

Cooperation Centre for Scientific Research Relative to Tobacco

CORESTA Guide N° 4

A User Guideline for the Measurement of Pressure Drop of Cigarettes and Cigarette Filter Rods

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Physical Test Methods Sub-Group



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A User Guideline for the Measurement of Pressure Drop of Cigarettes and Cigarette Filter Rods

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1. Introduction

1.1 Purpose

The pressure drop of cigarette filter rods and the draw resistance of cigarettes have a direct effect on the smoking performance of the assembled cigarette and are principal measures for the quality control of cigarette manufacture. This guideline is intended as a reference for manufacturers of cigarettes and cigarette filter rods as to the best practice for the measurement of pressure drop, so as to consistently obtain the most accurate and most reliable measurements despite the many external influences that can affect the measured value.

Note: - To simplify the text, the term pressure drop (abbreviated to PD) has been used throughout this document to refer to both the pressure drop of filter rods and the draw resistance of cigarettes. The user should refer to ISO 6565:2015 for the definitive definitions of Pressure Drop and Draw Resistance.

1.2 Outline

The definition of PD, as used in the tobacco industry, is given in CORESTA Recommended Method No. 41 (CRM 41) [1]. This definition is extended in ISO 6565:2015 [2] to limit the measurement to "standard conditions" but, unfortunately, these conditions are not unambiguously defined. Furthermore, whilst they form the basic reference documents for PD measurement, these two documents give only limited practical advice to the user on how to carry out some of the essential parts of the measurement process.

Work done within the CORESTA Task Force for the Calibration of Pressure Drop Standards has quantified the environmental influences on the measured PD and additional work carried out since has highlighted a number of other potential causes for variation in the measured PD of a filter rod or cigarette sample.

In order to maintain the validity of PD measurements, attention needs to be given to all of the factors that impact their accuracy. This can only be done by understanding the nature and magnitude of the influences and how they might be minimised and controlled.

2. The PD measurement process

2.1 Introduction

PD measurement brings together several factors, including samples, instruments, calibration standards and various procedures and techniques. Each of these factors must be optimised if it is not to disturb or corrupt the measurement process. Hence, it is important to understand the effect that external influences might have on each of these factors and the influence of each factor on the overall process. Where optimisation is not practicable or possible, allowance needs to be made for potential errors incurred.

2.2 Factors affecting the measured PD

PD is a measure of the static pressure difference across a cigarette or filter rod at a defined volumetric airflow. The magnitude of this airflow volume is sensitive to the temperature, pressure and humidity of the measurement air [3]. Also, the pressure difference across the sample is dependent upon the density and viscosity of the air flowing through it [3].

The measured PD is therefore affected by the following factors:

- ambient conditions at the time of measurement
- sample temperature and moisture content at the time of measurement
- temperature of standards and ambient conditions at the time when the instrument was last adjusted
- changes in ambient conditions since the instrument was last adjusted

In addition, any errors within the instrument that affect the measurement airflow (e.g. leaks, blockages) and any errors in the marked value of the calibration standard (e.g. damage or contamination) will also have an effect on the measured PD value.

Finally, when expressing and interpreting measurement results, it is necessary to have a knowledge of the capability and uncertainty of the measurement process. This will ensure that valid inferences are drawn from the results.

To maintain the validity of measurements, the full measurement process should take account of all of these influences. These factors will be considered here under the following headings:

- Confirming instrument function
 - Checking for correct instrument function
- Maintenance and care of calibration standards
- Checking the validity of measurements
 - Calibration
 - Verification
 - o Adjustment

Note: **Calibration** is the process of checking the accuracy of the instrument vs. standards; this definition means that 'calibration check' has no additional meaning, so is not used in this document. The results of the calibration are **verified** against the specified requirements and may be acceptable as they stand. If not, **adjustment** is the process of adjusting the indication of the instrument to optimise its performance. Following any adjustment (in general) a second calibration / verification is performed to establish that the instrument is performing within specification.

- Instrument performance testing
 - Assessing repeatability and instrument-to-instrument variability
- The PD measurement
 - Sample handling and preparation
 - Measuring samples
 - Expressing results
 - Assessing measurement uncertainty

3. Confirming correct instrument function

3.1 Introduction

Checks for PD instrument function are trying to assure all instrument components and systems are set up and operating correctly. They are separate from any measures of instrument performance and are a necessary pre-condition for all of the following checks and validations.

3.2 Methods of checking and assuring instrument function

The first step in assuring correct function should be a programme of regular preventative maintenance. Instrument manufacturers provide information and guidance on instrument maintenance and offer programmes of planned maintenance. These should include attention on a regular basis (e.g. say daily, weekly and annually). Differences between instruments of different manufacture mean that there will necessarily be some differences in their planned maintenance programmes. However, routine daily and weekly tasks should include:

- Checks on instrument supplies
 - Check that all air and vacuum levels are set correctly
 - Blow down air set filter bottles to remove collected moisture
- Checks of wear parts and consumables
 - PD sleeves should be changed regularly
 - Protective filters should be changed in good time
- Checks for build-up of contamination within the instrument
 - Remove all tobacco fall-out and clean out tobacco traps
- A test measurement run to confirm correct operation following any maintenance or adjustment

Planned maintenance should be supplemented by regular functional checks to provide early warning of faults. Many instruments have a self-diagnostic mode or a system of built-in diagnostics that may be used for this purpose. Alternatively, critical observation of key instrument functions under normal operating conditions of use can show up developing faults. Such observation can usefully be carried out as part of a test measurement run following routine preventative maintenance.

4. Care and maintenance of PD calibration standards

4.1 Introduction

This is a key area since the ultimate accuracy of any measurements will be directly affected by the accuracy of the PD standards used to make the instrument calibration.

4.2 Essential properties of calibration standards

Standards for use with CRM 41 and ISO 6565:2015 are multi-capillary standards manufactured from glass. These have 10 fine capillaries encapsulated in a glass rod and have the following properties:

- They are stable and chemically inert
- They closely resemble the physical size and shape of a filter rod or cigarette
- They have repeatable values with a low uncertainty of the calibrated value

All calibration standards should be provided with a certificate that:

- Records the value and unique identification of the standard
- Gives information about the conditions of calibration and the uncertainty of the assigned value
- Defines the method and validity of the calibration

4.3 Recommendations for the management of standards

PD calibration standards, being made of glass, are susceptible to damage by careless handling. In use, air is passed through the standards. Air from the surrounding atmosphere will almost certainly contain dust particles that will slowly contaminate the capillaries. Calibration standards should, therefore, be stored and handled with care to avoid damage or excessive contamination.

The validity of PD measurements can be improved if a suitable regime is used for the management of PD calibration standards. In particular:

- Store and handle standards carefully and appropriately
- Carry out a visual check on a regular basis. In particular check for:
 - Damage (blemishes, chips, cracks)
 - Contamination, particularly in the capillaries.

Note: - Contamination in the capillaries may not always be visible but will result in an increased PD. The easiest way to check for this non-visible type of contamination is to carry out a PD cross-check against the master set (see below)

- Maintain a master set of PD standards to allow for simple cross checking of standards. The set should cover the range of PD values of interest – a range of 50 mmWG to 800 mmWG is suggested, whereby 1 mmWG (mm Water Gauge) = 9,807 Pa.
- Introduce a documented programme of checking for calibration standards against the reference set, including: -
 - Any standard suspected of damage or contamination
 - Any new standard before it is released for use
 - All standards at regular, fixed intervals
- Any standard found to be damaged, contaminated or out of value should be withdrawn from service until it can be replaced or cleaned and recalibrated.

- Recalibrate standards regularly. The interval chosen should reflect the type of duty to which the standards are put heavily used standards will require recalibration more frequently than lightly used ones. Set a maximum interval for recalibration.
- Maintain the reference standard set within its validity period recalibrate it at least every two years.

4.4 Calibration of transfer standards

The procedure for the calibration of PD standards was established in work undertaken under the auspices of CORESTA, starting in the late 1990's. It was published in 2004 [3] and is incorporated into both CRM 41 and ISO 6565:2015. The work developed the means to compensate the actual PD measured during calibration *to what it would have been* under industry standard conditions of:

•	Outlet flowrate	17.5 ml/s (volume flow)
•	Ambient temperature	22 °C
•	Ambient pressure	1013.25 hPa (= 1013.25 mbar)
•	Ambient humidity	60 % RH

Calibration laboratories maintain conditions close to 22 °C and 60 % RH, but of course there is no control over pressure (or elevation!); in any one location air pressure varies typically within a span of ± 3 % from the long term mean, and occasionally more, and air pressure reduces by approximately 1 % for every 100 m increase in elevation.

The CORESTA study involved

- 1. characterising the PD of standards of a range of values over a range of conditions and
- 2. a theoretical consideration of the physical properties of air flowing through the standard

to isolate the individual effects of temperature, pressure and humidity on PD. These effects were then built into a compensation model. For example the PD is (nearly) directly proportional to flowrate and increases with temperature, since the viscosity of air increases with temperature.

Thus when an instrument is calibrated and/or adjusted, the PD under standard conditions is *transferred* to the instrument, whatever the actual conditions at the time. Therefore a standard in an instrument will only rarely measure its calibrated value and if conditions change to the point of significantly affecting the measured value the instrument should be adjusted, see Section 7.1.2.

5. Checking the validity of measurements

This process comprises the following stages:

- The calibration process
 - Calibration
 - Verification
 - Adjustment
 - Calibration following adjustment
- Checking Instrument performance

5.1 The calibration process

5.1.1 The purpose of calibration

Calibration is an essential part of the overall calibration process and is made to confirm whether the instrument is measuring within the required limits of accuracy. A calibration should be carried out at regular intervals and frequently enough to detect changes in instrument calibration due to the known influences. These influences include:

- The effects of measurement system drift
- The effects of changes in ambient conditions

As a minimum, it is recommended that a calibration check is carried out at least once per day or following any modification or maintenance.

If a calibration shows that an instrument is measuring outside the required limits then it shall be adjusted.

5.1.2 Precautions for calibration

A PD calibration of an instrument can be made by measuring a calibration standard in the instrument. The measured value of the standard will indicate how closely the instrument is measuring to the true value. A decision can then be made as to whether to recalibrate the instrument or not.

When carrying out calibration checking it is necessary to take precaution that the measured value is not corrupted and so avoid unnecessary or corrupt recalibration. In particular:

• The standard should be in thermal equilibrium with the measurement environment.

Note: The apparent value of the standard will change by 0.23 % for every °C of temperature difference between the standard and the measurement air [3]. Ignore any initial readings until the standard reaches equilibrium.

• Avoid handling the standard once it has achieved equilibrium with the measurement air.

Note: Holding a standard for 10 seconds raises its temperature by about 1.5 °C and it can take several minutes to equilibrate. Cotton gloves reduce heat transfer.

- Always include a check for leaks, blockages or other non-linearity within the measurement circuit (refer to the manufacturer's instructions).
- Ensure that the instrument is stable before checking.

Note: Allow sufficient warm-up and settling time after switching on (refer to the manufacturer's instructions).

5.1.3 A recommended process for calibration

To carry out a calibration, proceed as follows:

- Referring to the manufacturer's instructions, place the instrument into the correct mode for calibration.
- Insert the calibration piece into the instrument measurement head, using the appropriate holder for the standard.
- Record the value.

Note: It can take up to 5 minutes for a calibration standard to reach equilibrium with the measurement air.

• Compare the measured value with the assigned value of the standard – if the measured value differs from the assigned value by more than the allowable limit, then the instrument should be adjusted.

Note: The allowable limits should be set by reference to the uncertainty of the measurement and the capability of the process. This is most easily determined by regular use of a Shewhart Chart to plot the measured values for the calibration standards [4, 5]. For additional information on setting limits of uncertainty, please see Section 7.2

If the measured value is within the allowable limit then the instrument does not require adjustment.

5.1.4 Adjustment

The process for adjustment is given in CRM 41 and in ISO 6565. Reference should also be made to the instrument manufacturer's instructions. The essential points for adjustment are given here for convenience:

- Only adjust if there is a good cause:
 - The calibration is out of limits
 - After major work or disturbance to the instrument

Note: Refer to a Shewhart chart. Unnecessary adjustment can lead to a worsening of the measurement variability and hence of the manufacturing process that is being controlled

• Always ensure that the value of the calibration standard is larger than the largest value of sample PD that is to be measured or, when an upper and lower value standard is used, that they cover the full range of subsequent measurements.

Where the range of values of the sample PD is not known in advance, calibrate close to the full scale range of the instrument (80 % or 90 % of full scale is common). This will ensure the best possible accuracy across the complete range of the instrument.

• Observe the same precautions against bias and environmental effects as for calibration

Note: The easiest way to achieve this is to make the adjustment immediately following calibration – this will use the standard in its previously equilibrated state and will require the minimum amount of handling of the standard.

• Carry out a calibration *after* adjustment to confirm that no error has been made and that the instrument is now measuring within the allowable limits.

Note: Always calibrate with at least two standards of different values to ensure correct adjustment.

5.2 Checking instrument performance

This is a means of assessing the dynamic measurement performance of a measuring instrument. It is not necessary to carry out this step for routine calibration but it should be carried out periodically and particularly after any significant service work or repairs to the instrument.

The purpose of this check is to confirm:

- That the instrument is functioning and operating correctly
- The instrument is adjusted correctly for the product being measured and is capable of measuring the product correctly
- The measurement capability of the instrument, in particular it may be used to assess the measurement:
 - Repeatability
 - Instrument-to-Instrument variability

Note: These figures allow an assessment of the allowable limits for calibration.

An instrument cannot be more accurate or precise than its uncertainty of measurement

5.2.1 Measuring repeatability

Repeatability is defined as: "The maximum difference between two repeat measurements of a sample, made on one instrument, by one operator and within a short period of time, which is exceeded on average in not more than one in twenty cases."

Repeatability is normally tested by measuring a series of samples at least twice in quick succession in one instrument without any adjustment of the instrument.

The samples used should be stable and should closely represent the actual samples normally measured on the instrument (i.e. of similar dimensions and PD). Depending upon the type of instrument being tested, suitable samples are:

- Cured filter rods
- Artificial cigarettes
- Commercial cigarettes

Artificial cigarettes are often glass rods of similar dimensions as commercial cigarettes with a number of parallel capillaries along their length and may also contain ventilation holes. They can be obtained from instrument manufacturers.

Precautions should be taken to avoid sample degradation and hence to avoid the PD changing from measurement to measurement. Dependent upon the type of sample, these include:

- Minimising the amount of handling of the samples
- Providing a cushion in the sample collection bin to minimise damage as the samples are dropped through the instrument
- Avoiding significant moisture loss from cigarettes which might otherwise lead to sample shrinkage and embrittlement

It is also important to consult the manufacturer's instructions so that the instrument is configured correctly for the product being tested, especially that the sleeves are selected appropriately for the diameter of the product. Particularly for the measurement of open pressure drop (PDO) and closed pressure drop (PDC) the sleeves need to be positioned correctly to encapsulate the tipping region.

To obtain a truly representative figure, the repeatability test should be carried out in the same location in which the instrument is normally used. This figure will then include any environmental effects that are present.

Note: Before carrying out any testing of repeatability or instrument-to-instrument variability, users should ensure that the instruments concerned have been properly adjusted, as described in Section 7.1.

5.2.2 A process for repeatability testing

- Select a batch of samples a batch of 10 is normally satisfactory
- Number each sample consecutively at the butt end and lay out on suitable sample tray. If the samples are filter rods, this will allow the sample orientation during measurement to be determined.
- Measure each sample in numerical order in the system under test until the batch is complete.
- Collect the samples from the instrument rod bin and replace on the sample tray in numerical order.
- Carry out a repeat measurement of the complete batch in **reverse** numerical order.

Note: Measuring the second run of a repeatability test in the reverse order allows detection of instrument drifts during the batch run which otherwise might not be seen

Repeat measurement can itself have a small effect on measured PD. This can be due to change in moisture content as a result of measurement [6] or physical changes, such as the tamping down of the tobacco column, which slightly raised PD [7].

- Compare the two results for each of the numbered samples in the batch, noting:
 - o the batch mean and standard deviation for each run,
 - the difference in measured value of each sample between run 1 and 2,
 - \circ the difference between the batch means for run 1 and run 2.

• Limits of agreement will depend upon the type of samples and instrument used and the conditions of measurement. However, for guidance, the values should generally be within those given in the following table for instruments that are set up and functioning correctly:

PARAMETER	REPEATABILITY LIMITS (RUN 1 – RUN 2) (10 ROD BATCH)			
	INDIVIDUALS		BATCH MEAN	
Sample type / Parameter	Cigarette	Artificial cigarette or Filter Rod	Cigarette	Artificial cigarette or Filter Rod
PD (filters @ 600 mmWG level)	-	3 mmWG	-	2 mmWG
PD (cigarettes @ 100 mmWG level)	3 mmWG	2 mmWG	2 mmWG	1 mmWG

5.2.3 Measuring instrument-to-instrument variability

Note: - The term "reproducibility" has slightly different definitions in different standards and hence the term "instrument-to-instrument variability" has been chosen rather than "reproducibility" to avoid any such discrepancies and to clarify the intended purpose of the tests.

The CORESTA Sub-Group PTM (Physical Test Methods) conducts annual collaborative studies to monitor the repeatability and reproducibility of physical measurements of filters and cigarettes and it is recommended to refer to these for a wider context to inter-laboratory and inter-instrument comparisons [8].

Tests of instrument-to-instrument variability are a means of assessing the maximum difference to be exceeded not more than once in 20 cases between the measured values of a sample when measured on two different instruments. It can be influenced by many different factors, including the instrument repeatability, differences between the calibration standards used to adjust each instrument and local differences in operator procedure and technique.

Instrument-to-instrument variability testing is carried out in a similar way to repeatability testing, except that the two measurements are made on different instruments and may be carried out over an extended period of time.

In addition, there may be several measurement runs, each on a different instrument. This allows testing across a population of instruments within an organisation and allows comparison between any one instrument and the mean of all the instruments. This can be used to determine any biases between the instruments.

Samples for instrument-to-instrument variability testing can be the same as those for repeatability testing. However, cigarettes are not normally robust enough to be used and samples are generally confined to filter rods or artificial cigarettes.

Further, it may not be possible or desirable to circulate one sample batch and measure it on all the instruments. In such cases, the sample batches for each instrument should be randomly drawn from a larger population of samples and a check should be made to ensure that the chosen batch size allows the mean and standard deviation of the batch to match that of the population. Where the population standard deviation is large, it may be necessary to increase the sample batch size to achieve a properly representative batch mean and standard deviation.

The limits of agreement for instrument-to-instrument variability testing are again dependent upon the types of instrument and samples used and upon the measurement conditions. For guidance, the values should generally be within those given in the following table for instruments that are set up and functioning correctly:

INSTRUMENT-TO-INSTRUMENT LIMITS (INSTRUMENT 1 – INSTRUMENT 2) (10 ROD BATCH OF CURED FILTER RODS OR ARTIFICIAL CIGARETTES)			
	INDIVIDUALS BATCH MEAN		IMEAN
PARAMETER	INSTRUMENT - INSTRUMENT	INSTRUMENT - INSTRUMENT	INSTRUMENT – OVERALL MEAN
PD (filters @ 600 mmWG level)	6 mmWG	5 mmWG	4 mmWG
PD (artificial cigarettes @ 100 mmWG level)	4 mmWG	3 mmWG	2 mmWG

Note: - The actual instrument-to-instrument variability cannot be better than the repeatability of the individual instruments and will also include any differences between the standards used to adjust each instrument. These values will depend upon local conditions and hence the limits tabulated above are for guidance only and do not imply definitive limits.

6. Making PD measurements

The exact procedure for measuring the PD of samples will depend not only on the type of sample to be measured but also on the type of instrument used. There are a number of points, however, that need to be checked for all PD measurements:

- The instrument used should be suitable for the type of product to be measured. Generally, the type of sample holder and measurement head device is different for filter rods and cigarettes due to their different construction and measurement requirements.
 - Filter rods should be completely encapsulated in an impermeable sleeve to prevent air 'short-circuiting' down the outside of the filter
 - Cigarettes require that the butt end of the sample only is encapsulated by the sample holder [2].
- The instrument should be adjusted correctly for the sample to be measured with reference to the manufacturer's instructions.
 - Ventilated cigarettes require that the centre seal is located correctly so that values of PDO and PDC are correctly measured (particularly that the ventilation inlets are not blocked).

- For both cigarettes and filter rods, the correct size of seals/sleeve should be fitted to avoid leakage or crushing of the sample. As a guide, the following sizes are recommended:
 - Cigarettes:

Consult instrument instructions; typically several sleeve sizes are available to cover the full diameter range of products that includes ultra/micro slims (~4.7.mm), super slims (~5.2 mm), slims (~7 mm), conventional king size (~7.9 mm) and magnums (up to ~9 mm).

• Cured filter rod:

Sleeve inside diameter = (rod diameter - 0.1 mm) to (rod diameter - 1.0 mm)

• Uncured filter rods:

Sleeve inside diameter = (rod diameter -0.1 mm) to (rod diameter -0.6 mm)

- Samples should be in equilibrium with the measurement environment to avoid errors due to moisture and temperature changes within the sample during the measurement.
- Samples should be handled carefully and as little as possible to avoid damage and changes of sample moisture and temperature.
- Calibration should be made frequently enough to avoid errors in measurement due to PD changes with ambient conditions. The changes in measured PD value with changes in ambient conditions are given in [3]. For guidance, calibration is recommended if:
 - Ambient temperature has changed by more than 2 °C
 - Ambient moisture level has changed by more than 20 % RH
 - Ambient pressure has changed by more than 50 mbar

7. Glossary and abbreviations	7.	Glossary	and	abbreviations
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PD Pressure drop	
PDO The pressure drop of a cigarette with the filter ventilation open	
PDC The pressure drop of a cigarette with the filter ventilation closed	
Draw resistance	(Also resistance to draw, RTD) The pressure drop of a cigarette
°C	Degrees Celsius
RH	Relative humidity
ISO	International Standards Organisation
ISO TC 126	ISO technical committee for standardisation of terminology and test methods for unmanufactured tobacco, all types of tobacco products, materials used for manufacturing tobacco products and tobacco smoke including environmental tobacco smoke aspects
CORESTA	Cooperation Centre for Scientific Research Relative to Tobacco
CRM	CORESTA Recommended Method
	A test piece with the similar physical properties to a cigarette but constructed so that its primary physical parameters (i.e. weight, length, circumference, pressure drop and ventilation) are both stable and repeatable. There are generally two types of such a device: -
Artificial Cigarette	1. Articles made from plastics or light metals with the appropriate dimensions and primary physical parameter to match those found in "real" cigarettes
	2. Cigarettes in which the tobacco column has been replaced with a piece of filter rod material of suitable pressure drop.
	Generally, articles of the second type are preferred since they more closely mimic the properties of actual cigarettes.
Shewhart Chart	A statistical tool in the form of a control chart intended to assess the nature of variation in a process
Uncured filter rod	A filter soon after manufacturing, before the plasticiser has fully cured and hence before the filter rod hardness has achieved its ultimate value

8. References

- [1] CORESTA Recommended Method N° 41 (available at <u>www.coresta.org</u>)
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