CIG-2018C Proficiency Program Protocol

2018 Round 3

UNIVERSITY OF KENTUCKY
College of Agriculture, Food and Environment
Center for Tobacco Reference Products

Objective

This round of testing will include smoking the 1R6F reference cigarette using both the International Organization for Standardization (ISO) and the Health Canadian Intense (HCI) regimes and the determination of physical properties.

The mainstream smoke measured properties are:

- Nicotine-Free Dry Particulate Matter
- Nicotine
- Carbon Monoxide
- Water
- Total Particulate Matter
- Puff Count

Physical properties that will be measured are:

- Cigarette Resistance to Draw (pressure drop open)
- Cigarette Resistance to Draw (pressure drop closed)
- Filter Pressure Drop (fully encapsulated)
- Total Ventilation
- Filter Ventilation
- Tobacco Weight
- Cigarette Weight
- Air Permeability
- Firmness
- Circumference
- Cigarette Length
- Filter Plug Length
- Tipping Paper Length

Proficiency Study Timeframe

July 16, 2018: 09:00 AM EDT	*PT round Opens, Test Kits available for purchase*
August 8, 2018: 09:00 AM EDT	*Data submission portal Opens, First day of data submission*
October 10, 2018: 5:00 PM EDT	*Data submission portal Closes, Final day of data submission*
November 14, 2018	*Target date for issuance of Interim Report*
December 12, 2018	*Target date for issuance of Final Report, PT round Closes*

Eastern Daylight Time (EDT) (New York, NY time)

Test kits are available for purchase beginning on July 16, 2018. This round of testing for data submission will open on August 8, 2018 and close on October 10, 2018. The University of Kentucky, Center for Tobacco Reference Products (CTRP) data submission portal will be locked after the closing date and will no longer accept results. Results obtained after the closing date will not be included in the proficiency study report. The target date for issuance of the interim report is November 14, 2018. The participants are encouraged to review the interim report and provide feedback, i.e. comments, erroneous data entry, additional notes, etc., to the Proficiency Test Coordinator, Ms. Erin Pyrek, through the online feedback form available by clicking "Submit Comments" next to the interim report link located on the "My Proficiency Studies" tab of the CTRP website (ctrp.uky.edu). Feedback received will be considered and, if appropriate, incorporated in a final report which will be issued, tentatively, on December 12, 2018. The interim report and final report can be downloaded from the "My Proficiency Studies" tab located on the CTRP website by clicking the "Interim Report" or "Analysis Report" link, respectively.

References

Conditioning	ISO 3402:1999
TPM/ NFDPM	ISO 4387:2000
Carbon Monoxide	ISO 8454:2007
Nicotine	ISO 10315:2013
Water (GC)	ISO 10362-1:1999
Intense smoking regime, puff parameters	Health Canada Method T-115
ISO smoking regime, puff parameters	ISO 3308:2012, ISO 4387:2000
Resistance to Draw	ISO 6565:2015
Filter Pressure Drop	ISO 6565:2015
Total Ventilation	ISO 9512:2002
Filter Ventilation	ISO 9512:2002
Tobacco Weight	
Cigarette Weight	
Air Permeability	ISO 2965:2009
Firmness	
Circumference	ISO 2971:2013
Cigarette Length	
Filter Plug Length	
Tipping Paper Length	

Proficiency Test Material (1R6F Reference Cigarettes)

Proficiency Test Material for this round of proficiency sampling must be obtained from the CTRP by procuring the proficiency test kit. The materials will come with a test protocol and instructions to download the electronic reporting template in the form of a pre-formatted MS Excel file. It is not acceptable to use 1R6F reference cigarettes from your inventory. Using the materials provided will ensure that all participants are using cigarettes from a batch that is pre-characterized for the purposes of the Proficiency Test Scheme. Homogeneity of the Proficiency Test Material was determined by selecting 12 random samples and having them analyzed in at least triplicate. The testing was sub-contracted to a third-party laboratory meeting the quality requirements of the

proficiency testing scheme in accordance with ISO/IEC 17043. Test results confirm that the Proficiency Test Material is fit for proficiency testing.

General Guidance

Table 1 lists the smoking parameter and vent blocking specifications for each smoking regimen. The butt length for this testing is set at 35 mm or 1.38 inches.

It is important to note the need for participants to record any deviation from the standard methods in their report. Operating conditions considered optional reporting by the laboratory should also be recorded on their report. Any circumstances that arise during the analysis of these cigarettes which may influence either the precision or the bias of the result should be recorded in the report. Details of deviation from normal operations should be recorded in the "Notes" section of the Excel reporting template.

Table 1
Smoking parameter specifications

Smoking Regimen	*Puff Volume (mL)*	*Puff Interval (s)*	*Puff Duration (s)*	*Vent Blocking*
ISO	35± 0.3	60± 0.5	2 ± 0.02	0%
HCI	55 ± 0.5	30 ± 1	2 ± 0.02	100%

Note: Puff Interval is time in seconds from the start of one puff to the start of the next puff.[1]

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

Test Item Storage

The samples should be stored in plastic bags at 4°C prior to conditioning for the proficiency test.

Conditioning

Samples should be conditioned for a minimum of 48 hours, but no more than 10 days at 22 ± 1 °C and 60 ± 3% relative humidity before conducting each smoking and physical parameter test.

Replicates Required

Replicates must be obtained **under repeatability conditions** i.e. same instrument and same operator**.**

Smoking Parameter
Smoke 5 replicates for each smoking regime for both linear and rotary smoking machines. Laboratories should follow their routine smoking plan.

[1] 2012 CORESTA Collaborative Study for CORESTA Monitor #7 (CM7) for Determination of Test Piece, Weight, TPM, Water, Nicotine, NFDPM, Carbon Monoxide and Puff Count Obtained Under Mainstream ISO and HCI Smoking Regimes; May 2013

Physical Testing

For each physical parameter, 5 replicates of the mean of the measurements of 20 cigarettes should be recorded.

The analytes should be reported in units of mg/cig, on an as-is basis. Please report on as many analytes as you can. Note that only the mean values will be provided, if there are less than 5 reporting labs for that analyte.

Participants who order a Proficiency Test Kit should download the Excel reporting template which will be used to submit results for the proficiency testing. The Excel reporting template can be downloaded from the "My Proficiency Studies" tab located on the CTRP website (ctrp.uky.edu) after you have purchased a Proficiency Test Kit. If you order a linear and a rotary Proficiency Test Kit or multiple kits for each machine, you must download the Excel reporting template for each of the Proficiency Test Kits. Each Excel reporting template has a unique "Assigned Data Set ID" based on a customer's purchase. Please make sure that you enter the data into the correct Excel reporting template (linear or rotary). *Please note that there is no need to round results at any point in your calculations. Make sure to report results in the units indicated in the Excel reporting template.* The results should be submitted electronically through the CTRP website on the "My Proficiency Studies" tab. The participant will: (1) click the blue "Submit Proficiency Data" button for the correct reporting proficiency study; (2) browse their computer for the Excel reporting template for that proficiency study; (3) select the appropriate file; and (4) then click the "Load and Review Data" button. The participant will have the opportunity to review their data online before their final submission of data to the CTRP.

The study report will contain:

- Executive Summary
- Purpose of study
- Protocol
- Coded laboratory raw data
- Statistical summary and ***z***-score by laboratory (both graphical and numeric)

File Formatting Requirements for Data

To ensure clear and uninterrupted data processing among disparate computer systems, please use the Excel reporting template provided with the Proficiency Test Kit, which has been formatted for data entry. Please note that the downloadable Excel reporting template contains "locked" codes and a Proficiency Study ID (CIG-2018C) and an Assigned Data Set ID number specific to your test kit and this round of Proficiency Testing.

Below is a description of the file formatting, type, and expected contents of data files to be sent to the CTRP Proficiency Testing Program.

File Details

The data transport file should be formatted as an Excel file, specifically the XML-based format that Excel files are saved in by default. There should be no spaces in the filename. The Excel file extension should, by default, be *.xlsx.*

example: *linear_datasetid_3476.xlsx*

Proficiency Data

Please use the dropdown box in the top right section of the Excel reporting template to answer whether the lab has ISO Accreditation.

Machine Smoking Data

Please be sure to enter data for the specific smoking machine (linear or rotary) used in your analysis:

- *Smoking Machine Make (i.e., manufacturer)*
- *Smoking Machine Model*
- *Enter any notes on data collection (if necessary)*

ISO Data

Please be sure to enter measurements for the specific ISO Data:

- *ISO Data Test Date*
- *Linear Machines: Enter the number of ports used per replicate.*
  Rotary Machines: Enter the number of collections per replicate.
- *Linear Machines: Enter the number of cigarettes smoked per port.*
  Rotary Machines: Enter the number of cigarettes per collection.
- *Laboratory conditions (6 variables) for each of replicates*
- *Measurements for each of the 6 smoking parameters (Nicotine-Free Dry Particulate Matter, Nicotine, Carbon Monoxide, Water, TPM, and Puff Count) for each of the 5 replicates.*
- *Please use the dropdown menu to select the "instrument" used in testing for the* ***nicotine*** *and* ***water*** *smoking parameters.*
- *Please use the dropdown menu to select the "method" used in testing for the* ***nicotine*** *and* ***water*** *smoking parameters. If your method is not identified in the dropdown menu, please type your method in the box provided.*

	Nicotine	**Water**
Instrument	GC/MS or GC/FID	Karl Fischer apparatus or GC
Method 1	CRM#	Karl Fischer Method or GC method
Enter alternate method (text)		

For the instrument: GC/MS- Gas Chromatography/mass spectrometers, FID- flame ionization detector

For method: CRM: CORESTA Recommended Method; in-house method or others

- *Please type your internal standard for the **nicotine** and **water*** smoking parameters *in the box provided.*

	Nicotine	Water
Internal Standard (enter text)		

- If a participant does not have a measurement for a data field, please leave the Excel cell blank. When you upload your spreadsheet to the CTRP database, you will have the opportunity to review all your data on the web-based user interface. All cells that were left blank on the Excel reporting template, will appear as "< empty >" on the web-based user interface screen.

Intense Smoking Data

Please be sure to enter measurements for the specific Intense Smoking Data:

- *Intense Test Date*
- *Linear Machines: Enter the number of ports used per replicate.*
  Rotary Machines: Enter the number of collections per replicate.
- *Linear Machines: Enter the number of cigarettes smoked per port.*
  Rotary Machines: Enter the number of cigarettes per collection.
- *When smoking is done using the HCI smoking regime, please be sure to enter data for the Ventilation Blocking Method*
- *Laboratory conditions (6 variables) for each of replicates*
- *Measurements for each of the 6 smoking parameters (Nicotine-Free Dry Particulate Matter, Nicotine, Carbon Monoxide, Water, TPM, and Puff Count) for each of the 5 replicates.*
- If a participant does not have a measurement for a data field, please leave the Excel cell blank. When you upload your spreadsheet to the CTRP database, you will have the opportunity to review all your data on the web-based user interface. All cells that were left blank on the Excel reporting template, will appear as "< empty >" on the web-based user interface screen.

Physical Measurement Data

There are 5 data-entry rows (replicates 1 to 5) for each of the physical parameters. Determine the individual measurements (20 cigarettes for each replicate) and report the average. There are 13 physical parameters and associated standard deviations for each. These rows and cells should not be altered, as they are required for data import and transformation.

Please be sure to enter the following data for each of the 13 physical measurements:

- *Physical Measurements Machine Make (i.e., manufacturer)*
- *Physical Measurements Machine Model*
- *Physical Measurements Test Date*
- If a participant does not have a measurement for a data field, please leave the Excel cell blank. When you upload your spreadsheet to the CTRP database, you will have the opportunity to review all your data on the web-based user interface. All cells that were left

blank on the Excel reporting template, will appear as "< empty >" on the web-based user interface screen.

Please report the firmness measurements as an algorithmically adjusted firmness value pegged at 13.8% moisture.

File Data

Additional information/instructions are available on the home page of the CTRP website in the document section at How to Upload Proficiency Test Data .

Completed Files

When the Excel reporting template is completed and saved with the current date embedded in the file name, please submit the data file through the "My Proficiency Studies" tab located on the CTRP website following the instructions set forth above. The data will be stored anonymously, based on a randomly generated Assigned Data Set ID in a secured database for the study. All data will be treated in a confidential manner as set forth in the "Terms and Conditions for CTRP Proficiency Testing Programs," and agreed to by the participants.

Proficiency Test Coordinator

The Proficiency Test Coordinator is Erin Pyrek. She can be contacted at the address and phone number provided below:

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