces in a single smoking machine run, therefore it is the result of four ports, five test pieces per port.
    - Perform three smoke runs in one day following the smoke plan provided in Table 5 resulting in three (3) test results per sample type.

Table 5: Smoking Plan for 20-port Linear Machine (5 test pieces per pad)

Day	Run	Port number																			
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
1	1	M1	M2	M1	M2		B1	B2	B1	B2		B1	B2	B1	B2		M1	M2	M1	M2	
	2	B2	B1	B2	B1		B2	B1	B2	B1		M2	M1	M2	M1		M2	M1	M2	M1	
	3	B1	B2	B1	B2		M1	M2	M1	M2		M1	M2	M1	M2		B1	B2	B1	B2	

M1 = CM9, M2 = 1R6F, B1 =Dunhill, B2 = Rothmans

- Rotary machine smoking: one test result is defined as the mean yield obtained from smoking 20 test pieces in a single smoking machine run per day, therefore it is the result of 20 ports, smoking one (1) test piece per port.
    - Perform three smoke runs per sample type in one day following the smoke plan provided in Table 6 resulting in three (3) test results per sample type.

Table 6: Smoking Plan for 20-port Rotary Machine (20 test pieces per pad)

Day	Run number					
	1	2	3	4	5	6
1	M1	B1	M1	B1	B1	M1
2	M2	B2	M2	B2	B2	M2

M1 = CM9, M2 = 1R6F, B1 =Dunhill, B2 = Rothmans

8. Analysis

Note: Since the purpose of this initial study is to verify the feasibility of modifications to ISO 10315 to determine nicotine content in VLN smoke condensates and traditional nicotine content in smoke condensates, it is imperative that participants follow the ISO method specified below and the modifications detailed in this protocol Appendix 1:

8.1. Methods:

The following ISO method will be used for the determination of nicotine:

- ISO 10315, Cigarettes – Determination of nicotine in smoke condensates – Gas-chromatographic method, see appendix 1 for required modifications

8.2. Sample Analysis:

Follow the sample run order specified in the data reporting spreadsheet. This is to limit the chance of carryover due to the extremely wide range of nicotine in the test samples.

8.3. Data Reporting:

Email the completed data reporting spreadsheet to Anthony Brown, Hiromoto Yamazaki, Thomas Schmidt

The analytical variables to be reported for statistical analysis are:

- Mean puff number per test piece or cigarette
- Mean TPM per test piece or cigarette (mg/cig)
- Mean nicotine per test piece or cigarette (mg/cig)

The ancillary variables are:

- Date of test
- Type of smoking machine used
- Temperature (C°) and relative humidity (RH%) of conditioning enclosure
- Temperature (C°) and relative humidity (RH%) of smoking environment
- Atmospheric pressure in the laboratory during smoking
- Ambient air flow in the smoking machine before smoking (mm/sec)

Additionally:

- Follow the modification to ISO 10315 (see clause 6.3 Appendix 1 of the protocol) to construct two linear regression equations.
- Data for nicotine shall be reported in mg/cig and be reported to three decimal places.

- To evaluate the difference in linear regression weighting functions, laboratories shall report two sets of nicotine data using two linear regression models with different weightings.
    - Construct a linear regression equation with 1/x weighting, x being concentration. Use both the slope and intercept to quantitate test portions.
    - Construct a linear regression equation with $1/x^2$ weighting, x being concentration. Use both the slope and the intercept to quantitate test portions.
- If test portions are below limit of quantitation, report the estimated analytical result and note that the analytical result is an estimate beside the cell.
- Provide chromatograms for Cal 1, Cal 2, and Cal 7.
- Report linear regression equations (y = mx +b) with weighting.
- Report nicotine standard level concentrations and % accuracy for each standard level concentration calculated using the linear regression equation from expected concentration.

9. **Statistical Analysis**

A statistical analysis in general conformance with ISO 5725-2:1994 and ISO/TR 22971:2005 will be conducted using both calibration weightings and the results from the two weightings will be compared.

10. **Presentation of the Results**

The results will be presented for discussion at the Spring 2020 RAC meeting.

Appendix 1: Modifications required for ISO 10315: 2015

6. Calibration solutions

Prepare a series of at least five nicotine standard solutions whose concentrations cover the range expected to be found in the test portions (0.002 mg/ml to 1.0 mg/ml), provided in the example below for calibration levels 1 through 9. Prepare the calibration level 0, i.e. solvent blank with internal standard, and use as a quality control sample to gauge possible carry over.

Table 1. Summary of Modified Calibration Standards

Calibration Level	Volume of Nicotine Stock (ml)	Nicotine (mg/ml)[a]	Volume of Carvone Stock (ml)	Carvone (ISTD) (mg/ml)[b]
0	0	0,000	0,3	0,3
1	0,010	0,002	0,3	0,3
2	0,025	0,005	0,3	0,3
3	0,050	0,010	0,3	0,3
4	0,100	0,020	0,3	0,3
5	0,200	0,040	0,3	0,3
6	1,0	0,200	0,3	0,3
7	2,0	0,400	0,3	0,3
8	4,0	0,800	0,3	0,3
9	5,0	1,000	0,3	0,3

1. Calibration levels 0-3 are new levels not shown in the published ISO 10315 method.
2. [a] Based on 10,00 mg/ml nicotine stock solution concentration and volume of Nicotine Stock diluted to a final volume of 50 ml.
3. [b] Based on 50,0 mg/ml carvone stock solution concentration and a volume of carvone stock solution diluted to a final volume of 50 ml.

6.3 Calibration of the gas chromatograph

Note: Prime the GC system: To be performed after maintenance of the GC injection port. Use a sample extract, inject ~10 aliquots at an elevated GC oven temperature. This shall be performed before calibration of the gas chromatograph.

Note: For the calibration range listed in Table 1, a correlation coefficient of > 0.995 is achievable using a linear regression equation with 1/x weighting. If the laboratory is unable to achieve the calibration acceptance criteria stated below, start an investigation and take corrective action.

Use a linear regression model with 1/x weighting and $1/x^2$:

Calculate the ratio of the nicotine peak to the internal standard peak from the peak area (or height) data for each of the calibration solutions. Plot the graph of the nicotine concentrations in accordance with the area ratios and calculate a linear regression equation (concentration of nicotine according to the area ratios) from these data. The signal (peak area or height) obtained for all test portions must fall within the working range of the calibration curve.

1. Calculate a linear regression equation using 1/x weighting, x being concentration, from this data. Use both the slope and the intercept of the linear regression to process sample data.
2. Calculate a linear regression equation using $1/x^2$ weighting, x being concentration, from this data. Use both the slope and the intercept of the linear regression to process sample data.
3. If the correlation coefficient R2 is less than 0,990, then the calibration should be repeated. If calibration level 1 differs by more than 15 % and calibration levels 2- 9 differ by 10 % or more from the expected value (estimated by linear regression), the problem shall be investigated.

Note: Laboratories shall process the sample data using 1) a linear regression equation with 1/x weighting then 2) reprocess the sample data using a linear regression equation with 1/x2 weighting so that an evaluation of the difference between weighting functions may be performed.

Use Calibration level 0 to evaluate carry over and system performance: Inject two replicates of calibration level 0 before and after calibration of the gas chromatograph to evaluate possible carryover of nicotine. Inject two replicates of calibration level 0 after four (4) independent replicate analyses for each sample from linear smoking or three (3) independent replicate analysis from rotary smoking.

Inject an aliquot of the concentration standard level 2 (Cal 2) and a concentration standard level 7 (Cal 7) after every 20 sample determinations. If the calculated concentration for a solution differs by more than 10 % from the original value, start an investigation, take corrective action**,** repeat the full calibration procedure.

Follow the run order (Example: Samples generated from linear smoking) specified below.

Run Order	Sample Name
1	Cal Level 0
2	Cal Level 0
3	Cal Level 1
4	Cal Level 2
5	Cal Level 3
6	Cal Level 4
7	Cal Level 5
8	Cal Level 6
9	Cal Level 7
10	Cal Level 8
11	Cal Level 9
12	Cal Level 0
13	Cal Level 0
14	Smoke Run 1, B1 Rep-1
15	Smoke Run 1, B1 Rep-2
16	Smoke Run 1, B1 Rep-3
17	Smoke Run 1, B1 Rep-4
18	Cal Level 0
19	Cal Level 0

Run Order	Sample Name
20	Smoke Run 1, B2 Rep-1
21	Smoke Run 1, B2 Rep-2
22	Smoke Run 1, B2 Rep-3
23	Smoke Run 1, B2 Rep-4
24	Cal Level 0
25	Cal Level 0
26	Smoke Run 1, M1 Rep-1
27	Smoke Run 1, M1 Rep-2
28	Smoke Run 1, M1 Rep-3
29	Smoke Run 1, M1 Rep-4
30	Cal Level 0
31	Cal Level 0
32	Smoke Run 1, M2 Rep-1
33	Smoke Run 1, M2 Rep-2
34	Smoke Run 1, M2 Rep-3
35	Smoke Run 1, M2 Rep-4
36	Cal Level 0
37	Cal Level 0
38	Cal Level 2
39	Cal Level 7
40	Cal Level 0
41	Cal Level 0
42	Smoke Run 2, B1 Rep-1
43	Smoke Run 2, B1 Rep-2
44	Smoke Run 2, B1 Rep-3
45	Smoke Run 2, B1 Rep-4
46	Cal Level 0
47	Cal Level 0
48	Smoke Run 2, B2 Rep-1
49	Smoke Run 2, B2 Rep-2
50	Smoke Run 2, B2 Rep-3
51	Smoke Run 2, B2 Rep-4
52	Cal Level 0
53	Cal Level 0
54	Smoke Run 2, M1 Rep-1
55	Smoke Run 2, M1 Rep-2
56	Smoke Run 2, M1 Rep-3
57	Smoke Run 2, M1 Rep-4
58	Cal Level 0
59	Cal Level 0
60	Smoke Run 2, M2 Rep-1
61	Smoke Run 2, M2 Rep-2

Run Order	Sample Name
62	Smoke Run 2, M2 Rep-3
63	Smoke Run 2, M2 Rep-4
64	Cal Level 0
65	Cal Level 0
66	Cal Level 2
67	Cal Level 7
68	Cal Level 0
69	Cal Level 0
70	Smoke Run 3, B1 Rep-1
71	Smoke Run 3, B1 Rep-2
72	Smoke Run 3, B1 Rep-3
73	Smoke Run 3, B1 Rep-4
74	Cal Level 0
75	Cal Level 0
76	Smoke Run 3, B2 Rep-1
77	Smoke Run 3, B2 Rep-2
78	Smoke Run 3, B2 Rep-3
79	Smoke Run 3, B2 Rep-4
80	Cal Level 0
81	Cal Level 0
82	Smoke Run 3, M1 Rep-1
83	Smoke Run 3, M1 Rep-2
84	Smoke Run 3, M1 Rep-3
85	Smoke Run 3, M1 Rep-4
86	Cal Level 0
87	Cal Level 0
88	Smoke Run 3, M2 Rep-1
89	Smoke Run 3, M2 Rep-2
90	Smoke Run 3, M2 Rep-3
91	Smoke Run 3, M2 Rep-4
92	Cal Level 0
93	Cal Level 0
94	Cal Level 2
95	Cal Level 7

APPENDIX B: Raw Data

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
1	Linear	1R6F	1	1	10,82	7,9	0,7446	0,7554
1	Linear	1R6F	1	2	9,56	7,4	0,6784	0,6882
1	Linear	1R6F	1	3	9,02	7,3	0,6455	0,6548
1	Linear	1R6F	1	4	10,16	7,6	0,7045	0,7146
1	Linear	1R6F	2	1	10,00	7,6	0,6868	0,6967
1	Linear	1R6F	2	2	9,90	7,6	0,6817	0,6915
1	Linear	1R6F	2	3	9,78	7,6	0,6922	0,7022
1	Linear	1R6F	2	4	9,76	7,8	0,6854	0,6953
1	Linear	1R6F	3	1	9,80	7,9	0,7147	0,7250
1	Linear	1R6F	3	2	10,36	7,6	0,7437	0,7545
1	Linear	1R6F	3	3	10,20	7,4	0,7233	0,7337
1	Linear	1R6F	3	4	9,74	7,6	0,6935	0,7034
1	Rotary	1R6F	1	1	10,34	7,3	0,6956	0,7052
1	Rotary	1R6F	2	1	10,67	7,6	0,7269	0,7370
1	Rotary	1R6F	3	1	10,68	7,7	0,7393	0,7496
2	Rotary	1R6F	1	1	10,78	7,1	0,6930	0,7160
2	Rotary	1R6F	2	1	10,61	7,0	0,6750	0,6980
2	Rotary	1R6F	3	1	10,60	7,2	0,6870	0,7100
3	Linear	1R6F	1	1	11,10	7,7	0,7548	0,7731
3	Linear	1R6F	1	2	11,20	7,7	0,6858	0,7025
3	Linear	1R6F	1	3	10,80	7,6	0,6933	0,7101
3	Linear	1R6F	1	4	11,10	7,6	0,6979	0,7148
3	Linear	1R6F	2	1	10,30	7,5	0,7692	0,7879
3	Linear	1R6F	2	2	10,80	7,4	0,7101	0,7273
3	Linear	1R6F	2	3	10,50	7,5	0,6745	0,6908
3	Linear	1R6F	2	4	10,30	7,4	0,7319	0,7497
3	Linear	1R6F	3	1	11,50	7,9	0,8329	0,8532
3	Linear	1R6F	3	2	11,50	7,7	0,7163	0,7337
3	Linear	1R6F	3	3	11,20	7,3	0,6385	0,6539
3	Linear	1R6F	3	4	10,80	7,3	0,6374	0,6528
4	Rotary	1R6F	1	1	10,37	7,2	0,7144	0,7177
4	Rotary	1R6F	2	1	10,16	7,3	0,6973	0,7005
4	Rotary	1R6F	3	1	10,12	7,1	0,6975	0,7007
5	Linear	1R6F	1	1	10,24	7,2	0,7760	0,8000
5	Linear	1R6F	1	2	10,76	7,4	0,8320	0,8570
5	Linear	1R6F	1	3	10,92	7,7	0,8630	0,8900
5	Linear	1R6F	1	4	10,78	7,4	0,8240	0,8490
5	Linear	1R6F	2	1	10,22	7,6	0,7930	0,8180

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
5	Linear	1R6F	2	2	10,98	7,9	0,8540	0,8800
5	Linear	1R6F	2	3	10,66	7,8	0,8080	0,8330
5	Linear	1R6F	2	4	10,24	7,6	0,7970	0,8210
5	Linear	1R6F	3	1	10,52	7,6	0,7970	0,8210
5	Linear	1R6F	3	2	10,44	7,3	0,8090	0,8340
5	Linear	1R6F	3	3	9,54	7,5	0,7560	0,7790
5	Linear	1R6F	3	4	9,44	7,5	0,7420	0,7650
6	Linear	1R6F	1	1	10,38	7,5	0,7282	0,7089
6	Linear	1R6F	1	2	10,76	8,0	0,7957	0,7746
6	Linear	1R6F	1	3	10,52	7,8	0,7674	0,7471
6	Linear	1R6F	1	4	10,50	7,5	0,6890	0,6707
6	Linear	1R6F	2	1	10,94	7,6	0,6924	0,6741
6	Linear	1R6F	2	2	10,04	7,8	0,7682	0,7479
6	Linear	1R6F	2	3	11,30	8,1	0,8065	0,7851
6	Linear	1R6F	2	4	11,54	8,5	0,8407	0,8185
6	Linear	1R6F	3	1	10,70	7,9	0,7451	0,7254
6	Linear	1R6F	3	2	11,10	7,9	0,7998	0,7786
6	Linear	1R6F	3	3	11,02	8,1	0,7718	0,7514
6	Linear	1R6F	3	4	11,22	8,0	0,7965	0,7754
7	Linear	1R6F	1	1	10,30	8,0	0,7775	0,7827
7	Linear	1R6F	1	2	10,30	8,0	0,7543	0,7593
7	Linear	1R6F	1	3	10,70	8,3	0,8015	0,8068
7	Linear	1R6F	1	4	10,30	8,0	0,7689	0,7740
7	Linear	1R6F	2	1	10,30	8,3	0,7824	0,7877
7	Linear	1R6F	2	2	10,00	8,4	0,7616	0,7666
7	Linear	1R6F	2	3	10,00	8,3	0,7738	0,7790
7	Linear	1R6F	2	4	10,00	8,1	0,7730	0,7782
7	Linear	1R6F	3	1	10,10	7,7	0,7447	0,7497
7	Linear	1R6F	3	2	10,20	8,3	0,7767	0,7819
7	Linear	1R6F	3	3	10,20	8,1	0,7815	0,7867
7	Linear	1R6F	3	4	10,60	8,2	0,7997	0,8051
8	Rotary	1R6F	1	1	9,85	8,0	0,7433	0,7392
8	Rotary	1R6F	2	1	9,79	7,9	0,7413	0,7371
8	Rotary	1R6F	3	1	9,44	8,1	0,7309	0,7269
10	Linear	1R6F	1	1	11,40	7,6	0,7206	0,7594
10	Linear	1R6F	1	2	11,22	7,6	0,7016	0,7393
10	Linear	1R6F	1	3	10,64	7,2	0,6745	0,7106
10	Linear	1R6F	1	4	11,56	7,2	0,7028	0,7406
10	Linear	1R6F	2	1	9,22	7,2	0,6062	0,6385
10	Linear	1R6F	2	2	8,72	7,1	0,6083	0,6408

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
10	Linear	1R6F	2	3	8,58	6,8	0,5673	0,5974
10	Linear	1R6F	2	4	9,86	7,2	0,6529	0,6878
10	Linear	1R6F	3	1	10,50	7,4	0,7097	0,7479
10	Linear	1R6F	3	2	9,96	7,5	0,6994	0,7369
10	Linear	1R6F	3	3	8,90	7,0	0,6344	0,6683
10	Linear	1R6F	3	4	10,10	7,2	0,7010	0,7386
11	Rotary	1R6F	1	1	–	–	–	–
11	Rotary	1R6F	2	1	–	–	–	–
11	Rotary	1R6F	3	1	–	–	–	–
12	Linear	1R6F	1	1	9,46	7,0	0,6466	0,6547
12	Linear	1R6F	1	2	9,16	6,8	0,6457	0,6538
12	Linear	1R6F	1	3	9,10	7,0	0,6409	0,6489
12	Linear	1R6F	1	4	10,16	7,1	0,6757	0,6842
12	Linear	1R6F	2	1	9,38	6,9	0,6537	0,6619
12	Linear	1R6F	2	2	9,44	6,9	0,6480	0,6561
12	Linear	1R6F	2	3	9,10	6,9	0,6614	0,6697
12	Linear	1R6F	2	4	8,98	6,9	0,6677	0,6761
12	Linear	1R6F	3	1	8,62	6,4	0,6052	0,6128
12	Linear	1R6F	3	2	8,78	7,1	0,6406	0,6487
12	Linear	1R6F	3	3	8,92	6,7	0,6390	0,6470
12	Linear	1R6F	3	4	9,02	7,1	0,6698	0,6783
13	Linear	1R6F	1	1	10,18	7,4	0,6959	0,7618
13	Linear	1R6F	1	2	9,96	7,6	0,6832	0,7479
13	Linear	1R6F	1	3	9,20	7,5	0,6509	0,7125
13	Linear	1R6F	1	4	10,00	8,0	0,6963	0,7623
13	Linear	1R6F	2	1	9,72	7,9	0,6917	0,7289
13	Linear	1R6F	2	2	9,32	7,3	0,6572	0,6925
13	Linear	1R6F	2	3	9,98	7,7	0,7076	0,7456
13	Linear	1R6F	2	4	10,02	7,6	0,6890	0,7260
13	Linear	1R6F	3	1	9,92	7,6	0,6858	0,7226
13	Linear	1R6F	3	2	10,54	8,0	0,7242	0,7642
13	Linear	1R6F	3	3	9,90	7,6	0,6778	0,7143
13	Linear	1R6F	3	4	10,16	7,7	0,7101	0,7483
14	Linear	1R6F	1	1	9,92	8,0	0,7260	0,7240
14	Linear	1R6F	1	2	9,96	7,5	0,7070	0,7050
14	Linear	1R6F	1	3	10,08	7,7	0,7360	0,7340
14	Linear	1R6F	1	4	9,54	7,6	0,6720	0,6700
14	Linear	1R6F	2	1	9,78	7,6	0,6920	0,6900
14	Linear	1R6F	2	2	9,76	7,6	0,7130	0,7110
14	Linear	1R6F	2	3	9,84	7,8	0,7020	0,7000

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
14	Linear	1R6F	2	4	10,24	7,7	0,7470	0,7450
14	Linear	1R6F	3	1	9,16	7,2	0,6500	0,6480
14	Linear	1R6F	3	2	9,92	7,8	0,7060	0,7040
14	Linear	1R6F	3	3	9,78	7,7	0,7100	0,7080
14	Linear	1R6F	3	4	10,30	7,8	0,7340	0,7320
1	Linear	CM9	1	1	17,76	8,0	1,5031	1,5255
1	Linear	CM9	1	2	17,66	7,8	1,4761	1,4981
1	Linear	CM9	1	3	16,82	7,4	1,4094	1,4303
1	Linear	CM9	1	4	17,16	7,4	1,4440	1,4655
1	Linear	CM9	2	1	17,16	7,3	1,6248	1,6490
1	Linear	CM9	2	2	16,90	7,7	1,4544	1,4760
1	Linear	CM9	2	3	17,82	7,7	1,5058	1,5282
1	Linear	CM9	2	4	17,40	7,7	1,4783	1,5003
1	Linear	CM9	3	1	17,00	7,8	1,4338	1,4552
1	Linear	CM9	3	2	17,76	7,8	1,5041	1,5266
1	Linear	CM9	3	3	16,86	7,9	1,4621	1,4839
1	Linear	CM9	3	4	17,22	7,7	1,5152	1,5378
1	Rotary	CM9	1	1	17,05	7,3	1,4475	1,4692
1	Rotary	CM9	2	1	17,31	7,5	1,4730	1,4951
1	Rotary	CM9	3	1	16,58	7,4	1,4329	1,4544
2	Rotary	CM9	1	1	16,91	7,3	1,3760	1,4230
2	Rotary	CM9	2	1	16,69	7,2	1,3750	1,4220
2	Rotary	CM9	3	1	16,72	7,2	1,3950	1,4420
3	Linear	CM9	1	1	18,50	7,9	1,6023	1,6419
3	Linear	CM9	1	2	17,90	7,7	1,5896	1,6289
3	Linear	CM9	1	3	17,30	7,6	1,5874	1,6266
3	Linear	CM9	1	4	17,50	7,7	1,6117	1,6515
3	Linear	CM9	2	1	18,00	7,7	1,6391	1,6796
3	Linear	CM9	2	2	17,00	7,9	1,5750	1,6139
3	Linear	CM9	2	3	17,00	7,6	1,4777	1,5142
3	Linear	CM9	2	4	17,10	7,5	1,5633	1,6019
3	Linear	CM9	3	1	19,10	7,8	1,8215	1,8666
3	Linear	CM9	3	2	18,50	7,7	1,6539	1,6948
3	Linear	CM9	3	3	16,80	7,4	1,4732	1,5095
3	Linear	CM9	3	4	17,10	7,6	1,5137	1,5510
4	Rotary	CM9	1	1	17,63	7,5	1,4731	1,4813
4	Rotary	CM9	2	1	17,57	7,5	1,5199	1,5270
4	Rotary	CM9	3	1	17,53	7,5	1,5233	1,5304
5	Linear	CM9	1	1	18,52	7,9	1,6860	1,7390
5	Linear	CM9	1	2	17,36	7,7	1,6590	1,7120

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
5	Linear	CM9	1	3	17,50	8,0	1,6510	1,7030
5	Linear	CM9	1	4	16,68	8,1	1,6010	1,6510
5	Linear	CM9	2	1	17,16	7,8	1,7010	1,7550
5	Linear	CM9	2	2	17,02	8,1	1,6240	1,6750
5	Linear	CM9	2	3	16,42	8,0	1,6450	1,6960
5	Linear	CM9	2	4	16,74	7,2	1,4240	1,4680
5	Linear	CM9	3	1	16,42	7,6	1,5930	1,6430
5	Linear	CM9	3	2	17,30	7,8	1,6220	1,6730
5	Linear	CM9	3	3	16,52	7,8	1,5950	1,6450
5	Linear	CM9	3	4	15,36	7,3	1,4350	1,4800
6	Linear	CM9	1	1	18,42	7,8	1,5620	1,5206
6	Linear	CM9	1	2	17,78	7,7	1,5278	1,4873
6	Linear	CM9	1	3	17,90	8,0	1,5810	1,5392
6	Linear	CM9	1	4	19,08	8,3	1,6511	1,6074
6	Linear	CM9	2	1	18,40	7,9	1,6269	1,5838
6	Linear	CM9	2	2	18,02	8,2	1,6527	1,6089
6	Linear	CM9	2	3	18,44	7,8	1,5885	1,5464
6	Linear	CM9	2	4	17,28	7,6	1,5354	1,4947
6	Linear	CM9	3	1	19,06	8,1	1,4923	1,4528
6	Linear	CM9	3	2	18,34	7,8	1,5263	1,4859
6	Linear	CM9	3	3	17,54	7,9	1,4842	1,4448
6	Linear	CM9	3	4	18,26	8,1	1,5790	1,5372
7	Linear	CM9	1	1	17,40	7,9	1,5625	1,5733
7	Linear	CM9	1	2	17,80	8,1	1,6026	1,6136
7	Linear	CM9	1	3	18,50	8,0	1,6342	1,6454
7	Linear	CM9	1	4	18,60	8,6	1,6987	1,7104
7	Linear	CM9	2	1	17,80	8,5	1,6194	1,6306
7	Linear	CM9	2	2	16,70	8,3	1,5425	1,5531
7	Linear	CM9	2	3	15,70	8,2	1,4507	1,4606
7	Linear	CM9	2	4	18,90	8,2	1,6735	1,6851
7	Linear	CM9	3	1	18,50	8,4	1,6429	1,6542
7	Linear	CM9	3	2	17,90	8,2	1,5671	1,5779
7	Linear	CM9	3	3	18,80	9,0	1,6778	1,6894
7	Linear	CM9	3	4	18,10	8,6	1,6259	1,6370
8	Rotary	CM9	1	1	16,30	8,0	1,6274	1,6184
8	Rotary	CM9	2	1	16,25	7,8	1,6180	1,6090
8	Rotary	CM9	3	1	16,32	8,1	1,6074	1,5986
10	Linear	CM9	1	1	18,26	7,8	1,4526	1,5327
10	Linear	CM9	1	2	18,02	7,8	1,4266	1,5052
10	Linear	CM9	1	3	18,08	7,4	1,3992	1,4763

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
10	Linear	CM9	1	4	17,22	7,5	1,3941	1,4710
10	Linear	CM9	2	1	15,60	7,6	1,3382	1,4119
10	Linear	CM9	2	2	16,84	7,3	1,3787	1,4547
10	Linear	CM9	2	3	16,32	7,2	1,3198	1,3925
10	Linear	CM9	2	4	–	–	–	–
10	Linear	CM9	3	1	16,86	7,4	1,4271	1,5058
10	Linear	CM9	3	2	16,14	7,4	1,4234	1,5018
10	Linear	CM9	3	3	16,28	7,2	1,3728	1,4484
10	Linear	CM9	3	4	16,98	7,5	1,4607	1,5413
11	Rotary	CM9	1	1	–	–	–	–
11	Rotary	CM9	2	1	–	–	–	–
11	Rotary	CM9	3	1	–	–	–	–
12	Linear	CM9	1	1	16,54	7,4	1,3742	1,3921
12	Linear	CM9	1	2	15,88	7,3	1,3459	1,3634
12	Linear	CM9	1	3	15,46	7,4	1,3074	1,3243
12	Linear	CM9	1	4	16,68	7,3	1,3558	1,3734
12	Linear	CM9	2	1	16,44	7,5	1,3690	1,3864
12	Linear	CM9	2	2	15,86	7,4	1,3380	1,3553
12	Linear	CM9	2	3	15,36	7,0	1,2722	1,2887
12	Linear	CM9	2	4	16,64	7,2	1,4159	1,4344
12	Linear	CM9	3	1	16,18	7,3	1,3626	1,3803
12	Linear	CM9	3	2	16,58	7,5	1,4162	1,4346
12	Linear	CM9	3	3	16,88	7,2	1,4251	1,4437
12	Linear	CM9	3	4	17,50	7,5	1,4584	1,4774
13	Linear	CM9	1	1	16,38	7,5	1,4267	1,5633
13	Linear	CM9	1	2	17,10	7,4	1,4321	1,5692
13	Linear	CM9	1	3	16,04	7,8	1,3968	1,5306
13	Linear	CM9	1	4	16,16	7,7	1,4109	1,5460
13	Linear	CM9	2	1	16,58	7,9	1,3768	1,4502
13	Linear	CM9	2	2	16,50	7,8	1,4779	1,5567
13	Linear	CM9	2	3	15,86	8,0	1,4433	1,5202
13	Linear	CM9	2	4	16,68	8,0	1,4394	1,5161
13	Linear	CM9	3	1	15,80	7,8	1,4148	1,4902
13	Linear	CM9	3	2	16,48	7,8	1,4441	1,5264
13	Linear	CM9	3	3	17,36	8,0	1,4772	1,5560
13	Linear	CM9	3	4	16,46	7,7	1,4253	1,5013
14	Linear	CM9	1	1	17,48	8,0	1,5470	1,5430
14	Linear	CM9	1	2	17,18	7,9	1,4780	1,4750
14	Linear	CM9	1	3	17,00	8,0	1,4860	1,4820
14	Linear	CM9	1	4	18,12	7,7	1,5260	1,5220

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
14	Linear	CM9	2	1	18,12	7,8	1,5630	1,5590
14	Linear	CM9	2	2	16,32	7,8	1,4430	1,4400
14	Linear	CM9	2	3	16,68	7,7	1,4480	1,4450
14	Linear	CM9	2	4	17,38	7,8	1,5160	1,5120
14	Linear	CM9	3	1	15,38	7,8	1,3510	1,3480
14	Linear	CM9	3	2	16,30	7,7	1,4270	1,4240
14	Linear	CM9	3	3	16,78	7,8	1,4830	1,4800
14	Linear	CM9	3	4	16,66	7,6	1,4690	1,4660
1	Linear	Dunhill	1	1	0,12	6,0	0,0200	0,0196
1	Linear	Dunhill	1	2	0,26	6,2	0,0251	0,0249
1	Linear	Dunhill	1	3	0,14	6,0	0,0195	0,0192
1	Linear	Dunhill	1	4	0,28	6,0	0,0246	0,0243
1	Linear	Dunhill	2	1	0,26	6,0	0,0203	0,0200
1	Linear	Dunhill	2	2	0,28	6,0	0,0230	0,0227
1	Linear	Dunhill	2	3	0,22	5,8	0,0203	0,0200
1	Linear	Dunhill	2	4	0,24	6,0	0,0233	0,0230
1	Linear	Dunhill	3	1	0,30	6,0	0,0212	0,0209
1	Linear	Dunhill	3	2	0,32	6,0	0,0252	0,0250
1	Linear	Dunhill	3	3	0,24	6,1	0,0233	0,0231
1	Linear	Dunhill	3	4	0,24	5,8	0,0231	0,0228
1	Rotary	Dunhill	1	1	0,32	5,9	0,0100	0,0100
1	Rotary	Dunhill	2	1	0,32	6,0	0,0200	0,0200
1	Rotary	Dunhill	3	1	0,30	5,9	0,0200	0,0200
2	Rotary	Dunhill	1	1	0,33	5,8	0,0280	0,0280
2	Rotary	Dunhill	2	1	0,32	5,4	0,0260	0,0260
2	Rotary	Dunhill	3	1	0,37	5,6	0,0310	0,0310
3	Linear	Dunhill	1	1	0,18	6,2	0,0411	0,0415
3	Linear	Dunhill	1	2	0,18	6,0	0,0374	0,0378
3	Linear	Dunhill	1	3	0,16	6,0	0,0367	0,0371
3	Linear	Dunhill	1	4	0,22	5,7	0,0356	0,0360
3	Linear	Dunhill	2	1	0,34	6,2	0,0278	0,0279
3	Linear	Dunhill	2	2	0,34	6,3	0,0268	0,0269
3	Linear	Dunhill	2	3	0,38	6,0	0,0289	0,0291
3	Linear	Dunhill	2	4	0,32	6,0	0,0244	0,0245
3	Linear	Dunhill	3	1	0,26	6,2	0,0343	0,0346
3	Linear	Dunhill	3	2	0,26	6,0	0,0372	0,0376
3	Linear	Dunhill	3	3	0,28	5,9	0,0328	0,0331
3	Linear	Dunhill	3	4	0,34	6,0	0,0298	0,0300
4	Rotary	Dunhill	1	1	0,28	5,9	0,0193	0,0193
4	Rotary	Dunhill	2	1	0,27	5,9	0,0194	0,0194

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
4	Rotary	Dunhill	3	1	0,23	5,8	0,0184	0,0184
5	Linear	Dunhill	1	1	0,24	6,0	0,0320	0,0320
5	Linear	Dunhill	1	2	0,20	6,0	0,0300	0,0300
5	Linear	Dunhill	1	3	0,24	6,2	0,0340	0,0340
5	Linear	Dunhill	1	4	0,26	6,2	0,0320	0,0310
5	Linear	Dunhill	2	1	0,34	6,2	0,0360	0,0360
5	Linear	Dunhill	2	2	0,36	6,0	0,0330	0,0330
5	Linear	Dunhill	2	3	0,30	6,0	0,0290	0,0290
5	Linear	Dunhill	2	4	0,28	6,0	0,0270	0,0260
5	Linear	Dunhill	3	1	0,32	5,9	0,0340	0,0340
5	Linear	Dunhill	3	2	0,28	5,9	0,0340	0,0340
5	Linear	Dunhill	3	3	0,14	5,9	0,0290	0,0290
5	Linear	Dunhill	3	4	0,20	6,0	0,0320	0,0320
6	Linear	Dunhill	1	1	0,12	6,0	0,0201	0,0196
6	Linear	Dunhill	1	2	0,16	6,8	0,0304	0,0296
6	Linear	Dunhill	1	3	0,14	6,4	0,0271	0,0264
6	Linear	Dunhill	1	4	0,10	6,4	0,0216	0,0210
6	Linear	Dunhill	2	1	0,12	6,2	0,0207	0,0201
6	Linear	Dunhill	2	2	0,14	6,4	0,0262	0,0255
6	Linear	Dunhill	2	3	0,12	6,4	0,0219	0,0213
6	Linear	Dunhill	2	4	0,14	6,6	0,0241	0,0235
6	Linear	Dunhill	3	1	0,22	6,2	0,0251	0,0245
6	Linear	Dunhill	3	2	0,22	6,8	0,0252	0,0246
6	Linear	Dunhill	3	3	0,20	6,2	0,0175	0,0171
6	Linear	Dunhill	3	4	0,28	6,4	0,0229	0,0223
7	Linear	Dunhill	1	1	0,10	6,4	0,0283	0,0283
7	Linear	Dunhill	1	2	0,30	6,4	0,0284	0,0283
7	Linear	Dunhill	1	3	0,10	6,8	0,0312	0,0312
7	Linear	Dunhill	1	4	0,20	6,7	0,0265	0,0264
7	Linear	Dunhill	2	1	0,20	6,1	0,0288	0,0287
7	Linear	Dunhill	2	2	0,10	6,2	0,0291	0,0290
7	Linear	Dunhill	2	3	0,20	6,2	0,0274	0,0273
7	Linear	Dunhill	2	4	0,20	6,6	0,0335	0,0335
7	Linear	Dunhill	3	1	0,10	6,4	0,0313	0,0312
7	Linear	Dunhill	3	2	0,10	6,2	0,0323	0,0322
7	Linear	Dunhill	3	3	0,00	6,4	0,0273	0,0272
7	Linear	Dunhill	3	4	0,20	7,0	0,0321	0,0320
8	Rotary	Dunhill	1	1	0,34	6,1	0,0273	0,0270
8	Rotary	Dunhill	2	1	0,33	6,0	0,0288	0,0285
8	Rotary	Dunhill	3	1	0,39	6,2	0,0339	0,0336

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
10	Linear	Dunhill	1	1	0,06	6,4	0,0251	0,0246
10	Linear	Dunhill	1	2	0,06	6,4	0,0206	0,0198
10	Linear	Dunhill	1	3	0,00	6,4	0,0163	0,0153
10	Linear	Dunhill	1	4	0,06	6,2	0,0208	0,0200
10	Linear	Dunhill	2	1	0,02	6,0	0,0216	0,0209
10	Linear	Dunhill	2	2	-0,04	6,2	0,0170	0,0161
10	Linear	Dunhill	2	3	0,02	6,0	0,0212	0,0205
10	Linear	Dunhill	2	4	0,10	5,8	0,0190	0,0182
10	Linear	Dunhill	3	1	0,14	6,0	0,0202	0,0194
10	Linear	Dunhill	3	2	0,12	6,2	0,0260	0,0255
10	Linear	Dunhill	3	3	0,06	6,0	0,0244	0,0239
10	Linear	Dunhill	3	4	0,10	6,2	0,0232	0,0226
11	Rotary	Dunhill	1	1	0,30	5,5	0,0230	0,0230
11	Rotary	Dunhill	2	1	0,29	5,7	0,0220	0,0220
11	Rotary	Dunhill	3	1	0,29	5,7	0,0220	0,0220
12	Linear	Dunhill	1	1	0,22	5,8	0,0248	0,0245
12	Linear	Dunhill	1	2	0,16	6,0	0,0247	0,0244
12	Linear	Dunhill	1	3	0,18	5,4	0,0237	0,0234
12	Linear	Dunhill	1	4	0,30	6,0	0,0309	0,0308
12	Linear	Dunhill	2	1	0,16	5,7	0,0288	0,0286
12	Linear	Dunhill	2	2	0,20	6,0	0,0237	0,0235
12	Linear	Dunhill	2	3	0,16	6,0	0,0284	0,0282
12	Linear	Dunhill	2	4	0,32	6,0	0,0301	0,0300
12	Linear	Dunhill	3	1	0,30	5,8	0,0251	0,0249
12	Linear	Dunhill	3	2	0,16	5,9	0,0238	0,0236
12	Linear	Dunhill	3	3	0,42	5,7	0,0300	0,0298
12	Linear	Dunhill	3	4	0,20	5,8	0,0237	0,0234
13	Linear	Dunhill	1	1	0,16	6,4	0,0156	0,0158
13	Linear	Dunhill	1	2	0,20	5,8	0,0219	0,0226
13	Linear	Dunhill	1	3	0,20	6,2	0,0226	0,0234
13	Linear	Dunhill	1	4	0,12	6,0	0,0159	0,0160
13	Linear	Dunhill	2	1	0,16	6,4	0,0219	0,0236
13	Linear	Dunhill	2	2	0,12	6,0	0,0163	0,0177
13	Linear	Dunhill	2	3	0,12	6,0	0,0230	0,0249
13	Linear	Dunhill	2	4	0,10	6,0	0,0174	0,0189
13	Linear	Dunhill	3	1	0,26	5,8	0,0217	0,0235
13	Linear	Dunhill	3	2	0,24	5,6	0,0132	0,0144
13	Linear	Dunhill	3	3	0,15	6,0	0,0188	0,0204
13	Linear	Dunhill	3	4	0,24	5,7	0,0163	0,0178
14	Linear	Dunhill	1	1	0,18	5,8	0,0240	0,0240

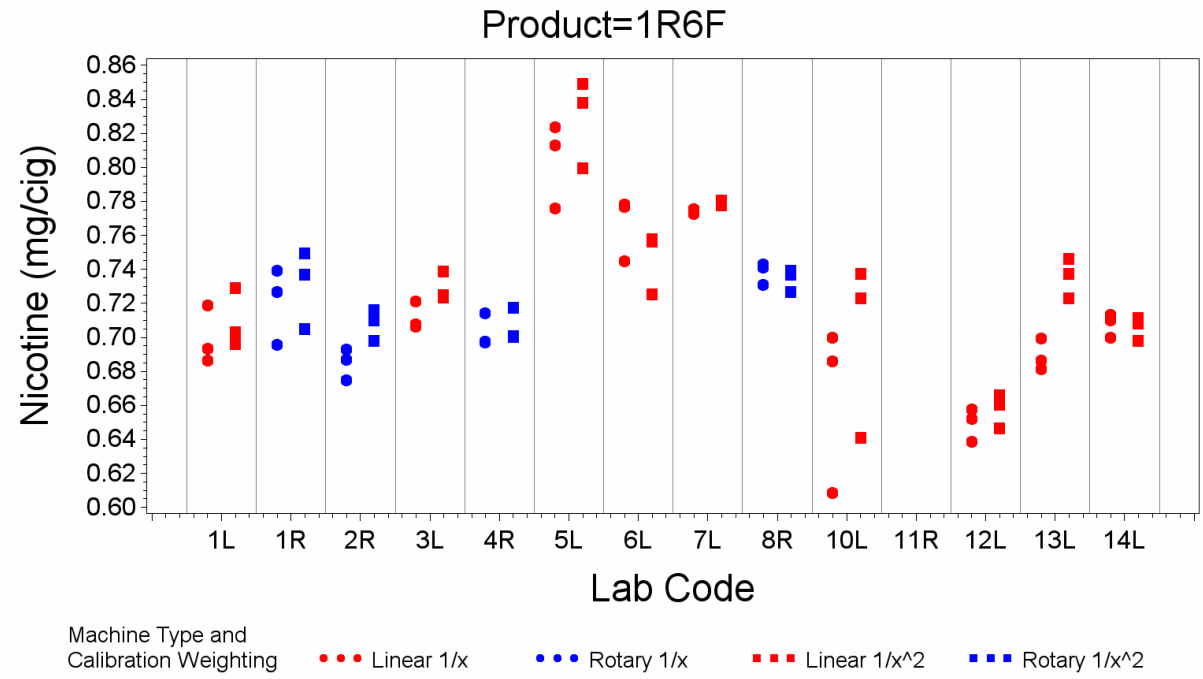
Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
14	Linear	Dunhill	1	2	0,16	6,1	0,0220	0,0220
14	Linear	Dunhill	1	3	0,24	6,0	0,0310	0,0310
14	Linear	Dunhill	1	4	0,28	5,8	0,0270	0,0270
14	Linear	Dunhill	2	1	0,28	6,1	0,0240	0,0240
14	Linear	Dunhill	2	2	0,20	6,0	0,0220	0,0220
14	Linear	Dunhill	2	3	0,24	6,0	0,0220	0,0220
14	Linear	Dunhill	2	4	0,22	6,2	0,0270	0,0270
14	Linear	Dunhill	3	1	0,30	6,5	0,0270	0,0270
14	Linear	Dunhill	3	2	0,22	6,0	0,0240	0,0240
14	Linear	Dunhill	3	3	0,22	6,4	0,0250	0,0250
14	Linear	Dunhill	3	4	0,22	6,0	0,0230	0,0230
1	Linear	Rothmans	1	1	0,62	5,3	0,0527	0,0529
1	Linear	Rothmans	1	2	0,70	5,6	0,0588	0,0591
1	Linear	Rothmans	1	3	0,72	6,0	0,0621	0,0625
1	Linear	Rothmans	1	4	0,70	6,0	0,0599	0,0602
1	Linear	Rothmans	2	1	0,86	5,6	0,0600	0,0603
1	Linear	Rothmans	2	2	0,94	5,6	0,0738	0,0743
1	Linear	Rothmans	2	3	0,64	6,0	0,0434	0,0434
1	Linear	Rothmans	2	4	0,68	6,0	0,0500	0,0502
1	Linear	Rothmans	3	1	0,76	5,8	0,0629	0,0632
1	Linear	Rothmans	3	2	0,86	6,0	0,0723	0,0728
1	Linear	Rothmans	3	3	0,70	5,6	0,0577	0,0580
1	Linear	Rothmans	3	4	0,66	5,8	0,0536	0,0538
1	Rotary	Rothmans	1	1	0,69	5,4	0,0515	0,0518
1	Rotary	Rothmans	2	1	0,69	5,4	0,0501	0,0505
1	Rotary	Rothmans	3	1	0,60	5,5	0,0424	0,0427
2	Rotary	Rothmans	1	1	0,75	5,2	0,0580	0,0590
2	Rotary	Rothmans	2	1	0,67	5,2	0,0490	0,0490
2	Rotary	Rothmans	3	1	0,69	5,3	0,0570	0,0580
3	Linear	Rothmans	1	1	0,96	5,9	0,0657	0,0668
3	Linear	Rothmans	1	2	0,88	5,7	0,0450	0,0455
3	Linear	Rothmans	1	3	0,60	5,7	0,0424	0,0429
3	Linear	Rothmans	1	4	0,68	5,3	0,0464	0,0470
3	Linear	Rothmans	2	1	0,80	6,2	0,0684	0,0696
3	Linear	Rothmans	2	2	0,86	5,6	0,0669	0,0680
3	Linear	Rothmans	2	3	0,84	6,1	0,0617	0,0627
3	Linear	Rothmans	2	4	0,70	5,6	0,0612	0,0621
3	Linear	Rothmans	3	1	0,80	5,5	0,0846	0,0862
3	Linear	Rothmans	3	2	0,58	6,0	0,0760	0,0773
3	Linear	Rothmans	3	3	0,62	5,5	0,0599	0,0609

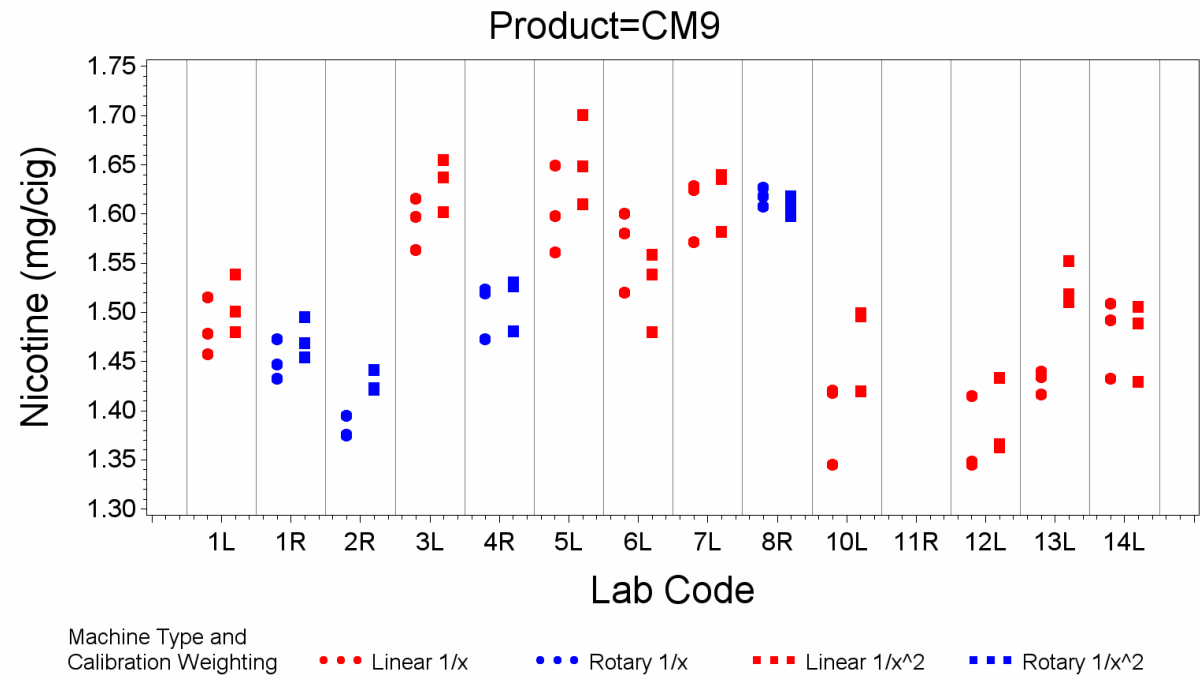
Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
3	Linear	Rothmans	3	4	0,52	6,0	0,0603	0,0613
4	Rotary	Rothmans	1	1	0,68	5,6	0,0566	0,0568
4	Rotary	Rothmans	2	1	0,63	5,6	0,0530	0,0533
4	Rotary	Rothmans	3	1	0,65	5,6	0,0568	0,0569
5	Linear	Rothmans	1	1	0,52	5,5	0,0530	0,0530
5	Linear	Rothmans	1	2	0,66	6,0	0,0660	0,0670
5	Linear	Rothmans	1	3	0,60	6,0	0,0730	0,0740
5	Linear	Rothmans	1	4	0,60	6,0	0,0670	0,0680
5	Linear	Rothmans	2	1	0,78	5,8	0,0710	0,0730
5	Linear	Rothmans	2	2	0,94	5,9	0,0820	0,0840
5	Linear	Rothmans	2	3	0,76	5,8	0,0690	0,0710
5	Linear	Rothmans	2	4	0,76	5,3	0,0650	0,0660
5	Linear	Rothmans	3	1	0,80	6,0	0,0790	0,0810
5	Linear	Rothmans	3	2	0,82	6,0	0,0750	0,0760
5	Linear	Rothmans	3	3	0,70	6,1	0,0750	0,0770
5	Linear	Rothmans	3	4	0,70	5,8	0,0730	0,0740
6	Linear	Rothmans	1	1	0,68	5,9	0,0636	0,0620
6	Linear	Rothmans	1	2	0,66	6,4	0,0660	0,0643
6	Linear	Rothmans	1	3	0,66	6,4	0,0665	0,0647
6	Linear	Rothmans	1	4	0,52	6,2	0,0569	0,0554
6	Linear	Rothmans	2	1	0,60	6,0	0,0580	0,0565
6	Linear	Rothmans	2	2	0,72	6,0	0,0613	0,0596
6	Linear	Rothmans	2	3	0,46	6,0	0,0530	0,0516
6	Linear	Rothmans	2	4	0,68	6,0	0,0681	0,0663
6	Linear	Rothmans	3	1	0,70	6,0	0,0656	0,0639
6	Linear	Rothmans	3	2	0,66	6,4	0,0618	0,0602
6	Linear	Rothmans	3	3	0,62	6,0	0,0565	0,0550
6	Linear	Rothmans	3	4	0,78	6,6	0,0697	0,0678
7	Linear	Rothmans	1	1	0,60	6,2	0,0675	0,0677
7	Linear	Rothmans	1	2	0,70	6,4	0,0704	0,0707
7	Linear	Rothmans	1	3	0,50	6,0	0,0592	0,0593
7	Linear	Rothmans	1	4	0,70	6,4	0,0743	0,0745
7	Linear	Rothmans	2	1	0,60	5,9	0,0654	0,0656
7	Linear	Rothmans	2	2	0,60	6,0	0,0632	0,0633
7	Linear	Rothmans	2	3	0,60	5,9	0,0672	0,0673
7	Linear	Rothmans	2	4	0,50	5,8	0,0693	0,0695
7	Linear	Rothmans	3	1	0,40	6,0	0,0563	0,0564
7	Linear	Rothmans	3	2	0,40	6,0	0,0588	0,0590
7	Linear	Rothmans	3	3	0,60	6,5	0,0748	0,0750
7	Linear	Rothmans	3	4	0,60	6,4	0,0687	0,0689

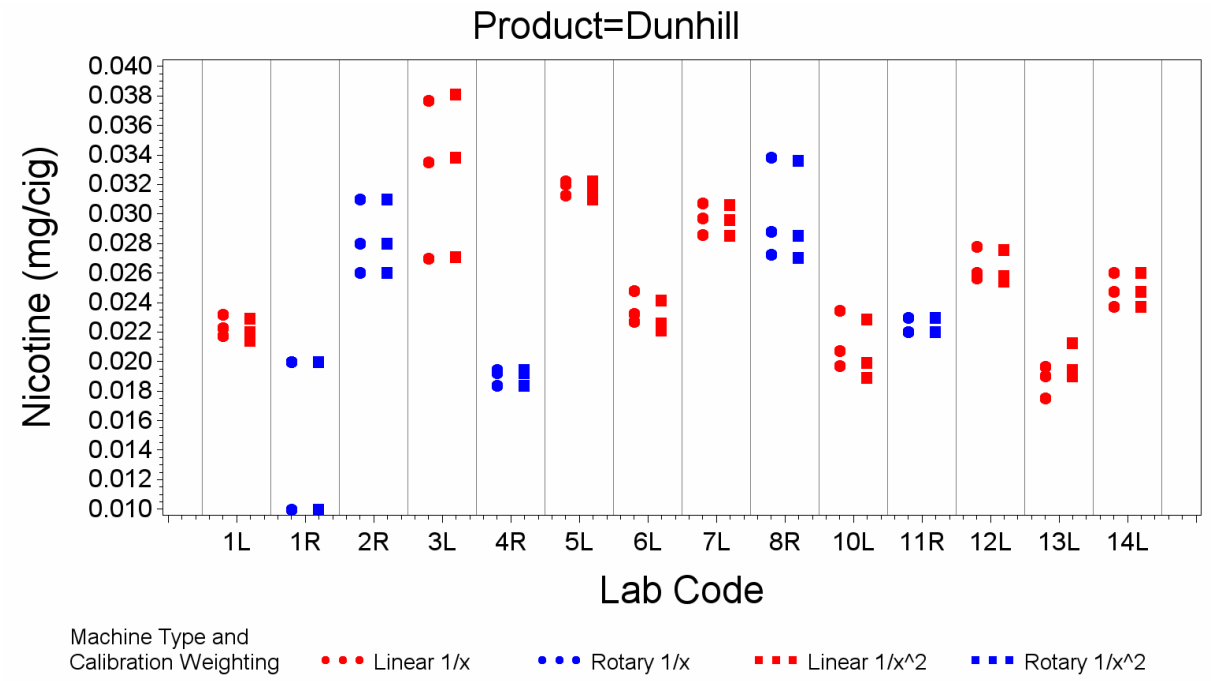
Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
8	Rotary	Rothmans	1	1	0,78	5,8	0,0604	0,0600
8	Rotary	Rothmans	2	1	0,75	6,0	0,0637	0,0633
8	Rotary	Rothmans	3	1	0,75	6,0	0,0628	0,0624
10	Linear	Rothmans	1	1	0,38	6,6	0,0367	0,0368
10	Linear	Rothmans	1	2	0,62	6,0	0,0552	0,0564
10	Linear	Rothmans	1	3	0,44	5,6	0,0418	0,0423
10	Linear	Rothmans	1	4	0,62	6,2	0,0535	0,0546
10	Linear	Rothmans	2	1	0,66	6,0	0,0613	0,0628
10	Linear	Rothmans	2	2	0,58	6,0	0,0565	0,0578
10	Linear	Rothmans	2	3	0,70	5,6	0,0537	0,0548
10	Linear	Rothmans	2	4	0,54	5,7	0,0530	0,0540
10	Linear	Rothmans	3	1	0,62	5,8	0,0638	0,0655
10	Linear	Rothmans	3	2	0,68	5,6	0,0607	0,0622
10	Linear	Rothmans	3	3	0,50	5,3	0,0528	0,0539
10	Linear	Rothmans	3	4	0,62	5,6	0,0577	0,0590
11	Rotary	Rothmans	1	1	0,63	5,3	0,0620	0,0620
11	Rotary	Rothmans	2	1	0,65	5,4	0,0640	0,0630
11	Rotary	Rothmans	3	1	0,68	5,4	0,0650	0,0640
12	Linear	Rothmans	1	1	0,64	5,5	0,0642	0,0645
12	Linear	Rothmans	1	2	0,74	5,6	0,0650	0,0653
12	Linear	Rothmans	1	3	0,78	5,5	0,0629	0,0632
12	Linear	Rothmans	1	4	0,56	5,8	0,0551	0,0553
12	Linear	Rothmans	2	1	0,68	5,4	0,0625	0,0628
12	Linear	Rothmans	2	2	0,62	5,2	0,0642	0,0644
12	Linear	Rothmans	2	3	0,82	5,7	0,0634	0,0637
12	Linear	Rothmans	2	4	0,76	5,4	0,0545	0,0547
12	Linear	Rothmans	3	1	0,66	5,6	0,0618	0,0621
12	Linear	Rothmans	3	2	0,88	5,7	0,0646	0,0649
12	Linear	Rothmans	3	3	0,68	5,2	0,0577	0,0579
12	Linear	Rothmans	3	4	0,72	5,8	0,0696	0,0699
13	Linear	Rothmans	1	1	0,60	5,8	0,0505	0,0540
13	Linear	Rothmans	1	2	0,46	5,4	0,0435	0,0463
13	Linear	Rothmans	1	3	0,46	5,7	0,0448	0,0477
13	Linear	Rothmans	1	4	0,38	5,7	0,0396	0,0420
13	Linear	Rothmans	2	1	0,54	5,8	0,0572	0,0608
13	Linear	Rothmans	2	2	0,28	5,8	0,0448	0,0477
13	Linear	Rothmans	2	3	0,26	5,6	0,0407	0,0435
13	Linear	Rothmans	2	4	0,52	5,6	0,0517	0,0551
13	Linear	Rothmans	3	1	0,70	5,8	0,0533	0,0567
13	Linear	Rothmans	3	2	0,62	6,0	0,0466	0,0497

Lab	Type	Sample	Test Result	Port	TPM mg/cig	Puffs	Nicotine mg/cig	Nicotine mg/cig
							1/x	$1/x^2$
13	Linear	Rothmans	3	3	0,48	5,8	0,0397	0,0424
13	Linear	Rothmans	3	4	0,52	6,1	0,0600	0,0638
14	Linear	Rothmans	1	1	0,62	6,0	0,0570	0,0570
14	Linear	Rothmans	1	2	0,58	5,8	0,0530	0,0530
14	Linear	Rothmans	1	3	0,58	5,8	0,0560	0,0550
14	Linear	Rothmans	1	4	0,70	6,0	0,0600	0,0590
14	Linear	Rothmans	2	1	0,80	6,4	0,0570	0,0570
14	Linear	Rothmans	2	2	0,58	6,0	0,0490	0,0490
14	Linear	Rothmans	2	3	0,66	6,0	0,0600	0,0600
14	Linear	Rothmans	2	4	0,56	6,0	0,0480	0,0480
14	Linear	Rothmans	3	1	0,70	6,2	0,0600	0,0600
14	Linear	Rothmans	3	2	0,72	6,0	0,0620	0,0620
14	Linear	Rothmans	3	3	0,84	6,2	0,0660	0,0660
14	Linear	Rothmans	3	4	0,84	6,0	0,0680	0,0680

APPENDIX C: Raw Data Plots



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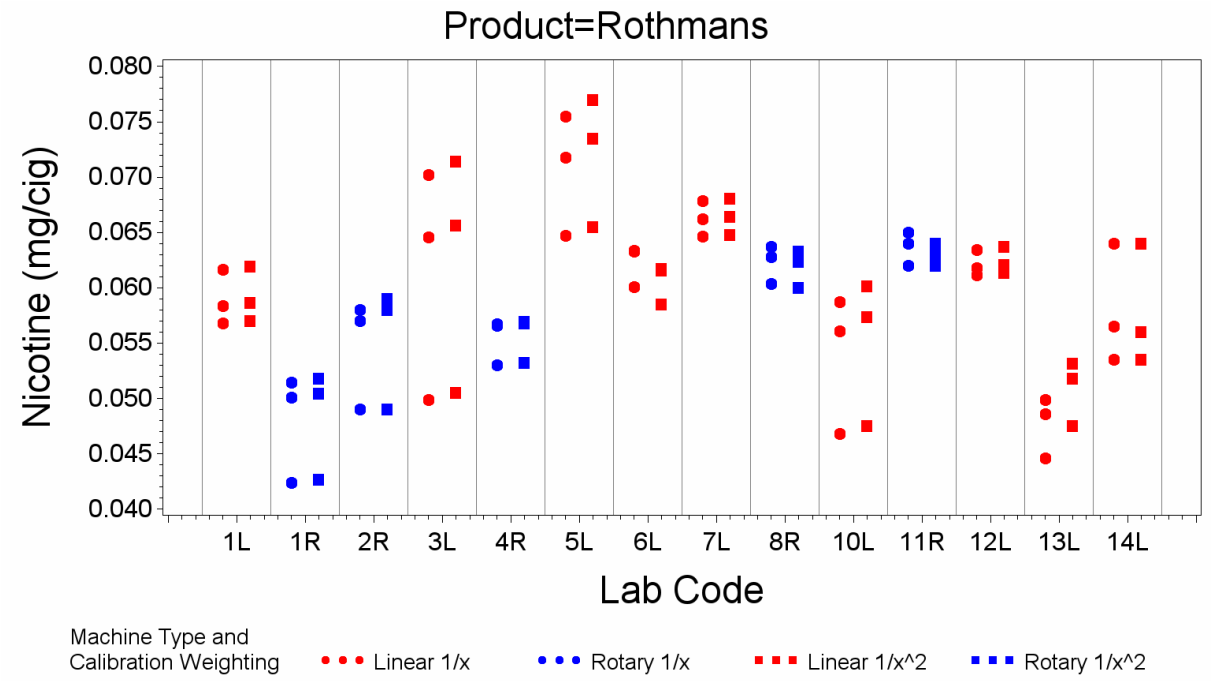
Nicotine (mg/cig)
0.040
0.038
0.036
0.034
0.032
0.030
0.028
0.026
0.024
0.022
0.020
0.018
0.016
0.014
0.012
0.010
1L
1R
2R
3L
4R
5L
6L
7L
8R
10L
11R
12L
13L
14L
Lab Code
Machine Type and Calibration Weighting
Linear 1/x
Rotary 1/x
Linear 1/x^2
Rotary 1/x^2


Product=Rothmans
Nicotine (mg/cig)
0.080
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
1L
1R
2R
3L
4R
5L
6L
7L
8R
10L
11R
12L
13L
14L
Lab Code
Machine Type and Calibration Weighting
Linear 1/x
Rotary 1/x
Linear 1/x^2
Rotary 1/x^2

APPENDIX D: Ancillary Data

Standard Levels, Signal to Noise Ratio (S/N), %RCR

Lab Code 1 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0,002	11,2	6,8	0,7
2	0,005	33,3	1,1	-0,4
3	0,010	NA	-1,8	-1,8
4	0,020	NA	-1,9	-1,2
5	0,040	NA	-2,4	-1,3
6	0,201	NA	-2,3	-0,9
7	0,402	NA	-0,9	0,6
8	0,803	NA	0,1	1,6
9	1,004	NA	0,8	2,4

Lab Code 1 %RCR Results (Rotary Smoke Day 1)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0,002	13,6	5,8	-0,4
2	0,005	38,4	0,8	-0,7
3	0,010	NA	-1,3	-1,4
4	0,020	NA	-2,3	-1,6
5	0,040	NA	-2,2	-1,1
6	0,201	NA	-2,4	-1,0
7	0,402	NA	-0,8	0,7
8	0,803	NA	0,2	1,8
9	1,004	NA	0,7	2,2

Lab Code 1 %RCR Results (Rotary Smoke Day 2)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0,002	12,5	5,4	-0,4
2	0,005	32,9	1,5	0,1
3	0,010	NA	-1,2	-1,2
4	0,020	NA	-2,5	-1,8
5	0,040	NA	-2,4	-1,3
6	0,201	NA	-2,2	-0,9
7	0,402	NA	-1,1	0,3
8	0,803	NA	0,3	1,7
9	1,004	NA	0,8	2,2

Lab Code 2 %RCR Results (Rotary Smoke Day 1)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	24	20.0	5.0
2	0.005	64	2.0	0.0
3	0.01	NA	-4.0	-4.0
4	0.02	NA	-2.0	-2.0
5	0.04	NA	-2.0	0.0
6	0.2	NA	0.0	3.0
7	0.4	NA	0.0	4.0
8	0.8	NA	1.0	4.0
9	1	NA	0.0	4.0

Lab Code 3 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	5	8	3
2	0.005	19	6	10
3	0.010	NA	9	10
4	0.020	NA	8	7
5	0.040	NA	10	8
6	0.199	NA	3	1
7	0.398	NA	1	1
8	0.796	NA	1	3
9	0.994	NA	4	1

Lab Code 4 %RCR Results (Rotary Smoke Day 1)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	161,437/3,1 (ISTD)	2.9	1.4
2	0.005	5,07/3,02 (nicotina); 160,795/2,77 (ISTD)	0.8	0.4
3	0.01	NA	0.2	0.2
4	0.02	NA	0.8	0.6
5	0.04	NA	1.4	1.1
6	0.2	NA	0.6	0.1
7	0.399	NA	0.2	0.2
8	0.798	NA	0.3	0.1
9	0.991	NA	0.6	1.0

Lab Code 5 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0,003	4,5	-13,1	-4,5
2	0,005	8,7	0,6	8,7
3	0,051	NA	5,8	3,9
4	0,102	NA	2,0	-0,6
5	0,204	NA	4,6	1,8
6	0,408	NA	2,5	-0,5
7	0,816	NA	1,1	-2,0
8	1,020	NA	-3,1	-6,4

Lab Code 6 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.001	20.97	-8.3	-5.5
2	0.010	125.4	-2.5	0.2
3	0.060	NA	-4.1	-1.4
4	0.201	NA	-2.0	0.7
5	0.805	NA	-0.1	2.5
6	1.006	NA	0.8	3.4

Lab Code 7 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	147	2.3	-0.5
2	0.005	346	2.7	2.0
3	0.010	NA	-0.3	-0.3
4	0.020	NA	-0.6	-0.3
5	0.040	NA	-1.2	-0.7
6	0.196	NA	-2.0	-1.4
7	0.393	NA	-1.9	-1.2
8	0.854	NA	6.8	7.5
9	0.984	NA	-1.6	-0.9

Lab Code 8 %RCR Results (Rotary Smoke Day 1)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	21	0.5	0.5
2	0.005	65	0.5	0.5
3	0.010	NA	0.5	-0.5
4	0.020	NA	0.0	-0.5
5	0.040	NA	-0.5	-1.8
6	0.201	NA	-0.2	-2.9
7	0.402	NA	-0.1	-2.9
8	0.804	NA	-0.1	-2.9
9	1.005	NA	-0.1	-2.7

Lab Code 8 %RCR Results (Rotary Smoke Day 2)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	34	0.5	0.5
2	0.005	77	0.5	0.5
3	0.010	NA	0.5	0.5
4	0.020	NA	-0.5	-1.5
5	0.040	NA	0.7	0.5
6	0.201	NA	0.2	-0.7
7	0.402	NA	-0.8	-2.9
8	0.804	NA	-0.6	-3.3
9	1.005	NA	-1.0	-3.9

Lab Code 10 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	NA	21.34	8.45
2	0.003	NA	5.49	-4.34
3	0.006	NA	-6.29	-8.9
4	0.012	NA	-7.69	-6.43
5	0.024	NA	-5.73	-2.38
6	0.061	NA	-4.42	0.18
7	0.122	NA	-3.92	1.11
8	0.244	NA	-1.42	3.95
9	0.488	NA	2.66	8.36

Lab Code 11 %RCR Results (Rotary Smoke Day 1 and Day 2)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	152	-11.5	-11.5
2	0.005	152	-8.7	-6.3
3	0.010	NA	-4.3	-3.1
4	0.019	NA	-3.2	-1.6
5	0.038	NA	-5.0	-2.3
6	0.192	NA	-4.0	-1.6
7	0.384	NA	-1.6	-0.7
8	0.768	NA	-0.3	-0.8
9	0.960	NA	0.7	0.0

Lab Code 12 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	5.1	10.5	5.2
2	0.005	12.3	1.4	0.0
3	0.010	NA	0.5	0.5
4	0.022	NA	0.5	1.2
5	0.043	NA	1.7	0.7
6	0.210	NA	0.7	0.6
7	0.408	NA	0.7	0.6
8	0.833	NA	0.9	2.2
9	1.056	NA	0.9	2.3

Lab Code 13 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.004	18.4	-5.5	-7.4
2	0.010	57.5	-13.7	-9.8
3	0.019	NA	-11.5	-5.3
4	0.058	NA	-10.8	-2.9
5	0.116	NA	-8.8	-0.3
6	0.217	NA	-4.9	4.1
7	0.360	NA	-2.0	7.4
8	0.721	NA	1.2	10.9
9	1.465	NA	0.0	9.6

Lab Code 14 %RCR Results (Linear Smoke)

Calibration Level	Nicotine (mg/ml)	Signal to Noise (S/N)	1/x % Relative Concentration Residual (%RCR)	$1/x^2$ % Relative Concentration Residual (%RCR)
0	0	NA	NA	NA
1	0.002	8.5	-2.2	-1.3
2	0.005	19.3	3.2	3.4
3	0.04	NA	-0.7	-0.9
4	0.2	NA	-0.9	-1.2
5	0.4	NA	0.7	0.5
6	0.8	NA	0.3	0.1
7	1	NA	-0.3	-0.5

Reported Observations for GC Inlet Conditions and Column

Lab	Injection Vol (µl)	Injection Mode	Split Ratio	Column
1	1	Split	20	HP-20M (25m, 320um, 0.3µm)
2	1	Split	20	DB ALC1 (30 m, 0,320 mm, 1,8 µm)
3	2	Split	20	DB-Wax (0.180mm x 0.30 µm)
4	1	Split	10	COL-ELITE-VAX (30 [m] x 530 [µm] x 1 [µm])
5	2	Split	50	DB-Wax (3.0 M X .18 mm X .3 µm df)
6	1	Split	5	DB-WAX (15m x 0.53mm x 1µm)
7	2	Split	20	DB-Wax (15 m X 0.025 mm X 0.25 µm)
8	2	Split	20	HP-INNOWAX (30m X 0.250mm, 0.25 µm)
10	1	Splitless	NA	6’ x 1/8” OD, stainless steel, 7 % Carbowax-20M, 2 % KOH, and 3 % OS-138 on Chromosorb 80/100 mesh)
11	1	Split	10	HP-5 (30 m × 0.25 mm ID)
12	1	Splitless	NA	DB-5 (30m)
13	1	Split	14	ZB-WAX (10m x 0.18 mm x 0.18 um)
14	1	Split	20	Stabilwax DB (15m x 0.32mm, 0.25µm film thickness) w/5-m deactivated guard column