



Physical Test Methods Sub-Group

Technical Report

**11th Round Robin Test for
Multi-Capillary Pressure Drop
Calibration Standards
(2016)**

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1. Introduction and Background

The CORESTA Physical Test Methods (PTM) Sub-Group organizes a nominally annual round robin test which is open to the member laboratories that have a calibration laboratory to compare their capability to calibrate standards used in physical test instrumentation. This report covers the results of the 11th pressure drop (PD) standards round robin test to provide a baseline of PD instrument performance across the industry, since this standard type is used in the PD instrumentation of each supplier. Each laboratory is also able to use the result set in internal and external audit assessments.

The relevant international standard is ISO 6565:2011 “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 air-flow is applied. 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 6565:2011 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·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 atmosphere plays a significant part in standardising pressure drop measurements across the industry.

During the development of ISO 6565:2011 the precision of calibration of pressure drop transfer standards was determined between three suppliers, as presented in Table 1:

Table 1: ISO 6565:2011 - r and R estimations for calibration of standards (mmWG)

	Standard			
	Nom 200	Nom 400	Nom 600	Nom 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

The three instrumentation suppliers use the same physical test piece design and test pieces that are all supplied from a single source, thus only a single set of standards was circulated. These presented pressure drops of nominally 200 mmWG (~2 kPa), 400 mmWG (~4 kPa), 600 mmWG (~6 kPa) and 800 mmWG (~8 kPa).

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

Table 2: Participating Laboratories

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

2. Experimental Protocol

The protocol involved:

- acclimatisation of the standards to laboratory conditions
- testing to the method detailed in ISO 6565:2011
- making three PD determinations under repeatability conditions for each standard on two separate days

After circulation of the pressure drop calibration standards, the standards were rechecked by the originator.

3. 11 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 11 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	199.7	0.35	0.18 %	0.8	0.42 %
Nom 400	391.8	0.79	0.20 %	1.8	0.45 %
Nom 600	648.0	1.38	0.21 %	3.2	0.49 %
Nom 800	837.6	1.52	0.18 %	3.5	0.41 %

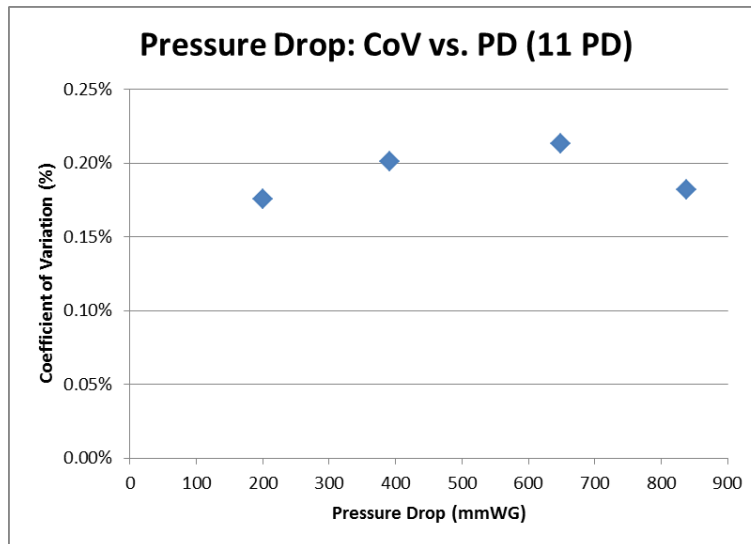


Figure 1: Pressure Drop - CoV vs. PD

3.2 Individual laboratory results

The mean value 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 deviation by laboratory is shown in Figure 2 and by calibration standard in Figure 3.

Table 4: PTM PD Round Robin Test – Lab Mean by Sample (mmWG)

	LABORATORIES			
	A	B	C	D
Nom 200	199.2	200.0	199.8	199.8
Nom 400	390.8	392.5	392.3	391.8
Nom 600	646.4	649.6	648.6	647.3
Nom 800	835.5	838.9	838.4	837.5

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

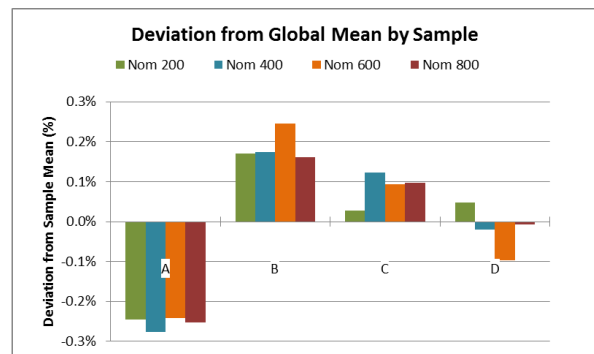
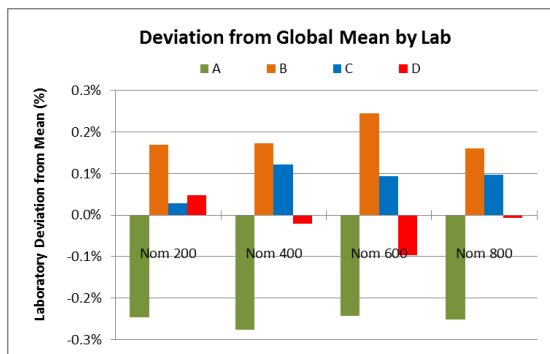
	LABORATORIES			
	A	B	C	D
Nom 200	-0.25 %	0.17 %	0.03 %	0.05 %
Nom 400	-0.28 %	0.17 %	0.12 %	-0.02 %
Nom 600	-0.24 %	0.24 %	0.09 %	-0.10 %
Nom 800	-0.25 %	0.16 %	0.10 %	-0.01 %

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

	LABORATORIES			
	A	B	C	D
Nom 200	0.19	0.08	0.05	0.07
Nom 400	0.25	0.08	0.12	0.19
Nom 600	0.34	0.21	0.12	0.49
Nom 800	0.28	0.15	0.17	0.26

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

	LABORATORIES			
	A	B	C	D
Nom 200	0.09 %	0.04 %	0.03 %	0.04 %
Nom 400	0.06 %	0.02 %	0.03 %	0.05 %
Nom 600	0.05 %	0.03 %	0.02 %	0.08 %
Nom 800	0.03 %	0.02 %	0.02 %	0.03 %



Figures 2 (left) and 3 (right): Deviation from Global Mean by Lab & by Sample

3.3 Recheck of standards

The PD values of the standards were rechecked after the circulation was complete. The PD value of each standard had increased by $(0.13 \pm 0.10) \%$. This level of difference could be due to a systematic drift or re-calibration adjustment of instrumentation in the fifteen months between measurements, or to accumulation of dirt systematically blocking the capillary flow paths in the standards. Whatever the cause, the change is within the overall 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.

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 Laboratory A was close to the lower limit for h . With the participation of just four laboratories, only r and R standard deviations are presented.

Table 8 presents the summary data and r and R estimations (s_r and s_R) as % ventilation 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	199.69	391.84	647.96	837.57
Std Dev of Lab Means	0.35	0.79	1.38	1.52
Repeatability Std Dev (s_r)	0.11	0.17	0.32	0.22
Reproducibility Std Dev (s_R)	0.36	0.80	1.41	1.54
Repeatability (s_r) CoV	0.06 %	0.04 %	0.05 %	0.03 %
Reproducibility (s_R) CoV	0.18 %	0.21 %	0.22 %	0.18 %

3.5 Comparison between results from ISO 6565:2011 and the 8th, 10th & 11th round robin tests

A direct comparison between the results of the 8th (2011), 10th (2014) and 11th round robin tests is presented in Figure 4 in terms of the CoV of laboratory means vs PD for each standard. The 8th test included only the three instrument suppliers. The 9th test was aborted due to an extended delay during shipping the standards between participants.

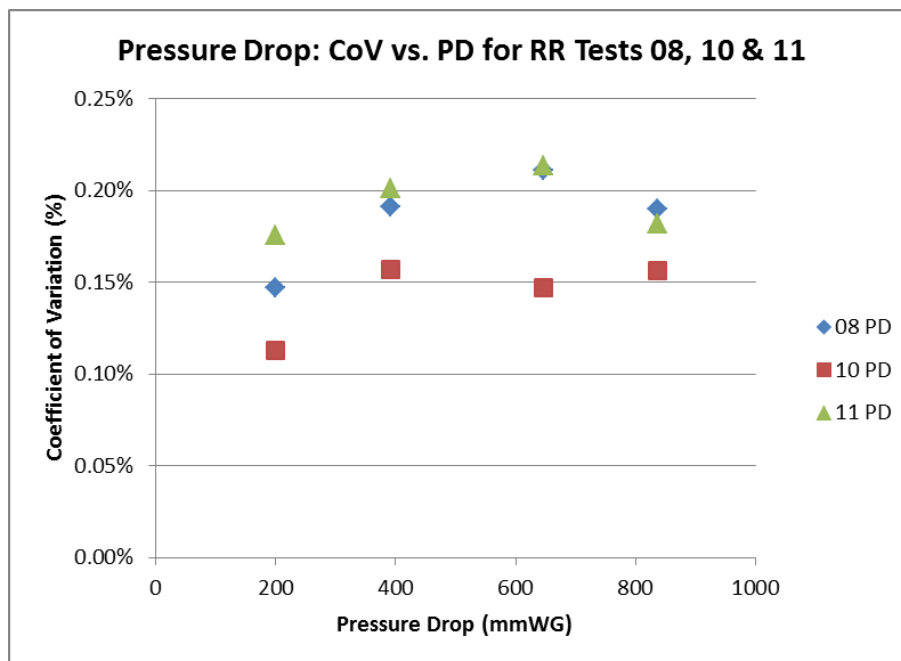


Figure 4: Pressure Drop – CoV vs. PD for RR tests 08, 10 & 11

It is not possible to make a *direct* comparison between the *r* and *R* standard deviations presented in ISO 6565:2011 (Table 2) and the values for 11 PD because of differences in the experimental protocol, however it can be seen that the absolute values of *sr* and *sR* are generally somewhat lower in the 11 PD round robin test than in ISO 6565:2011.

4. Comments on Results

The PD results conform to the historical performance of the method and in fact almost duplicate the results of the 10th round robin test, with laboratory A's determinations consistently about 0.25 % less than the global mean for each standard. This is likely to be due to a calibration offset of instrumentation or equipment.

The worst-case offset between laboratories is within 0.5 %, which is small compared to the reproducibility limit for PD of typically 6 % of value that was seen in the annual PTM Proficiency Tests from 2007 – 2011. Thus instrumental variation deriving from any offset in the calibration of standards is negligible as a contributing cause of inter-laboratory variation seen in the proficiency study.