Physical Test Methods Sub-Group

Technical Report

12th Round Robin Test for Multi-Capillary Pressure Drop Calibration Standards (2016/2017)

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1. Introduction and Background

The CORESTA Physical Test Methods (PTM) Sub-Group organizes a nominally annual series of 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 result set in internal and external audit assessments.

This report covers the results of the 12th pressure drop (PD) standards round robin test conducted between May 2016 and July 2017. 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 relevant 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 long 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 °C
- Atmospheric humidity 60 %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 Std Dev (sr)	0.21	0.33	0.44	0.48
Reproducibility Std Dev (sR)	0.43	0.96	1.18	1.83

In the PD round robin tests 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 ("Nom 200"), 400 mmWG ("Nom 400"), 600 mmWG ("Nom 600") and 800 mmWG ("Nom 800"), approximately equivalent to 2 kPa, 4 kPa, 6 kPa and 8 kPa.

The five participating laboratories are listed in Table 2. The laboratory identities are coded in the results presented below.

Table 2: Participating Laboratories

Participating laboratory	Function	Accreditation
Borgwaldt KC, Hamburg, Germany	Calibration lab & instrumentation supplier	ISO 9001 & 17025
Cerulean, Milton Keynes, UK	Calibration lab & instrumentation supplier	ISO 9001 & 17025
JA King, Winston-Salem, NC, USA	Calibration laboratory	ISO 17025
SODIM, Fleury-les-Aubrais, France	Calibration lab & instrumentation supplier	ISO 9001 & 17025
ZTRI of CNTC, Zhengzhou, PRC	Calibration laboratory	

There have been different participants in the round robin tests since the publication of ISO 6565:2015. The 8th test (2011) included only the three instrument suppliers; ZTRI (China) joined from test 10 (2014) onwards and JA King (USA) joined for this latest test. 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.

3. 12 PD Results

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: PTM 12 PD Round Robin Test – Overall Results

	Global mean (mmWG)	Std dev of lab means* (mmWG)	CoV of lab means (%)	Range (mmWG)	Range (% of value)
Nom 200	199.4	0.42	0.21 %	1.0	0.50 %
Nom 400	391.7	0.62	0.16 %	1.4	0.35 %
Nom 600	647.4	0.84	0.13 %	1.9	0.29 %
Nom 800	837.9	0.76	0.09 %	1.9	0.22 %

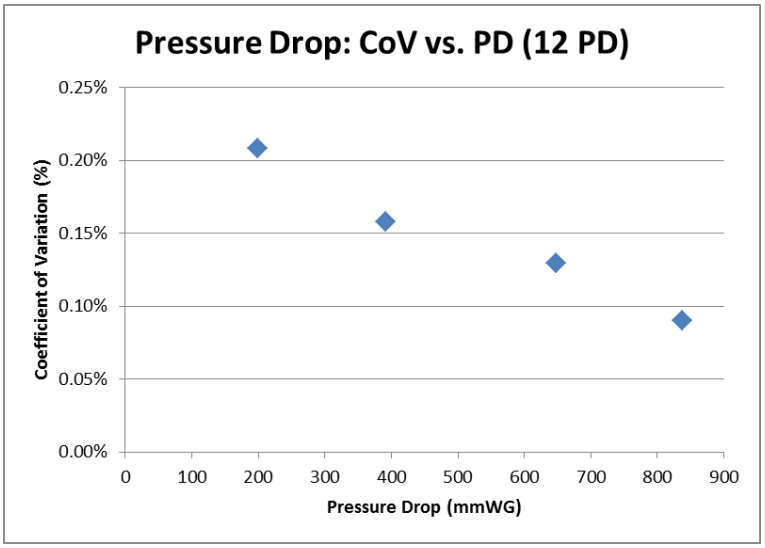

Figure 1: Pressure Drop - CoV of Lab Means vs. PD

3.2 Individual laboratory results

The results of each laboratory are presented as the means and standard deviations of the six determinations. The mean PD 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: PTM 12 PD Round Robin Test – Lab Mean by Sample (mmWG)

	LABORATORIES				
	A	**B**	**C**	**D**	**E**
Nom 200	199.3	199.7	199.6	199.7	198.7
Nom 400	391.2	392.3	392.1	392.1	390.9
Nom 600	646.4	648.3	647.9	647.8	646.6
Nom 800	836.6	838.3	838.3	838.5	837.8

Table 5: PTM 12 PD Round Robin Test – Deviation from Global Mean (%)

	LABORATORIES				
	A	**B**	**C**	**D**	**E**
Nom 200	-0.05 %	0.15 %	0.10 %	0.14 %	-0.34 %
Nom 400	-0.13 %	0.14 %	0.11 %	0.09 %	-0.21 %
Nom 600	-0.15 %	0.14 %	0.08 %	0.06 %	-0.12 %
Nom 800	-0.15 %	0.05 %	0.05 %	0.07 %	-0.01 %

Table 6: PTM 12 PD Round Robin Test – Lab Std Deviation by Sample (mmWG)

	LABORATORIES				
	A	**B**	**C**	**D**	**E**
Nom 200	0.16	0.08	0.10	0.06	0.05
Nom 400	0.21	0.32	0.15	0.22	0.11
Nom 600	0.13	0.12	0.08	0.08	0.17
Nom 800	0.61	0.67	0.50	0.16	0.07

Table 7: PTM 12 PD Round Robin Test – Lab CoV by Sample (%)

	LABORATORIES				
	A	**B**	**C**	**D**	**E**
Nom 200	0.08 %	0.04 %	0.05 %	0.03 %	0.02 %
Nom 400	0.05 %	0.08 %	0.04 %	0.06 %	0.03 %
Nom 600	0.02 %	0.02 %	0.01 %	0.01 %	0.03 %
Nom 800	0.07 %	0.08 %	0.06 %	0.02 %	0.01 %

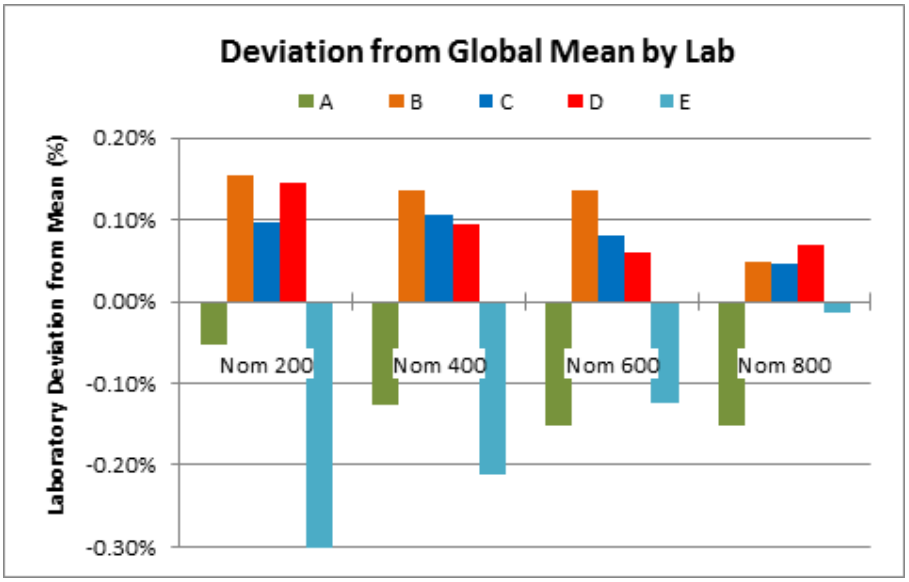

Figure 2: Deviation from Global Mean by Lab

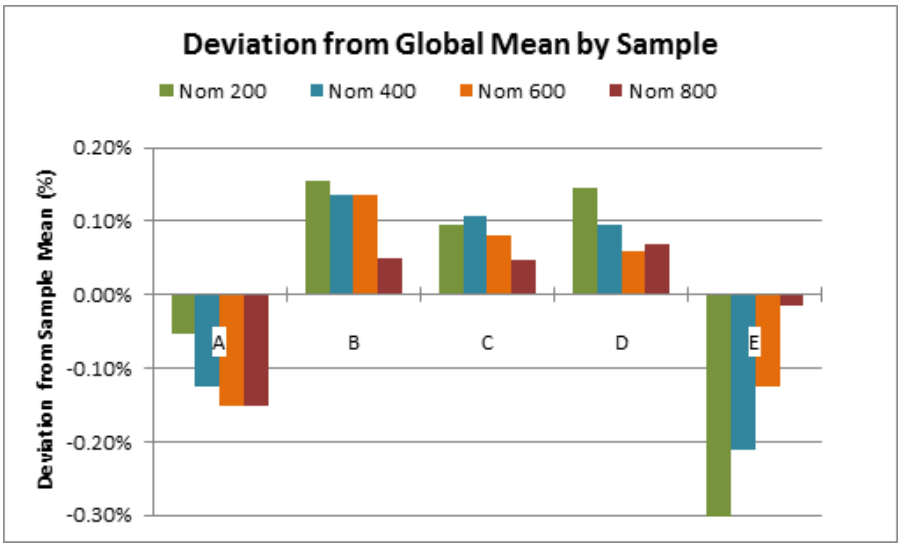

Figure 3: Deviation from Global Mean by Sample

3.3 Recheck of standards

The PD values of the standards were rechecked after the circulation was complete, a period of about 14 months. The change in PD value of each standard is presented in Figure 4. The average change was an increase of 0.05 %, but even the largest shift of 0.20 % is 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.

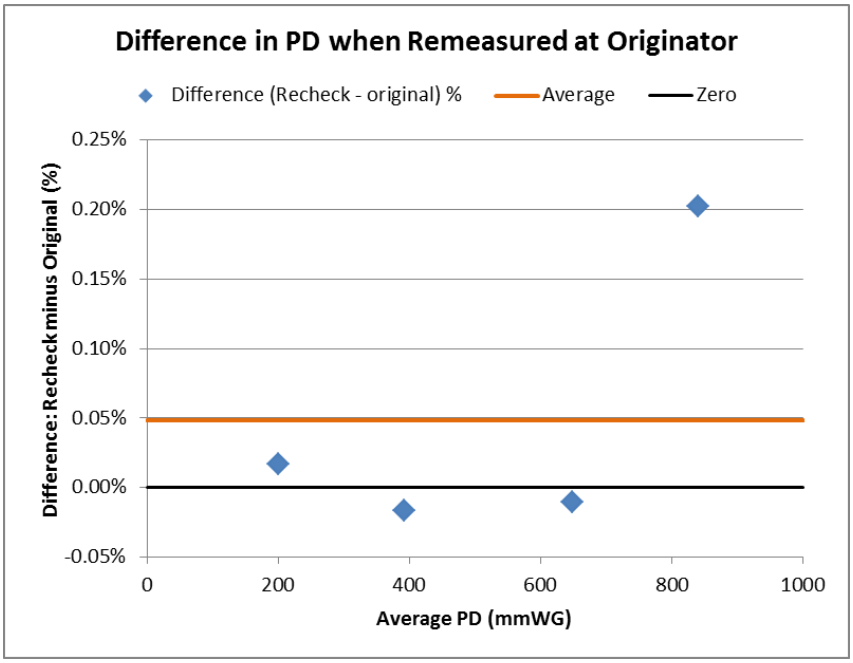

Figure 4: Recheck of PD Standards

3.4 Repeatability and reproducibility estimations

Repeatability and reproducibility (r and R) estimations were calculated according to the principles of ISO 5725:1994. No outliers were detected according to Mandel's *h* and *k* statistics, although laboratories A and E were close to the lower limit for *h* for the 800 mmWG and 200 mmWG standards respectively. With the participation of just five laboratories, only r and R standard deviations are presented.

Table 8 presents the summary data and r and R estimations (sr and sR) as PD (mmWG) and CoV%.

Table 8: Summary data and r and R estimations (mmWG and CoV%)

	Standard			
	Nom 200	**Nom 400**	**Nom 600**	**Nom 800**
Grand Mean for All Labs	199.43	391.72	647.40	837.89
Std Dev of Lab Means	0.42	0.62	0.84	0.76
Repeatability Std Dev (sr)	0.10	0.21	0.12	0.47
Reproducibility Std Dev (sR)	0.43	0.65	0.85	0.87
Repeatability (sr) CoV	0.05 %	0.05 %	0.02 %	0.06 %
Reproducibility (sR) CoV	0.21 %	0.17 %	0.13 %	0.10 %

3.5 Comparison between results from ISO 6565:2015 and the round robin tests

A direct comparison between the results of the last four completed tests is presented in Figure 5 in terms of the CoV of laboratory means vs overall average PD for each standard. Also plotted in the Figure is the overall average of CoV(sR) 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 sr and sR in the 12 PD round robin test are equal or lower than those in ISO 6565:2015.

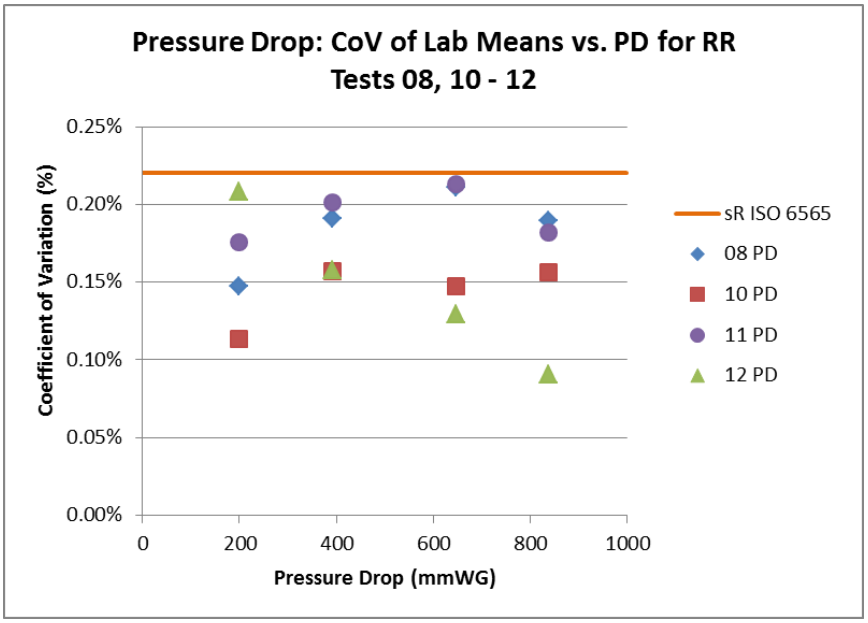

Figure 5: CoV of Laboratory Means vs. PD for RR Tests 08, 10 – 12 Plus the CoV of sR for ISO 6565

4. Comments on Results

The results of the 12th PD round robin test continue to conform to the historical performance of the method presented in ISO 6565:2015 and in subsequent tests. This was not affected by the addition of a new laboratory in this round.

The differences between laboratories (see Figure 3) 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 CoV (see Table 7) was <0.1 % for all labs and all 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 PD calibration between laboratories is within 0.5 %, which is small compared to the reproducibility limit for PD of typically 5 % of value that was seen in the 9th annual PTM Proficiency Test for physical parameters of cigarettes and filters undertaken in 2016. Thus instrumental variation deriving from any offset between calibration standards would be expected to represent at most 1 % of the total inter-laboratory variation seen in the proficiency study for both filter and cigarette products.