Physical Test Methods Sub-Group

Technical Report

1st Round Robin Test for Pressure Drop Calibration Standards with Low Pressure Drop (2021-2023)

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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.

In regular round robin tests carried out since 2011 pressure drop calibration standards with a nominal pressure drop of 200 mmWG, 400 mmWG, 600 mmWG and 800 mmWG were circulated. This range of pressure drop has been found useful for measurements of combustible cigarettes and filter rods. However, new product categories, in particular Heated Tobacco Products (HTPs) and their components, may have substantially lower pressure drops. It was thus of interest, whether pressure drop standards with a value below 200 mmWG can be manufactured and calibrated and which repeatability and reproducibility can be expected in the calibration process. The term "low pressure drop" in the context of this Technical Report generally refers to a pressure drop below 200 mmWG.

This report covers the results of the 1st Round Robin Test on Low Pressure Drop (PD) Calibration Standards, circulated between the participating laboratories from September 2021 to January 2023. The test provides a baseline of instrument performance across the industry when measuring low pressure drops.

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. It has to be noted, however, that the calculation model used for this compensation has not yet been verified for such low pressure drops, so that its application in this case is purely based on the – not unreasonable – assumption that it at least approximately also applies to low pressure drops.

The 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, for pressure drops from 200 mmWG to 800 mmWG, i.e. above the range of current interest in this study. However,

these results are useful for a comparison with results achieved with low pressure drop calibration standards.

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 this round robin test a set of low pressure drop calibration standards, supplied by the Zhengzhou Tobacco Research Institute, was circulated between the five participating laboratories. The calibration standards had a nominal pressure drop of 20 mmWG, 40 mmWG and 60 mmWG, respectively, approximately equivalent to 200 Pa, 400 Pa and 600 Pa.

The five participating laboratories in the 1st Round Robin Test on Low 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 other reports on the round robin tests for pressure drop calibration standards.

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 1st 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: 1st 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)
20	20,4	0,12	0,57 %	0,3	1,44 %
40	42,3	0,16	0,37 %	0,4	0,90 %
60	60,2	0,17	0,28 %	0,4	0,64 %

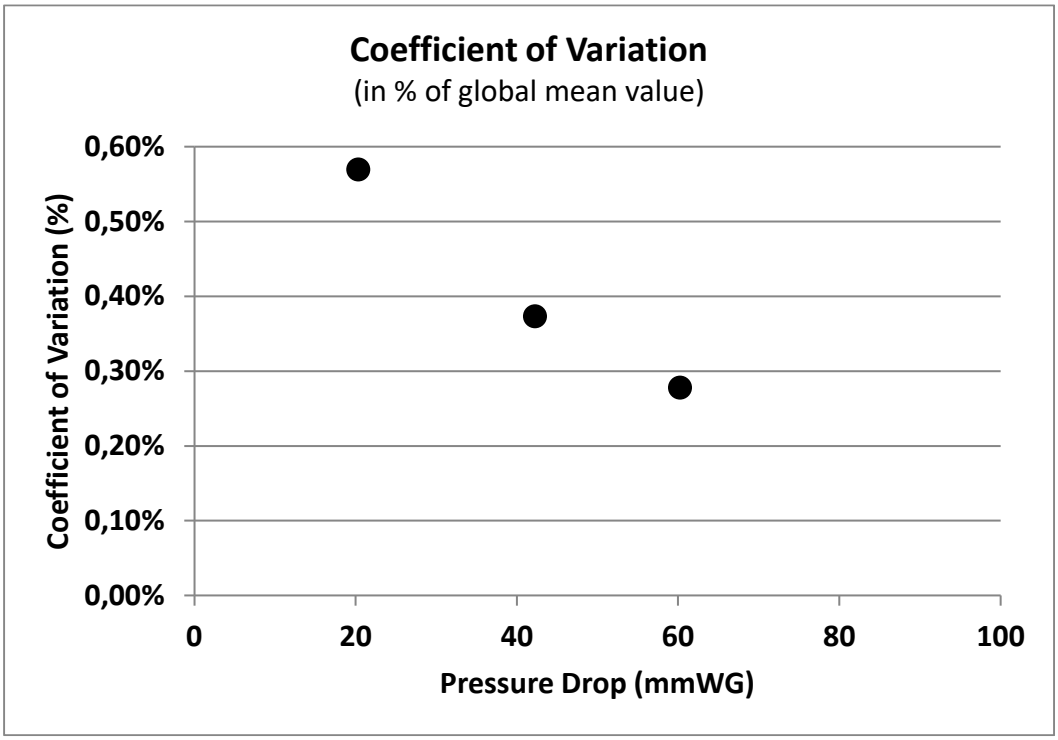

Figure 1: Coefficient of variation of the global mean vs. pressure drop

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**
20	20,3	20,4	20,5	20,3	20,2
40	42,2	42,4	42,5	42,1	42,2
60	60,1	60,4	60,5	60,2	60,1

Table 5: Deviation from Global Mean (%)

	Laboratory Code				
Sample	**A**	**B**	**C**	**D**	**E**
20	-0,11 %	0,32 %	0,79 %	-0,36 %	-0,65 %
40	-0,16 %	0,23 %	0,53 %	-0,37 %	-0,24 %
60	-0,19 %	0,23 %	0,36 %	-0,13 %	-0,28 %

Table 6: Laboratory Standard Deviation by Sample (mmWG)

	Laboratory Code				
Sample	**A**	**B**	**C**	**D**	**E**
20	0,004	0,020	0,041	0,006	0,030
40	0,009	0,023	0,000	0,035	0,044
60	0,009	0,015	0,052	0,084	0,043

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

	Laboratory Code				
Sample	**A**	**B**	**C**	**D**	**E**
20	0,020 %	0,098 %	0,199 %	0,030 %	0,149 %
40	0,021 %	0,053 %	0,000 %	0,083 %	0,105 %
60	0,015 %	0,025 %	0,085 %	0,140 %	0,071 %

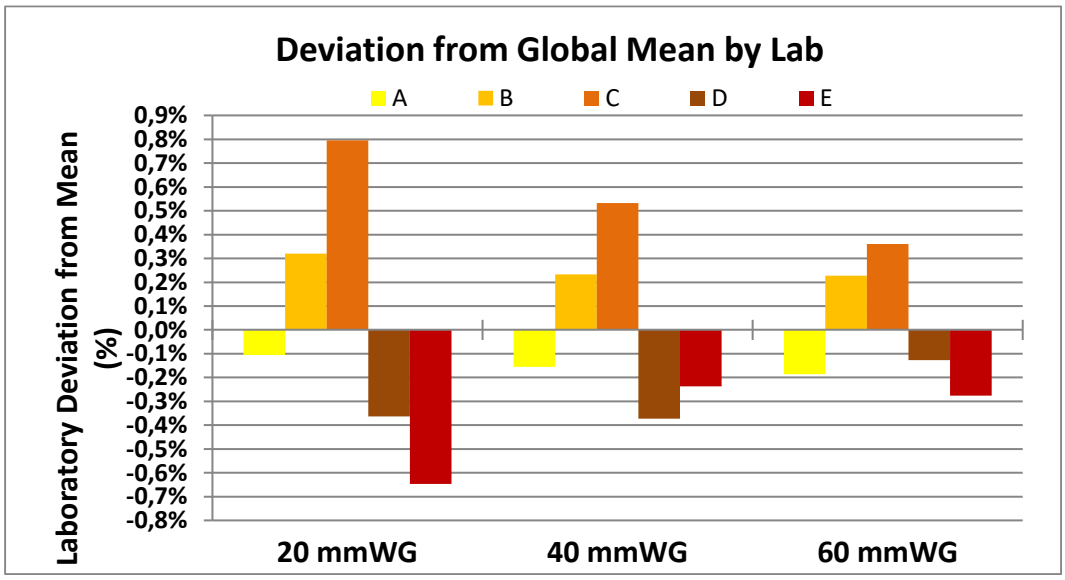

Figure 2: Deviation from Global Mean by Laboratory

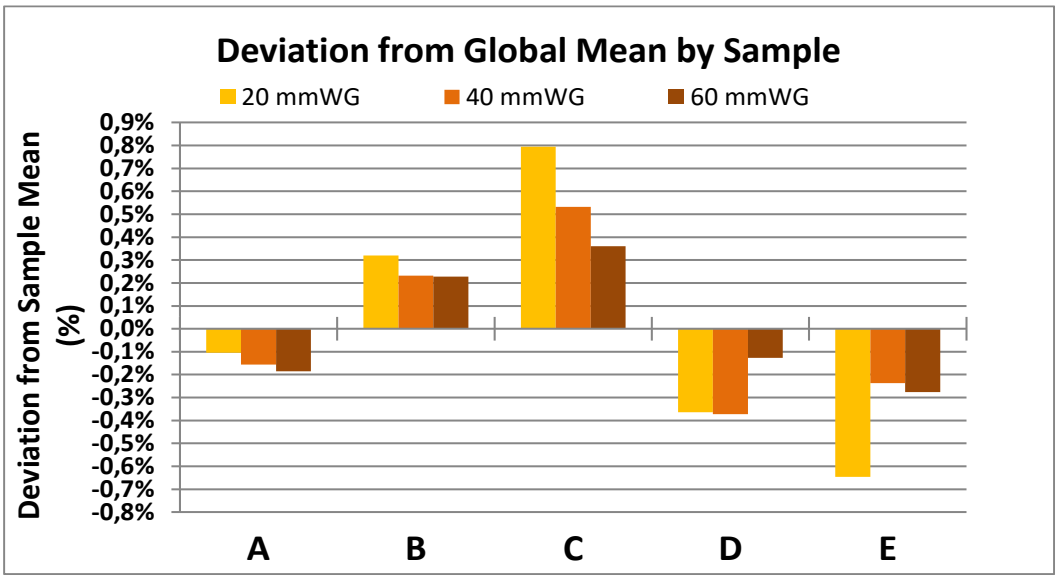

Figure 3: Deviation from Global Mean by Sample

4. Re-check of Standards

The pressure drop of the standards was rechecked after the circulation was complete, a period of about 16 months. The change in pressure drop of each standard is presented in Figure 4. The average change was an increase of 0,892 %, with a largest shift of 1,032 %. This is by a factor of 4 or 5 higher than generally observed for pressure drop calibration standards with nominal values at or above 200 mmWG, but it has to be taken into account that the absolute values for the low pressure drop standards are an order of magnitude lower so that variability as a percentage automatically tends to be higher. Consequently, the change in pressure drop is still considered to be acceptable and for the analysis it is assumed that there was no change to the values of the standards that have affected the results. This is supported by the fact that the pressure drop standards were measured by the laboratories in the order D-C-A-E-B, while

ordering the results approximately from low to high the order is E-D-A-B-C, which indicates that values have not increased from laboratory to laboratory during the circulation.

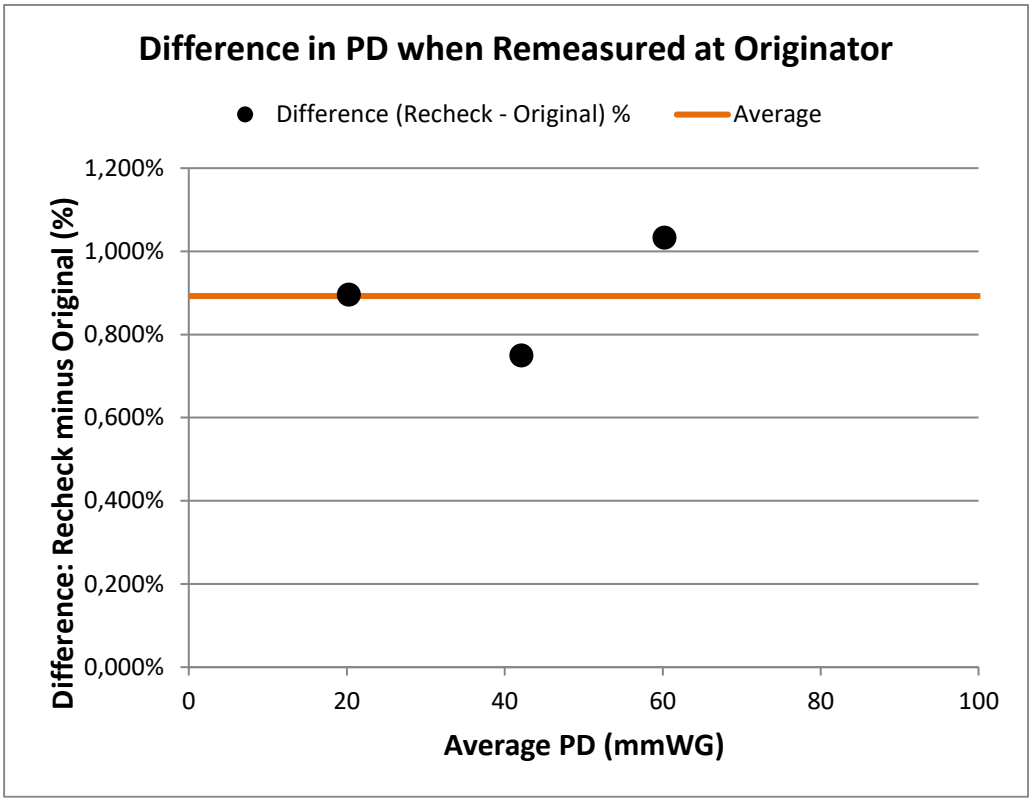

Figure 4: Recheck of the Pressure Drop Standards

5. Repeatability and Reproducibility Estimations

Repeatability and reproducibility standard deviations 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 relevant laboratories. To account for the low number of laboratories only repeatability and reproducibility standard deviations are presented. Table 8 shows 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)		
	20	**40**	**60**
Global Mean for All Laboratories	20,35	42,28	60,25
Standard Deviation of Lab Means	0,12	0,16	0,17
Repeatability Standard Deviation	0,02	0,03	0,05
Reproducibility Standard Deviation	0,12	0,16	0,17
Repeatability Coefficient of Variation	0,121 %	0,065 %	0,081 %
Reproducibility Coefficient of Variation	0,580 %	0,378 %	0,287 %

6. Comparison with Other Results

A direct comparison between the results of round robin tests involving pressure drop standards with nominal values at or above 200 mmWG or with the repeatability and reproducibility standard deviations reported in ISO 6565:2015 is only possible with much caution, as the pressure drop values in this study are much lower and the experimental protocols have been different.

Figure 5 shows the coefficients to variation of the laboratory means obtained in this study (yellow) for comparison with the results obtained in the 16th Round Robin Test on Pressure Drop Calibration Standards (PTM-271) (black) and the repeatability and reproducibility values reported in ISO 6565:2015 (orange line).

However, the overall picture shows that at least the pressure drops with 40 mmWG and 60 mmWG achieve similar results as pressure drops above 400 mmWG. Nevertheless, the values are above the baseline provided by ISO 6565:2015 and it has to be noted that compared to other round robin tests on pressure drop calibration standards the coefficients of variation were rather high in the 16th round robin test. Normally the coefficients of variation are below the baseline reported in ISO 6565:2015.

On an absolute basis the standard deviation over all labs found for the 20 mmWG pressure drop standard is 0,12 mmWG while the same parameter is 0,31 mmWG for the 200 mmWG pressure drop standard, as measured in the 16th Round Robin Test on PD Calibration Standards. Thus, on an absolute scale the precision achieved when calibrating instruments with the low pressure drop calibration standards seems to be sufficient for all practical purpose.

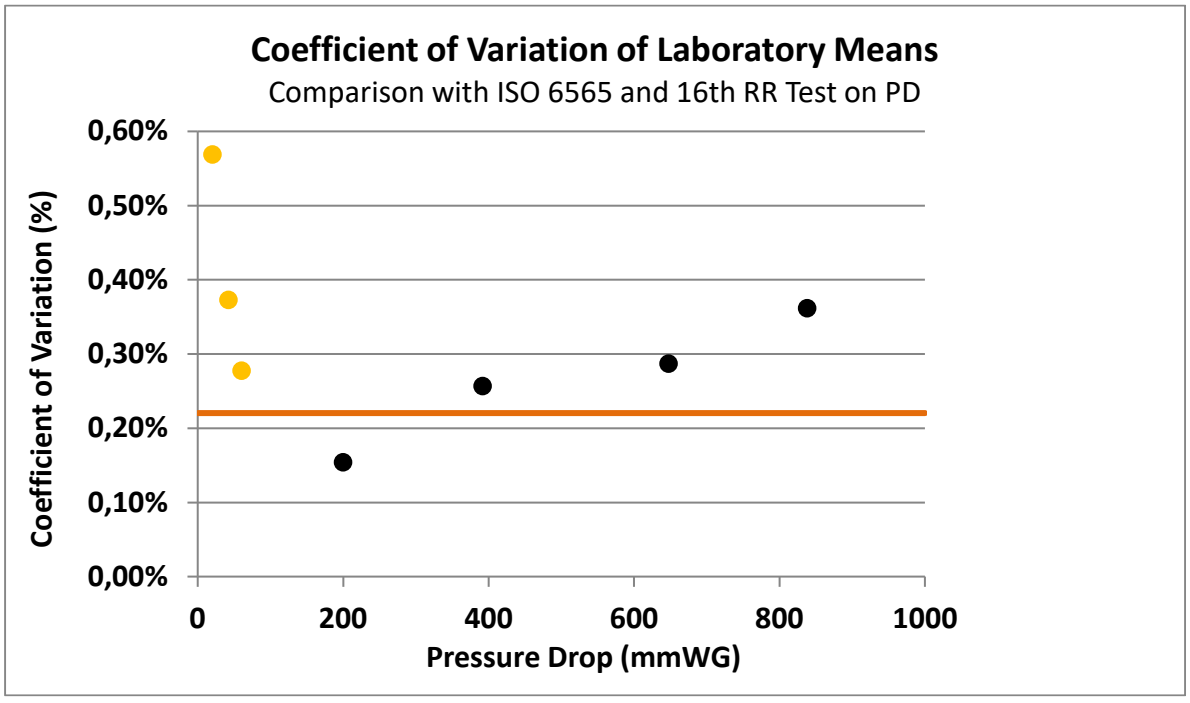

Figure 5: Coefficient of variation of the laboratory means for the 16th Round Robin Tests (black) and this Round Robin Test (yellow) with the baseline from ISO 6565 (orange)

7. Comments on the Results

The results of the 1st Round Robin Test on Low Pressure Drop Calibration Standards serve as an initial baseline for the precision to be expected when calibrating instruments for the measurement of pressure drops below 200 mmWG.

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 a percent or less. There also appears to be a smaller additional random contribution, the repeatability coefficient of variation, see Table 7, averaged <0,08 % 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 and is in line with results obtained for pressure drop standards with a nominal value of 200 mmWG or higher.

The worst-case relative offset for pressure drop calibration between laboratories is within approximately 1,5 % (laboratories C and E for the 20 mmWG pressure drop standard). This is still 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 part of the total inter-laboratory variation seen in the collaborative study for both filter and cigarette products. While the relative variability is unsurprisingly increased compared to pressure drop standards with higher nominal values, the absolute variability is at least comparable and thus should be sufficient for all practical purposes.

It is recommended that the calculation model used to compensate deviations in absolute pressure, temperature, relative humidity and flow rate from standard conditions is verified for its applicability to pressure drops below 200 mmWG. Also it is recommended that calibration standards for low pressure drops are regularly circulated in round robin tests.