Technical Report

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Physical Properties of Special Filters

Sub-Group Physical Test Methods

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1. Introduction

The CORESTA subgroup PTM decided in 2007 to set up a project team to assess and evaluate specific issues related to the pressure drop measurement of special filters.
Sub-Group members raised several concerns about possible differences of pressure drop measurement results between mono-acetate and special (e.g. charcoal, flavour, multi-segment, etc) filters because such filters were not included in the development process of the current CRM and ISO standard. There was also mutual agreement that the group has to focus on the most common types of special filters.
A pre-study with a limited number of laboratories was conducted early 2009 and statistical results presented at the meeting in April 2009. Conclusions were discussed at the meeting in November 2009 and it was then decided to provide the results as a technical report. Currently Sub-Group members see no necessity to conduct a bigger study.

1.1. Objectives

General objectives:
Collect available information about problems and concerns related to the pressure drop (PD) measurement of special filters.
Conduct a study on the most common types of special filters if appropriate.

The considerations as listed in Section 1.4 were discussed at the PTM meeting in 2008 and it was decided to conduct a pre-study with a restricted number of participants (experienced manufacturers or laboratories).

Pre-study objectives:
Evaluate pressure drop mean values and variability for a set of selected special filter rods.
Compare results with existing data for mono-acetate filters.
Prepare a technical report on the results which includes recommendations for a best practice of PD measurements of special filters.

1.2. Definitions and references

PD measurement refers to the existing Coresta RM 41 and ISO standard 6565. Additional guidance is available from Coresta Guide No. 4.
All references made in these documents, e.g. ISO 3402 ‘Atmosphere for conditioning and testing’, also apply to the work presented in this study.

Naming conventions for this study report:
“Filter” means filter rod. All data in this report refer to the measurement of entire filter rods.

Filter plug and filter tip: A filter rod commonly delivers after cutting 2 to 8 filter plugs either as base plugs in multi-segment filter rod makers or as the “final product” for cigarettes, cigars, hand-rolled tobacco products or pipes.
If the filter plug is the final single filter for a smoking article, e.g. cigarette, it is also named filter tip.

Filter Segment: If the final filter plug consists of more than one part these parts are named segments in this report.
Special filter means any filter rod other than a mono-acetate filter rod.

The quantitative results from this study are limited to the filter rods investigated (see sample list in 2.2) within the scope. General conclusions can be applied to similar filter designs.

1.3. **Scope and classification of special filters**

The focus was on the most common special filters. A monoacetate filter was included because it is widely used as a base rod for mouth-end plugs and also for comparison reasons.

1. Base rods
   a) mono-acetate rods
   b) acetate rods loaded with filter additives, preferably carbon granules
   c) filter rods with (a high level) of liquid application; e.g. menthol,
   d) rods made of paper or paper-like materials (because of moisture influence).

The base rode can also be the final rod, e.g. mono-paper filter, flavour filter or filter rods for production lines with segment combination in the filter attachment part of the cigarette maker.

2. Multi-segment filter rods
   a) Dalmatian type; dual or triple segment filter rods; at least one segment is loaded with (carbon) granules; filter material can be either acetate or paper;
   b) Cavity filters; at least one segment is either an empty cavity or a cavity filled with (preferably carbon) granules.

Filter plugs with more than 3 segments are technically possible, but they are not in the scope of this study. Theoretically, PD variation ought to increase if the number of segments increases because e.g. the number cut surfaces increases.

3. Standard & slim-line product ranges
   a) Slim-line dual filter rods with granules (carbon)

*PD and PD variation is a function of the rod circumference (diameter), e*specially *for rods with high granule loading. The shape and size of particles may also be relevant .*

Samples from categories 1.a, b, c, 2.a, b and 3.a were investigated in this study. Base rod 1.d was investigated as a dual filter segment and additionally a plastic rod that was expected to be mostly inert against moisture and circumference changes was included. (See also Table 1 in Section 2.2.1)

Beside these most common types of special filter many other designs exist:
- Filters with recess, bore-shaped or other ‘zero-PD’ segments
  Logically, such segments will not influence PD variability.
- Filters with other additives than active charcoal
  PD variability ought to be similar if amount, particle size and distribution are comparable to the investigated carbon filters.

1.4. **Issues of concern**

1.4.1 Contamination

It still seems to be unclear if frequent measurement of carbon filters or flavour filters lead to a higher risk of contamination of the instrument. There has also been some discussion about the significance of the flow direction (pressure or vacuum). The team members have found no evidence of contamination problems. Routine instrument maintenance should be sufficient to minimise any potential issue.

1.4.2 Product variability

Undoubtedly, the PD variation of special filters (especially multi-segment filter rods) is equal or higher compared with mono-acetate filter rods. A simple calculation from base rod variation by using the law of error propagation is usually insufficient because the production process (cutting, pressing) plays a significant role.
Variation depends on two main factors:
Complexity of the filter specification (e.g. number and type of segments) and production quality which again strongly depends on the type of maker (principle of plug combination), plug length variation, plug positioning, etc.

1.4.3 Cavity filters

Horizontal or vertical measurement could lead to different results. The influence of flow direction (vacuum or pressure) is still unclear.

1.4.4 Sensitivity to moisture and temperature

The effect of ambient conditions on acetate filters have been evaluated elsewhere, e.g. study report by Celanese: "Effects of ambient conditions on filter rod properties". The effect on PD is quite small, but clearly observable for weight and circumference.
Some observations indicate that pressure drop of paper filters can increase with water content because of swelling effects. For results from an ITG in-house study see appendix B. The influence of temperature was seen as less important because it can easily be controlled in laboratory environments, and therefore it was not investigated in this study.

1.4.5 Other issues

a) Flavour filters

Some effects on diameter and PD for highly loaded flavour filters have been reported. However proprietary design information allows these issues to be combated.
Therefore, we recommend handling flavoured filters separately.

b) Vacuum or pressure measurement, flow direction

Vacuum or pressure certainly gives different answers which may not be interchangeable. It is believed that some original ISO documents were principles of measurement rather than the precise standards we have today. One of the first of these gave a principle of measurement for PD with a flow of 17.5 ml/s using conditioned air (22 and 60%RH) and the exit pressure from the object being tested as atmospheric. This caused the current situation of a mix of vacuum and pressure instruments as vacuum allows the use of conditioned air drawn into the test piece but the exit pressure is atmospheric minus the PD of the test piece. A pressure instrument uses compressed air that is not conditioned but the exit pressure is atmospheric.

2. Study results

2.1. Summary

Because of the restricted number of participants (6 laboratories with 7 instruments) more qualitative rather than quantitative results are represented here. Special filters exist in such a wide variety of different designs and physical properties that this report can only give some guidance for the appropriate treatment of special filters. The aim is to raise awareness of certain issues, e.g. conditioning and influence of moisture, to avoid problems if laboratories compare PD measurement results with each other.

Main conclusions

PD variability:
If filter samples are ranked by SD they give the following order:
Plastic tube < Mono-Acetate < base rod, carbon dual, cavity, paper dual, flavour < slims dual
This is in line with common experience. The high variability for slims was also observed in studies on mono-acetate slims filter. Because the repeatability within each lab looks acceptable differences in handling and treatment by the instruments must play an important role. PTM will continue to investigate best practise of slim filters physical measurements.

Contamination
No specific problems were reported. The use of vacuum instruments is appropriate.

Flow direction – vacuum or pressure
One lab took part with a pressure instrument. Results indicate that PD values are lower in comparison to vacuum devices.

Conditioning - Moisture
One lab started with unconditioned samples
→ lower weights and circumferences were observed, except for sample E (plastic tube)
→ but no effect on PD was found.
As expected, repeated conditioning of flavour filters shows a trend towards lower weights.

2.2. Study protocol

2.2.1 Sample description
All samples were provided by Filtrona FTC, Jarrow UK, except sample E (plastic tube) that was made by Filtrona Germany and provided by ITG. Sample E was included because a very low weight, circumference and PD variability was expected. This can provide meaningful information about instrument versus product variability. Samples and physical properties are listed in Table 1. Table 2 outlines the measurement scheme and the instruments that were used by the participants are listed in Table 3. For the recommended positioning for circumference (diameter) measurements see appendix A.

Table 1: Sample Description

Sample	A	B	C	D	E	F	G	H
Filter type	Mono-acetate	Carbon base	Carbon dual	Carbon cavity	Plastic rod	Slim dual	Paper dual	Flavour filter
Manufacture date	21.10.08	22.10.08	29.10.08	29.10.08	Jan 09	29.10.08	19.01.09	02.12.08
Rod length (mm)	108	96	126	120	82.5	108	100	120
Rod circ. (mm)	24.0	24.0	24.5	24.5	22.6	16.8	24.1	24.2
Plug wrap	porous	porous	porous	porous	non-por.	porous	standard	porous
Target PD (mmWG)	410	495	568	380	155	493	408	360
Tip spec	base rod n. a.	base rod n. a.	9AC+12AA	7Ac+6Cav +7AA	mono rod	11Ac+ 16AA	15Ac+ 10paper	mono rod
Tip length (mm)			21	20	not applicable	27	25	20
Tips per rod			6	6		4	4	6
Additive type	nil	carbon	carbon	carbon		carbon	nil	menthol
Additive (mg/rod)	nil	384	288	780 *)		28	nil	30
Base rod 1	base rods n. a.		sample A	acetate		acetate	acetate	n. a.
Base rod 2			sample B	AA		AA	paper	
Plasticiser	triacetin	triacetin	triacetin	triacetin		triacetin	triacetin	triacetin

AA = carbon on tow (Dalmatian rods)
*) Total carbon approx. 600 mg in the cavities & 180 mg in AA

Parameters to be measured:
- Pressure drop, diameter and rod weight after conditioning.
  Optionally: the same parameters before conditioning

To be recorded: date, time, RH, temperature and conditioning time

Timeline: Samples were manufactured in December 08. Measurements and data evaluation were carried out in Q1 2009.

Table 2: Measurement Scheme

	Day1	**Day2**	**Day3**
Batch1 = #1	#1 sample 1-8 Measurement No.1 Recording sheet **<u>1 and 2</u>**	#1 sample 1-8 Measurement No.2 Recording sheet 2	#1 sample 1-8 Measurement No.3 Recording sheet 2
Batch2 = #2		#2 sample 1-8 Measurement No.2 Recording sheet 1	
Batch3 = #3			#3 sample 1-8 Measurement No.3 Recording sheet 1
Individual measurements per day	1*8*10 = **80 rods**	2*8*10 = **160 rods**	2*8*10 = **160 rods**
Individual measurements in total	5*8*10= **400 rods**		

Instruments
Six laboratories with 7 instruments participated.

Table 3: Participating laboratories, lab codes and instruments

Laboratory codes for data & evaluation section	Instrument
L1	OMI+
L2	QTM
L3	QTM
L4	Sodimat
L5P	modified instrument for pressure measurements
L5	Sodimat
L6	Sodimax

2.3. Data & evaluation

Table 4 shows mean values and SDs for weight, circumference and pressure drop. In general sample E gives the lowest variation. This demonstrates that such samples are meaningful to investigate instrument against product variability.
The carbon cavity filter is highest in weight variation. Probably this is due to carbon loading variability during manufacturing.

Charcoal absorbs moisture over a longer period of time. So it is probable that there are corresponding changes in the rod parameters. Therefore not only weight but also diameter (changes due to moisture content) and PD (because of changes in diameter) have to be registered.
Normally in the industry two kinds of measurements exist: fresh rods measured after their production and rods sent to a lab and measured after a certain period of time. In this period normally the diameter increases and PD in parallel decreases.
As a consequence of this all relevant parameters (e.g. age, conditioning time, ambient conditions during measurement) have to be recorded in a study/ring trial.

Both dual filter samples are highest in circumference variability. This indicates that combining of segments with different physical properties, e.g. hardness, usually increase circumference variation.
Differences in sample treatment by operators and by the instruments can be a possible reason for the very high PD standard deviation of slim dual filters that cannot be explained by product variation alone. Within lab variability for this type of special filter is on a normal level.

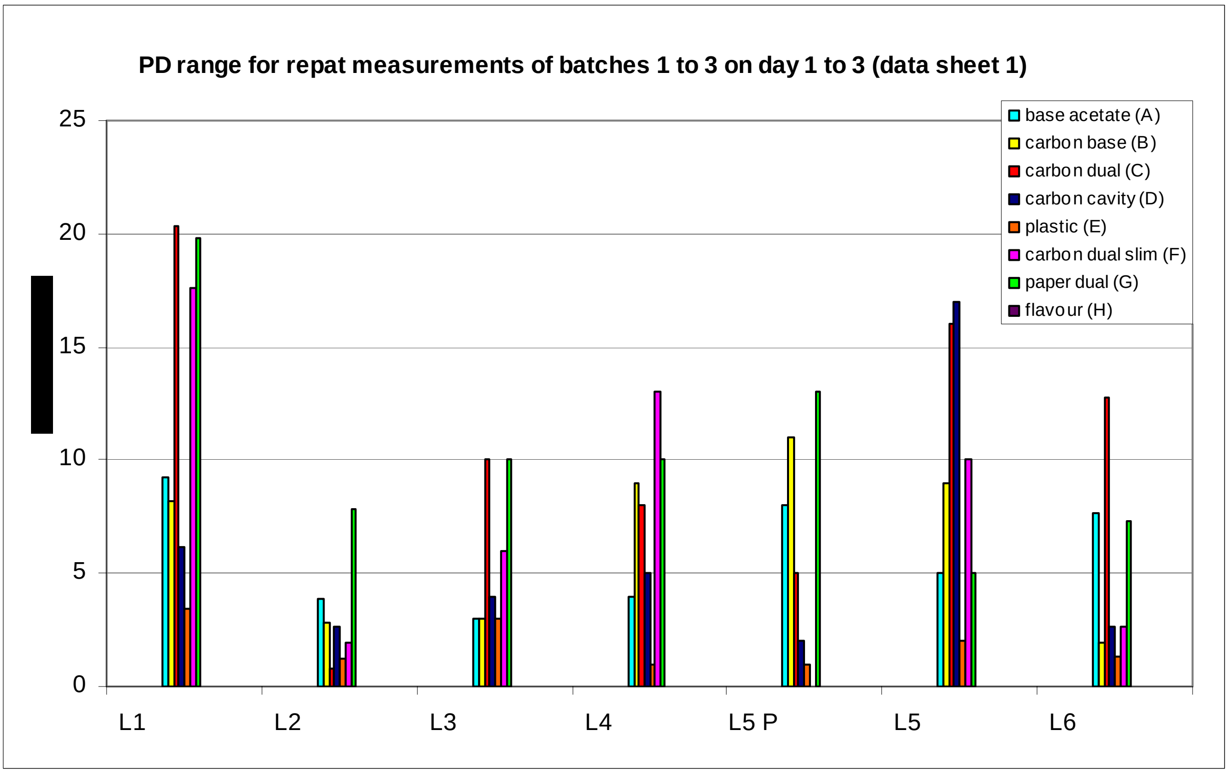
Table 4: Summary of results (data sheet 1)

Variable	Sample	n	Mean value	Standard Deviation
Individual weight [mg]	A - acetate	21	673.0	5.88
	B - carbon base	21	1038.2	31.40
	C - carbon dual	21	1204.8	27.50
	D - carbon cavity	21	1593.6	65.20
	E - plastic rod	21	631.2	2.06
	F - slim dual	18	409.4	4.29
	G - paper dual	21	754.7	5.45
	H - flavour	21	695.7	8.34
Circumference [mm]	A - acetate	21	23.96	0.034
	B - carbon base	21	24.05	0.030
	C - carbon dual	21	24.54	0.039
	D - carbon cavity	21	24.54	0.032
	E - plastic rod	21	22.59	0.025
	F - slim dual	18	17.09	0.068
	G - paper dual	21	24.09	0.062
	H - flavour	21	24.18	0.042
Pressure drop [mmWG]	A - acetate	21	415.2	6.73
	B - carbon base	20	477.9	9.25
	C - carbon dual	21	553.7	9.68
	D - carbon cavity	21	398.1	10.74
	E - plastic rod	21	156.4	4.64
	F - slim dual	18	420.8	135.30
	G - paper dual	21	412.8	11.18
	H - flavour	21	347.4	12.84

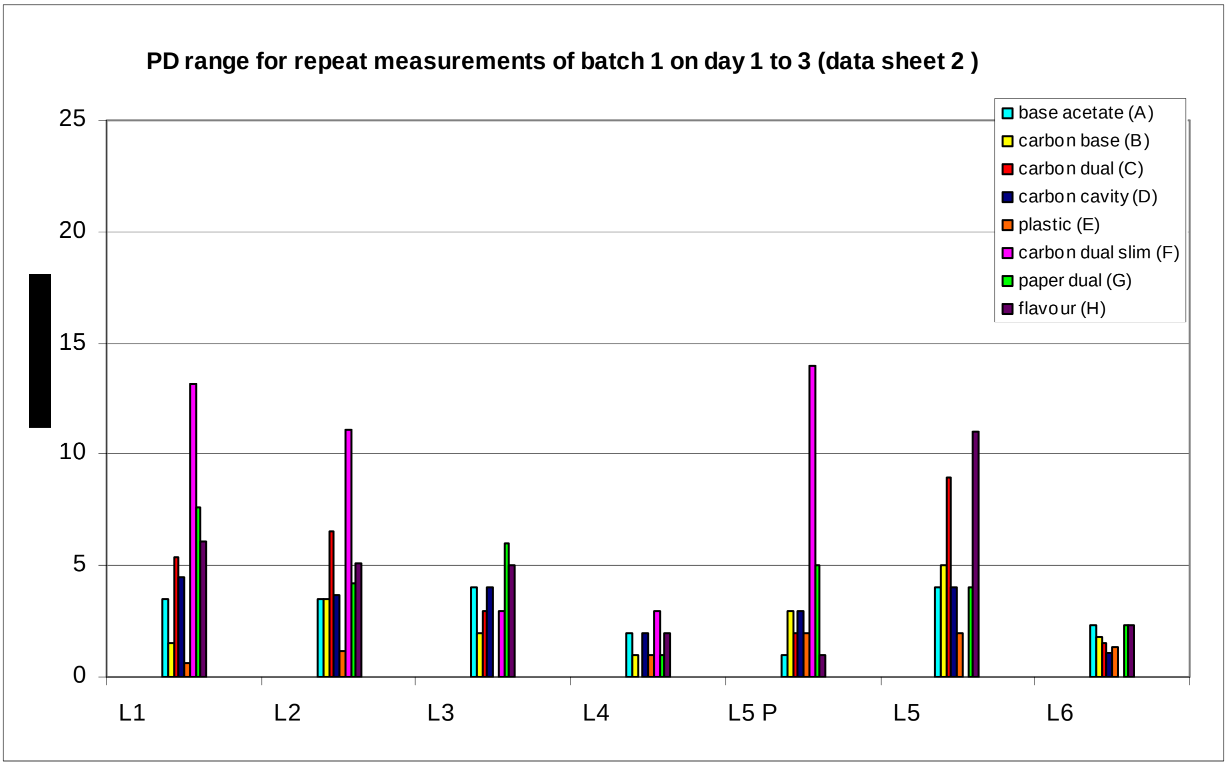
Repeatability within laboratories

The next two graphs visualise the differences between measurements of samples from the same batch on 3 consecutive days against repeat measurements of an identical sample in the same manner. As expected, repetition of identical samples results in lower variation, but again slim dual filters have the highest variability.

Graph 1: PD ranges by lab – datasheet 1

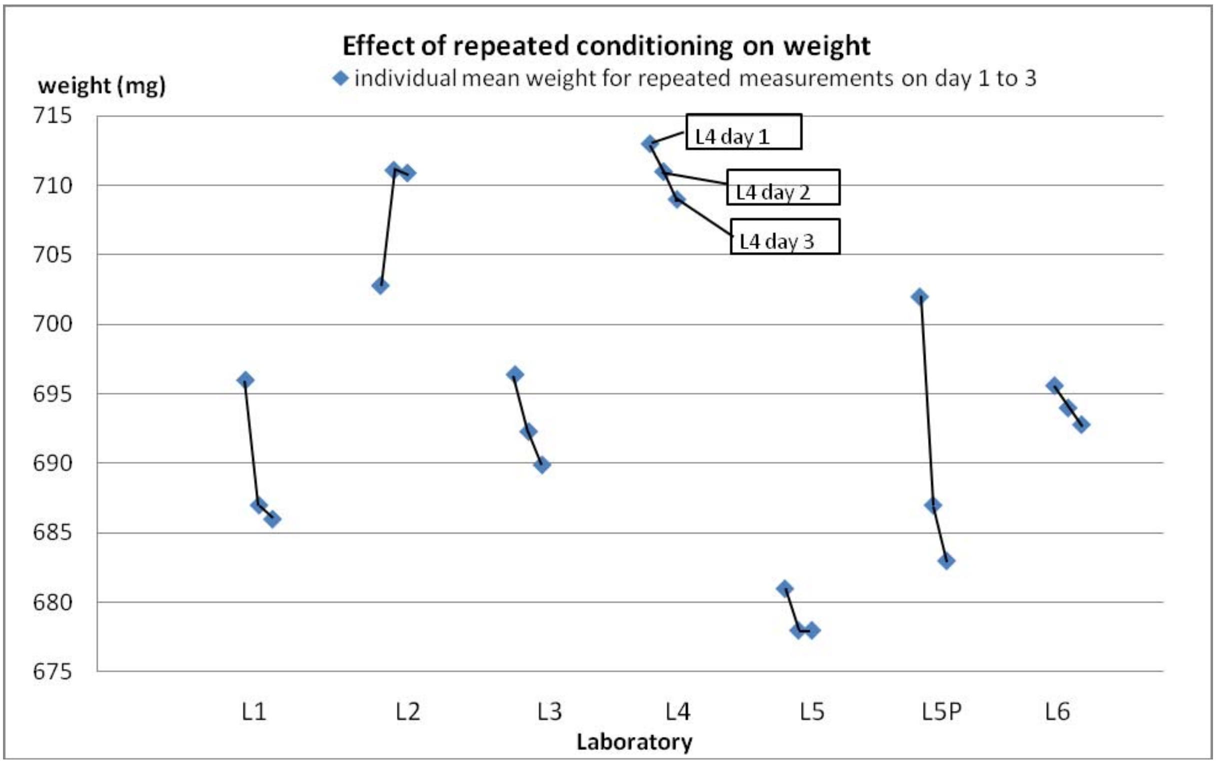


Graph 2: PD ranges by lab – datasheet 2



Flavour Filters
One of the specific issues investigated was the influence of conditioning on flavour content of flavour filter. In general a small loss of weight can be observed when flavour filters were conditioned repeatedly (1st data point for lab 2 was measured without conditioning). PD and circumference values do not show a specific trend.

Graph 3: Flavour filters (sample H), repeated measurement of batch 1

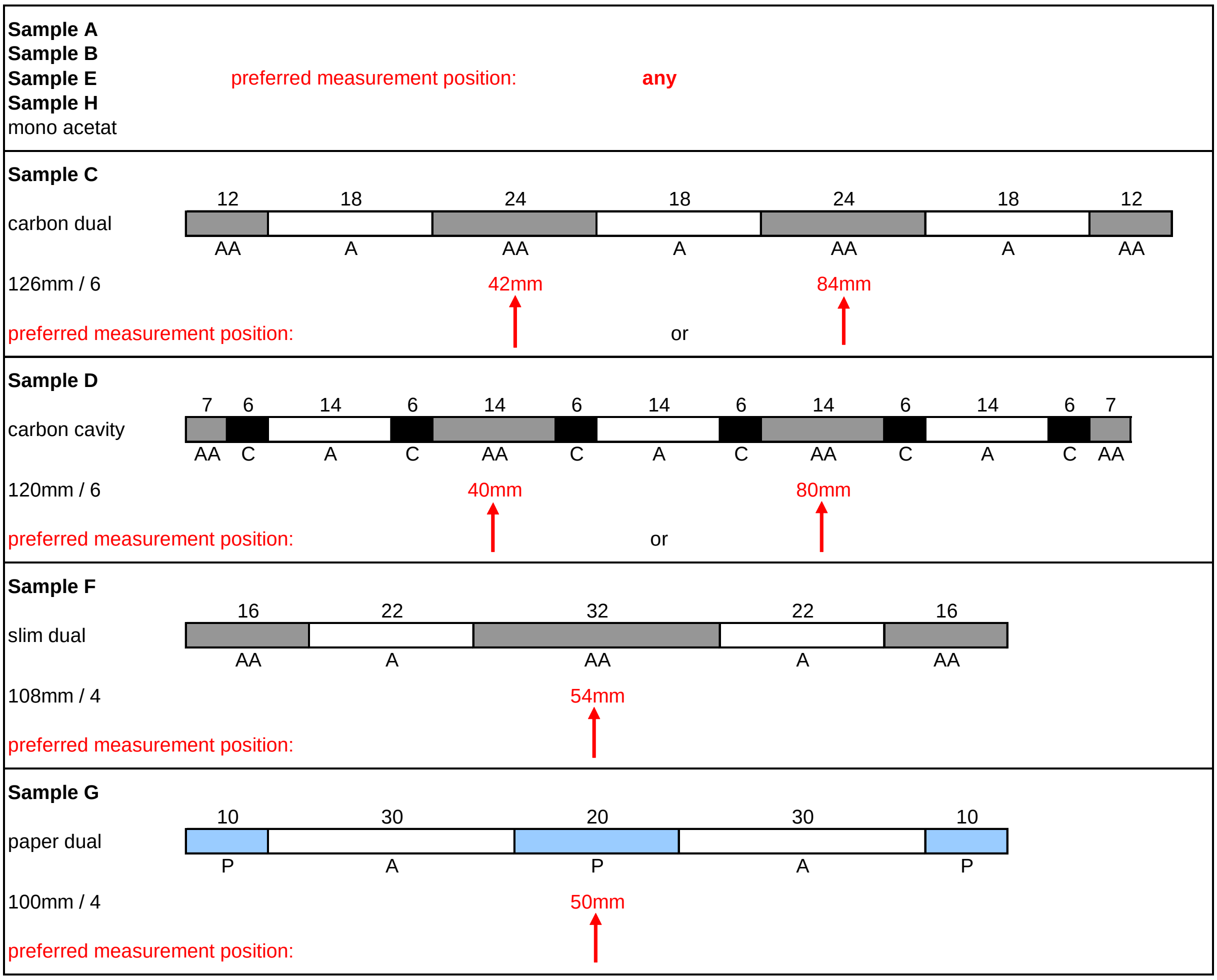


Effect of moisture on paper filters
We refer to previous work that is attached in appendix B.

3. **Appendices**

Appendix A: Preferred position for circumference measurements
Appendix B: Effect of moisture on PD of acetate-paper dual filters

Appendix A: Preferred position for circumference measurements



Appendix B: Effect of moisture on PD of acetate-paper dual filters

Ref: R. Ortlepp, Reemtsma, presented at 36. Tabakkolloquium, Lübeck 1994

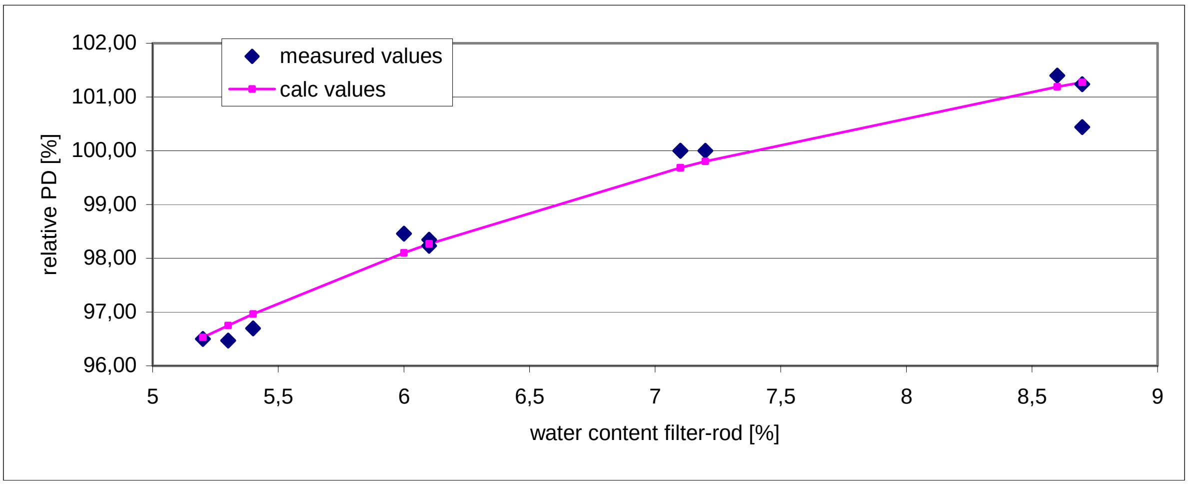
The pressure drop of paper filters depends on the moisture content. A significant swelling effect in comparison to acetate filters was observed that led to an irreversible pressure drop increase. So, conditioning before and during measurement is important.

Table B1: Study results

	Temp.	RH	PD meas.	relative PD	Water-content	%calc	PD calc. 1)
	°C	%	mmWG	%	%	%	mmWG
Dual-filter1 Ac-paper	20	40	468	96.69	5.4	96.965	469
	20	50	476	98.35	6.1	98.268	476
	20	60	484	100.00	7.2	99.804	483
	20	70	490	101.24	8.7	101.27	490
Dual-filter2 Ac-paper	20	40	437	96.47	5.3	96.751	438
	20	50	445	98.23	6.1	98.268	445
	20	60	453	100.00	7.1	99.684	452
	20	70	455	100.44	8.7	101.27	459
base- filter paper	20	40	689	96.50	5.2	96.528	689
	20	50	703	98.46	6	98.1	700
	20	60	714	100.00	7.1	99.684	712
	20	70	724	101.40	8.6	101.19	722

1) function used: y=a/x+b   y=61.31267 / %WG+108.31916

Graph B1: Measured and calculated relative PD values against water content



Graph B2: Comparison of measured and calculated PD