Physical Test Methods Sub-Group

Technical Report

16th Round Robin Test for Multi-Capillary Pressure Drop Calibration Standards (2021-2023)

December 2023

Author:
Bernhard Eitzinger, delfortgroup AG, Austria

Table of Contents

1. Introduction and Background

The CORESTA Physical Test Methods (PTM) Sub-Group organizes regular round robin tests that are open to the member laboratories that have a calibration laboratory. The tests cover the calibration methods for pressure drop, ventilation and air permeability. The tests enable the participants to compare their capability to calibrate standards used in physical test instrumentation and each laboratory is also able to use the results in internal and external audit assessments.

This report covers the results of the 16th Round Robin Test on Pressure Drop (PD) Calibration Standards between January 2021 and October 2023. The test provides a baseline of PD instrument performance across the industry, since this standard type is used in the PD instrumentation of each supplier.

The current international standard is ISO 6565:2015 "Tobacco and tobacco products — Draw resistance of cigarettes and pressure drop of filter rods — Standard conditions and measurement". The pressure drop standards are glass rods of 120 mm length by approximately 8 mm diameter that contain 10 parallel capillaries along their length to create a pressure drop when an airflow is drawn through the standard. The diameter of the capillaries determines the pressure drop. These standards are calibrated under measured conditions of flow rate, pressure, temperature and humidity – all of which affect the measured pressure drop to a greater or lesser extent – and the result is then converted according to ISO 6565:2015 Annex A to the value that would have been observed had the standard been calibrated under industry-standard conditions of:

- Flow rate 17,5 $ml \cdot s^{-1}$ at the outlet to the standard
- Atmospheric pressure 1013,25 hPa
- Atmospheric temperature (22±2) °C
- Atmospheric humidity (60±5) %RH

All pressure drop values reported here include compensation to these conditions. This ascribed pressure drop is then transferred on calibration to an instrument in use so that, even if conditions are different, as is usually the case, the standard is observed to record its calibrated value. The use of pressure drop standards to transfer these defined conditions of flow rate and atmospheric conditions plays a significant part in standardising pressure drop measurements across the industry.

During the development of ISO 6565:2015 the precision of calibration of pressure drop transfer standards was determined between three suppliers, as presented in Table 1:

Table 1: ISO 6565:2015 - r and R Estimations for Calibration of PD Standards (mmWG)

Standard				
Nominal Value [mmWG]	200	400	600	800
Repeatability Standard Deviation (sr)	0,21	0,33	0,44	0,48
Reproducibility Standard Deviation (sR)	0,43	0,96	1,18	1,83

In the round robin test on PD calibration standards a single set of standards is circulated since the three instrumentation suppliers use the same physical test piece design and test pieces that are all supplied from a single source. The circulated standards have pressure drops of nominally 200 mmWG, 400 mmWG, 600 mmWG and 800 mmWG, approximately equivalent to 2 kPa, 4 kPa, 6 kPa and 8 kPa.

The five participating laboratories in the 16th Round Robin Test on Pressure Drop Calibration Standards are listed in Table 2.

Table 2: Participating Laboratories

Participating laboratories	Function	Accreditation
Cerulean Milton Keynes, United Kingdom	Calibration lab & instrumentation supplier	ISO 9001 & 17025
Cross Precision Measurement Winston-Salem, NC, USA	Calibration laboratory	ISO 17025
Körber Technologies Instruments SAS Saint Jean de Braye, France	Calibration lab & instrumentation supplier	ISO 9001 & 17025
Körber Technologies Instruments GmbH Hamburg, Germany	Calibration lab & instrumentation supplier	ISO 9001 & 17025
Zhengzhou Tobacco Research Inst. Zhengzhou, China	Calibration laboratory	

The laboratory identities are coded in the results presented below. The coding is the same as used in previous reports on the round robin tests for pressure drop calibration standards.

There have been different participants in the round robin tests since the publication of ISO 6565:2011 that first presented the improved calibration method used in this round robin test. The 8th test (2011) included only the three instrument suppliers; the Zhengzhou Tobacco Research Institute (China) joined from the 10th (2014) and Cross Precision Measurements (formerly JA King) (USA) joined from the 12th (2017). In the 14th test there was a mistake in the sample distribution such that the standards were recleaned by the originator before the final laboratory had made its measurements, so it was unable to participate. The 9th test was aborted due to an extended delay during shipping the standards between participants.

2. Experimental Protocol

The protocol involved:

- acclimatisation of the standards to laboratory conditions
- testing to the method detailed in ISO 6565:2015
- making three PD determinations under repeatability conditions for each standard on two separate days, i.e. six independent determinations.

After circulation, the standards were rechecked by the originator.

For the determination of repeatability and reproducibility this study followed ISO 5725-2. This study conforms to the principles described in ISO 17043 *Conformity assessment — General requirements for proficiency testing*; however, CORESTA is not an accredited proficiency testing provider and does not adhere to certain aspects of ISO 17043.

3. Results of the 16th Round Robin Test

3.1 Overall Results

The overall results of all the participants are given in Table 3 and are presented as a scatterplot of coefficient of variation (CoV) of laboratory means against the global mean PD of each test piece in Figure 1.

Table 3: 16th Round Robin Test – Overall Results

Nominal Value (mmWG)	Global mean (mmWG)	Std dev of lab means (mmWG)	CoV of lab means (%)	Range (mmWG)	Range (% of value)
200	199,6	0,31	0,15 %	0,7	0,33 %
400	392,0	1,01	0,26 %	2,4	0,62 %
600	647,7	1,86	0,29 %	4,5	0,70 %
800	837,1	3,03	0,36 %	7,8	0,94 %

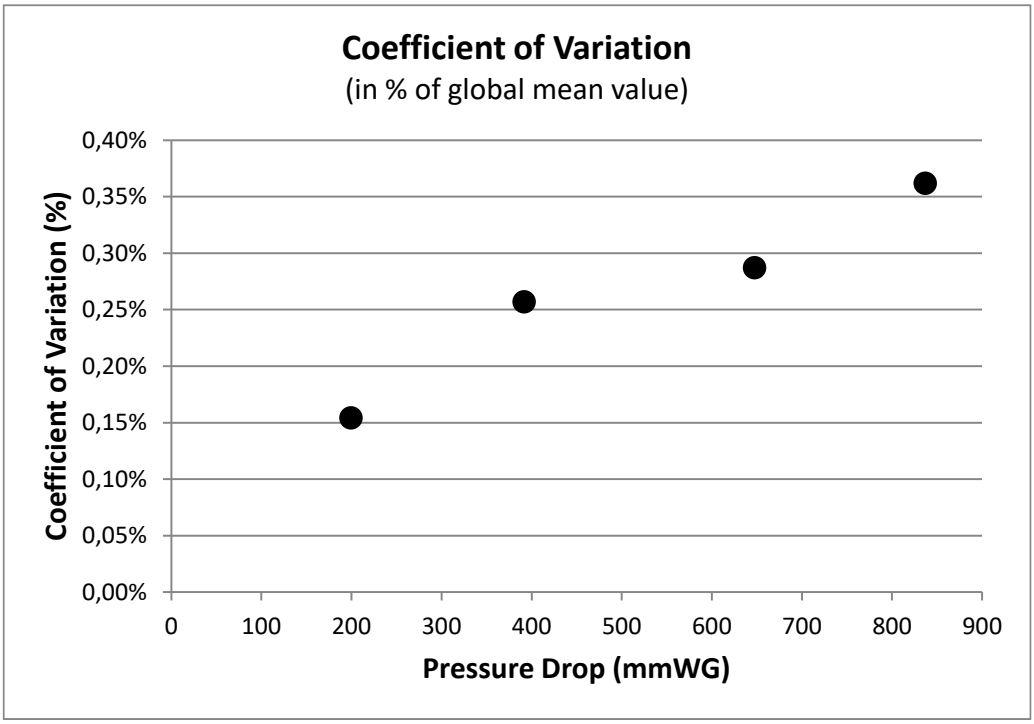

Figure 1: Coefficient of variation of the global mean vs. pressure drop

3.2 Individual Laboratory Results

The results of each laboratory are presented as the means and standard deviations of the six determinations. The mean pressure drop obtained by each laboratory for each calibration standard is given in Table 4. The deviation from the global mean value for each laboratory and calibration standard is given in Table 5. The standard deviation and the coefficient of variation obtained by each laboratory and calibration standard are given in Tables 6 and 7, respectively.

A graphical representation of the percentage deviation from the global mean is shown by laboratory in Figure 2 and by calibration standard in Figure 3.

Table 4: Laboratory Mean by Sample (mmWG)

	Laboratory Code				
Sample	**A**	**B**	**C**	**D**	**E**
200	199,2	199,9	199,7	199,3	199,9
400	390,9	392,7	391,7	391,2	393,4
600	645,9	648,4	647,6	646,0	650,4
800	835,8	839,0	833,4	836,2	841,2

Table 5: Deviation from Global Mean (%)

	Laboratory Code				
Sample	**A**	**B**	**C**	**D**	**E**
200	-0,19 %	0,14 %	0,04 %	-0,14 %	0,14 %
400	-0,27 %	0,17 %	-0,07 %	-0,19 %	0,35 %
600	-0,27 %	0,11 %	-0,01 %	-0,25 %	0,42 %
800	-0,16 %	0,22 %	-0,45 %	-0,10 %	0,49 %

Table 6: Laboratory Standard Deviation by Sample (mmWG)

	Laboratory Code				
Sample	**A**	**B**	**C**	**D**	**E**
200	0,20	0,05	0,23	0,09	0,06
400	0,35	0,15	0,22	0,13	0,14
600	0,18	0,15	0,30	0,17	0,09
800	0,72	0,31	0,35	0,37	0,10

Table 7: Laboratory Coefficient of Variation by Sample (%)

	Laboratory Code				
Sample	**A**	**B**	**C**	**D**	**E**
200	0,10 %	0,03 %	0,12 %	0,05 %	0,03 %
400	0,09 %	0,04 %	0,06 %	0,03 %	0,04 %
600	0,03 %	0,02 %	0,05 %	0,03 %	0,01 %
800	0,09 %	0,04 %	0,04 %	0,04 %	0,01 %

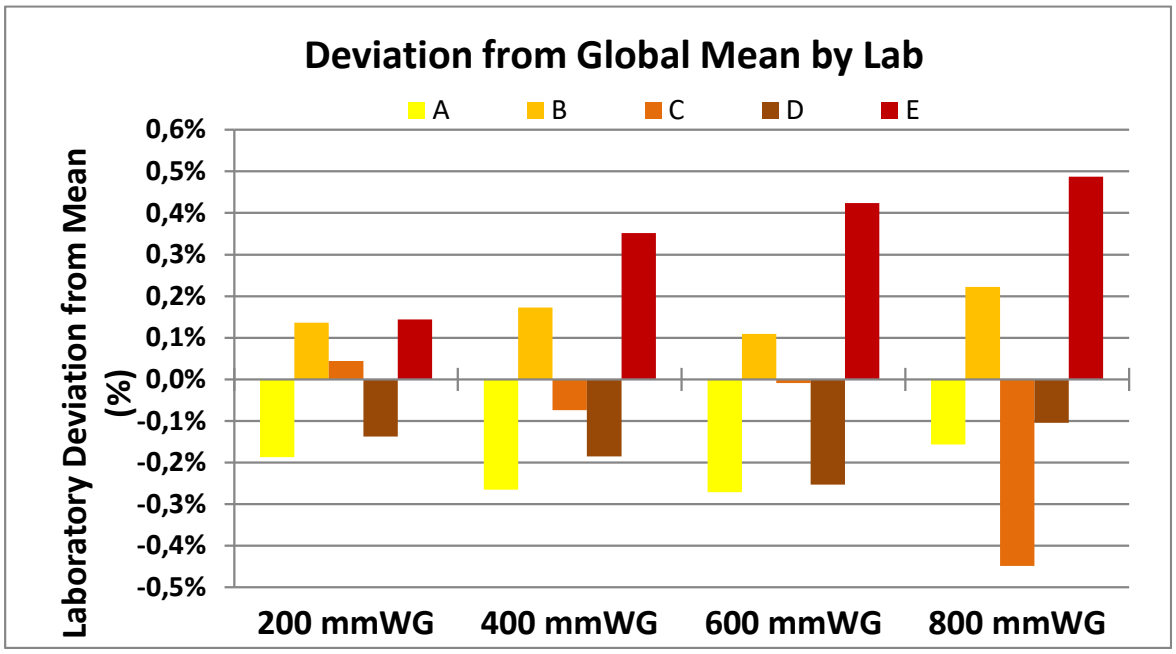

Figure 2: Deviation from global mean by laboratory

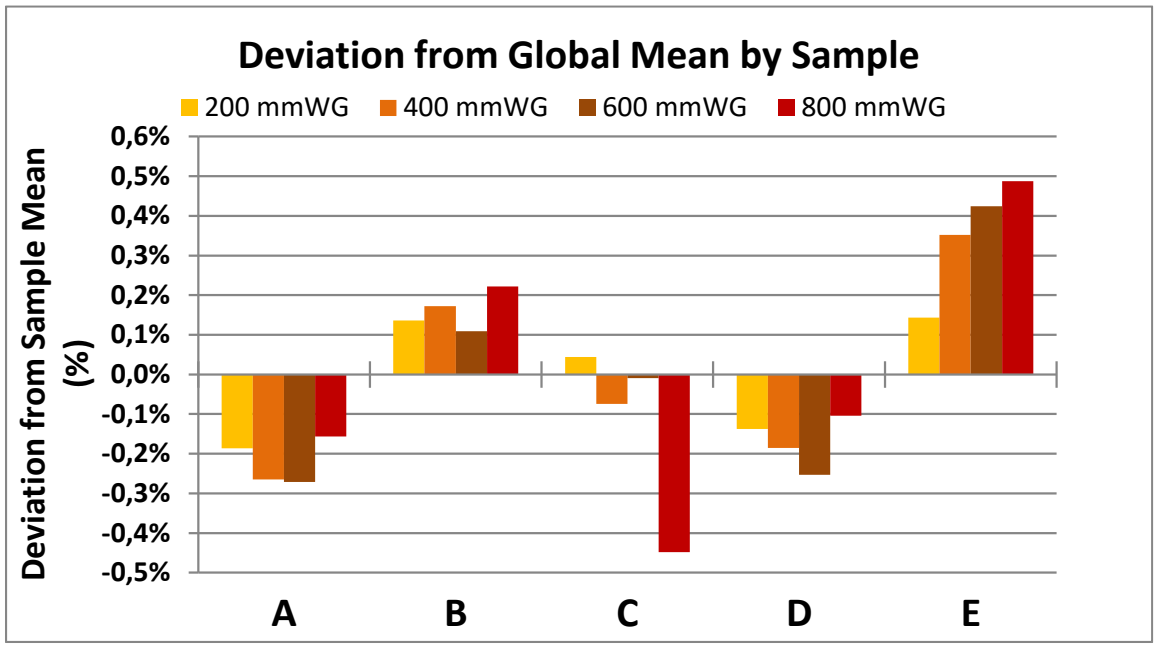

Figure 3: Deviation from global mean by sample

3.3 Re-check of Standards

The pressure drop of the standards was re-checked after the circulation was complete, a period of about 27 months. The change in pressure drop of each standard is presented in Figure 4. The average change was an increase of 0,158 %, with a largest shift of 0,233 %. This is higher than observed in the 15th Round Robin Test on Pressure Drop Calibration Standards and may be due to the longer time that was needed for the circulation of the standards, but it is still within the expected performance of the method. It is thus concluded that there was no change to the value of the standards during circulation that has affected the results.

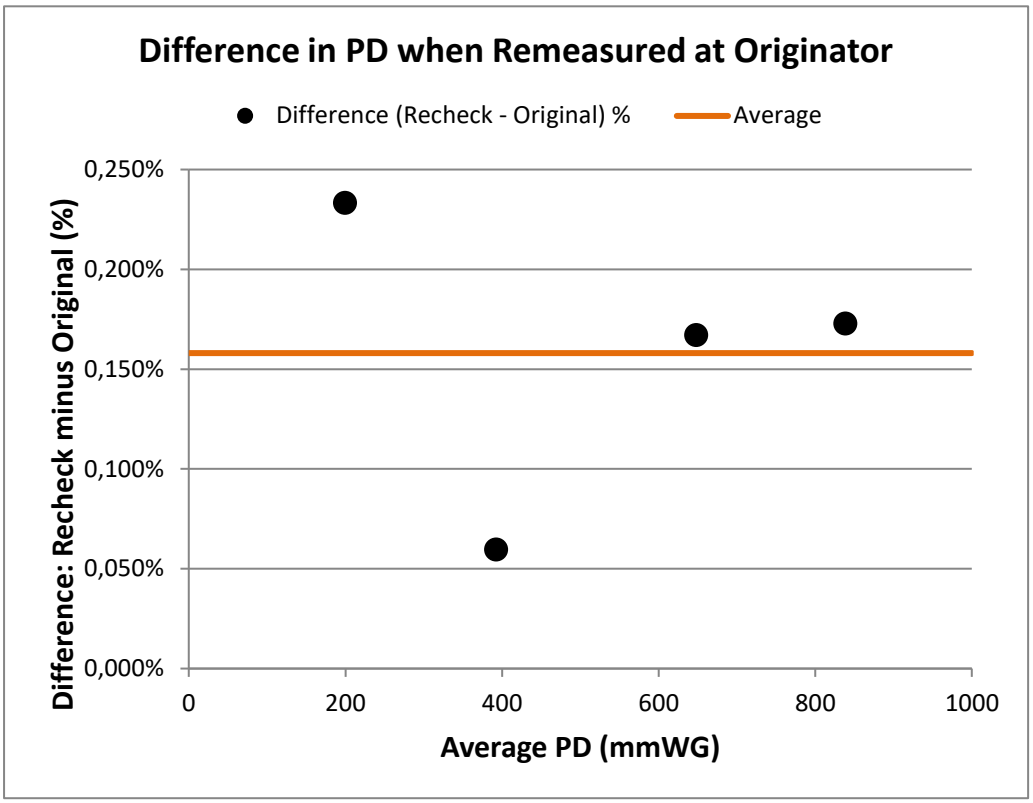

Figure 4: Re-check of the pressure drop standards

3.4 Repeatability and Reproducibility Estimations

Repeatability and reproducibility (r and R) estimations were calculated according to the principles of ISO 5725-2:2019. No outlier testing was performed because there are only five participating laboratories, which represent almost all calibration laboratories available in the industry. Thus the results are not estimates derived from a representative sample of laboratories, but rather represent the values over all laboratories. To account for the low number of laboratories only repeatability and reproducibility standard deviations are presented.

Table 8 presents the summary data and repeatability and reproducibility standard deviations in mmWG and as coefficient of variation in % of the global mean.

Table 8: Repeatability and Reproducibility Standard Deviations (mmWG and CoV%)

	Standard (in mmWG)			
	200	**400**	**600**	**800**
Global Mean for All Laboratories	199,59	391,97	647,66	837,11
Standard Deviation of Lab Means	0,31	1,01	1,86	3,03
Repeatability Standard Deviation	0,15	0,21	0,19	0,42
Reproducibility Standard Deviation	0,34	1,03	1,87	3,05
Repeatability Coefficient of Variation	0,073 %	0,055 %	0,029 %	0,050 %
Reproducibility Coefficient of Variation	0,168 %	0,262 %	0,288 %	0,365 %

3.5 Comparison with Results from Previous Round Robin Tests

A direct comparison between the results of all previous completed round robin tests is presented in Figure 5 in terms of the coefficient of variations of the laboratory means vs overall average pressure drop for each standard. Also plotted in the Figure is the overall average of coefficient of variation for ISO 6565:2015 calculated from the data in Table 2, although this is not a direct comparison due to differences in the experimental protocol and in the calculation of the results. However, the overall picture of the comparison is supported in that the absolute values of repeatability and reproducibility in this round robin test are similar to those in ISO 6565:2015, although as for the 15th Round Robin Test the coefficients of variation have fallen systematically above this historical baseline. This can be ascribed in the results to the fact that laboratories A and D generally measured low values while laboratory E had mostly high values. The comparably higher coefficients of variation may also be attributable to the long time needed for the circulation of the calibration standards.

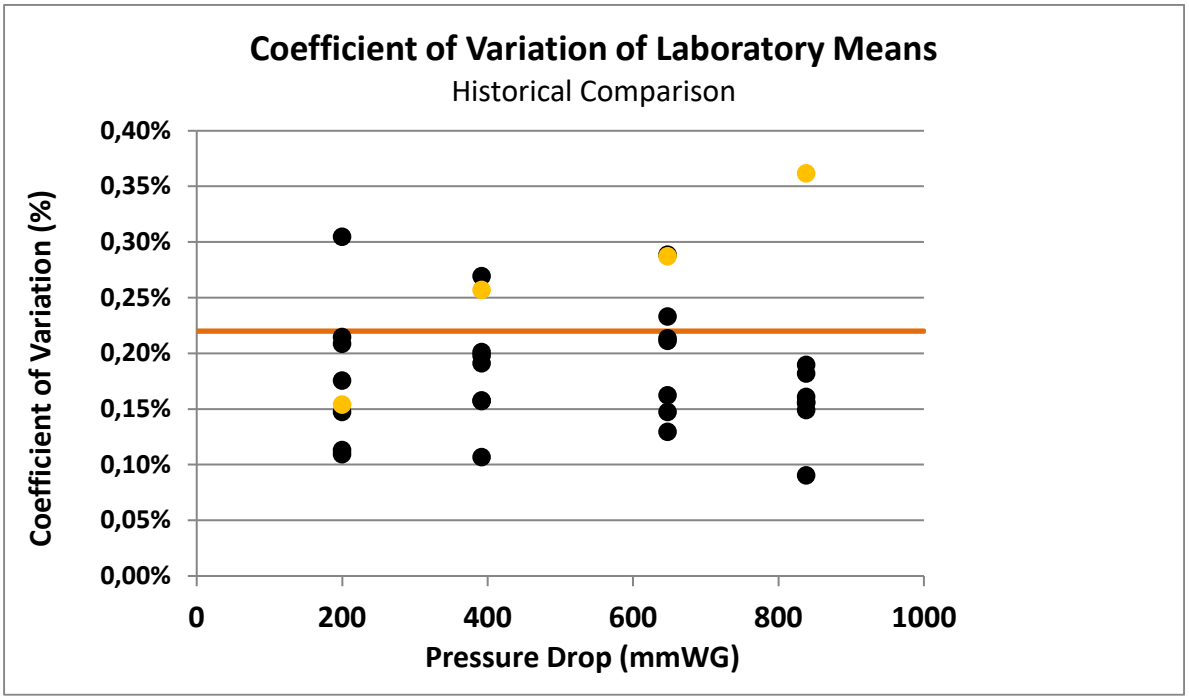

Figure 5: Coefficient of variation of the laboratory means for past Round Robin Tests (black) and this Round Robin Test (orange) with the baseline from ISO 6565

4. Comments on the Results

The results of the 16th Round Robin Test on Pressure Drop Calibration Standards continue to conform to the historical performance of the method presented in ISO 6565:2015 and to the results of previous round robin tests.

The differences between laboratories, see Figure 3, generally appear to be systematic, either as an inter-laboratory offset or scale error, and of the order of tenths of a percent. There also appears to be a smaller additional random contribution, the repeatability coefficient of variation, see Table 7, averaged <0,03 % over all laboratories and standards. This overall difference is likely to be fully accountable from the precision and accuracy of the instrumentation used at the five laboratories for pressure, flow and temperature measurement.

The worst-case offset for pressure drop calibration between laboratories is within approximately 0,9 %. This is consistent with the expanded uncertainty that each lab cites for the method (where available) of typically 0,5 % of value and is small compared to the reproducibility limit for pressure drop of typically 5 % of value for filter rods and 7 % of value for cigarettes that was seen in the 15th Collaborative Study on Physical Parameters of Cigarettes and Filters (PTM-335) undertaken in 2022. Thus instrumental variation deriving from any offset between calibration standards would be expected to represent only a small fraction of the total inter-laboratory variation seen in the collaborative study for both filter and cigarette products.