TECHNICAL REPORT

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2021-08

Evaluation of physical parameters of filter paper substrates for the determination of the ignition propensity of cigarettes

Évaluation des paramètres physiques des substrats de papier-filtre pour déterminer le potentiel incendiaire des cigarettes

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The ISO 12863 standard test method for cigarette ignition propensity specifies Whatman No. 2 (or an equivalent paper) as a suitable substrate. An expert group was established within ISO/TC 92/SC 1 in 2012 with the objective of defining a set of paper parameters that a substrate paper ought to meet. When testing cigarettes according to the ISO 12863 standard, it is recommended that the paper produce test results that:

— are statistically indistinguishable from the currently available substrate paper; and

— that provide no degradation of repeatability and reproducibility.

The study presented in this document used Whatman No. 2 filter papers that were produced after 2011, when Whatman moved the production facility from UK to China[1]. An in-depth analysis of the effect of this change on ignition propensity testing results was published in Reference [1].

Evaluation of physical parameters of filter paper substrates for the determination of the ignition propensity of cigarettes

1 Scope

This document recollects and evaluates physical parameters of filter paper substrates for the determination of ignition propensity of cigarettes according to ISO 12863.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 12863, *Standard test method for assessing the ignition propensity of cigarettes*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 12863 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Parameters to specify filter papers for use as substrates in ignition propensity testing

The expert group reviewed previous activities related to cigarette ignition propensity standard development and the main scientific literature on substrate paper properties. Experts from the filter paper industry also shared commonly used filter paper parameters used for quality control and discussed the specifications of Whatman No. 2 outlined in ISO 12863.

It was agreed by the experts that paper weight alone is unlikely to represent sufficient specification.

A draft set of physical parameters was defined, comprising:

— basis weight (ISO 536);

— thickness (ISO 534);

— paper weight (ISO 12863);

— surface roughness (ISO 8791-2);

— air permeability (ISO 5636-5);

— ingredients: 100 % cotton fibre free of additives.

As detailed research into the influence of each filter paper parameter on the results of ignition propensity testing would be beyond the capabilities of the expert group, it was decided to proceed with a conservative approach, keeping all substrate paper physical parameters. Although this approach

could lead to the over-specification of the substrate, experts from filter paper manufacturers agreed that such a filter paper could be produced on an industrial scale.

5 Establishing acceptable ranges for filter paper physical parameters

Correlation testing between physical filter paper parameters and ignition propensity results would require significant effort and ought therefore to be limited to a minimum. Therefore, initial experiments were designed in order to obtain initial insights with a feasible amount of effort.

The first collaborative study used Whatman No. 2 substrates collected from LIP laboratories. The aims of this study were:

— to compare test methods between laboratories;

— to establish a range for the critical substrate parameters based on the measurements on Whatman No. 2 substrates used in LIP laboratories;

— to compare the range for critical parameters between Whatman No. 2 substrates and substrates from other suppliers.

For this study, 9 different Whatman No. 2 batches were sourced from 3 LIP testing laboratories.

Four alternative substrate manufacturers provided their own substrates and took part in the testing:

— delfortgroup

— MACHEREY-NAGEL

— Munktell & Filtrak

— Sartorius-Stedim

In this study, the mean ±3 standard deviation ranges for the Whatman papers were:

— basis weight: 91 gsm to 102 gsm

— thickness: 177 µm to 215 µm

— air permeability: 12 s to 26 s

— surface roughness (wire side): 763 ml/min to 1 181 ml/min

— surface roughness (felt side): 345 ml/min to 853 ml/min

A second collaborative study was performed using three substrate papers from an earlier NIST study[1],[2] on ignition propensity testing. The aim was to use these data as a specification for other substrates.

The papers from the NIST-study[1],[2] were from the following manufacturers:

— Ahlstrom

— Tela-Kimberly (Swiss Quality)

— Tervakoski (delfortgroup)

The following laboratories participated in this study:

— Ahlstrom Germany GmbH

— Arista Laboratories, Inc.

— BAT Germany

— GE Healthcare

— Hahnemühle FineArt GmbH

— ISEGA Forschungs- und Untersuchungsgesellschaft mbH

— MACHEREY-NAGEL GmbH & Co. KG

— Papierfabrik Wattens GmbH & Co. KG

— R.J. Reynolds Tobacco Company

— SWM International

Table 1 shows the proposed specifications for ignition propensity substrate paper that were selected based on the statistical evaluation of the results.

Table 1 — Proposed specifications for ignition propensity substrate paper

Critical substrate parameter	Proposed specification range	Test Method
Weight (15 conditioned sheets)	(26,1 ± 0,5) g	ISO 12863
Weight (15 dried sheets)	(24,7 ± 0,5) g	ISO 536
Air permeability (Gurley)	(20 ± 8) s	ISO 5636-5[a]
Roughness[b]	(2 000 ± 500) ml/min	ISO 8791-2[b]
Thickness	(190 ± 20) µm	ISO 534[c]
[a] 5 oz cylinder, 1 inch^2 orifice plate, 100 ml air volume.		
[b] Measurement pressure: 1,47 kPa.		
[c] Measurement pressure: 53 kPa. Area of clamping zone: 200 mm^2.		

6 Correlation of paper physical parameters with ignition propensity testing

No correlation tests between ignition propensity results and paper physical parameters had previously been performed. However, such tests are necessary in order to confirm the set of parameters. In order to validate the proposed set of substrate specifications, a further study was therefore designed. The aims were:

— to carry out ignition propensity testing with the different filter paper substrates;

— to check whether the different substrates (when manufactured to the same specifications) provide statistically indistinguishable results in ignition propensity (IP) testing;

— to enable the use of different filter paper substrates that meet the criteria specified by the expert group.

Testing was carried out with the help of CORESTA and the voluntary participation of 14 laboratories:

— British American Tobacco Germany

— British American Tobacco Brazil

— British American Tobacco Indonesia

— British American Tobacco Korea

— British American Tobacco Poland

— Japan Tobacco Inc., Japan

— Japan Tobacco International, Germany

— KT&G Corp., Korea

— Global Laboratory Services Inc., USA

— R.J. Reynolds Tobacco Company, USA

— Landewyck Tobacco S.A., Luxembourg

— Delfort Group, Austria

— Monte Paz S.A. Uruguay

— Altria Client Services USA

— Philp Morris International, Poland

— Philp Morris International, Germany

— Imperial Tobacco/Reemtsma, Germany

— ITG Brands, USA

The three paper manufacturers (MACHEREY-NAGEL, Delfort Group and Hahnemuehle) provided 120 000 filter paper circles each as substrates for the CORESTA study on ignition propensity testing[3]. The paper manufacturer Whatman did not provide samples. Rather, the participating laboratories used the available papers in their lab. In order to have sufficient material for the subsequent testing of the physical parameters, the participating CORESTA laboratories sent seven packs (700 filter circles) of the Whatman lot they used for ignition propensity testing.

The statistical evaluation showed no significant difference in ignition propensity results between the substrates from Whatman and those from the other paper manufacturers.

In the last step, a collaborative study on the physical parameters of the substrates used in the IP-study was performed with the aim of confirming the ranges previously agreed upon. For this test, the papers from of the same lots as for CORESTA testing[3] were used.

The following labs participated in the physical parameter testing:

— Hahnemuehle

— Delfort Group

— ISEGA

— IW Tremont

— R.J. Reynolds Tobacco

— Gebr. Hoffsümmer

— Enthalpy Analytical

— BAT Bayreuth

— MACHEREY-NAGEL

The study confirmed that physical parameter values for the substrates were within the specified ranges.

7 Report — substrate papers physical parameter testing

7.1 Substrate specifications

The aim of this analysis was to gain an understanding of the different substrates measured (Whatman, Tervakoski, Hahnemuehle and Macherey-Nagel) across the different laboratories. The two main questions were:

— whether there were significant differences between the substrates (and laboratories) for each of the physical parameters (noting that basis weight was not specified, only measured); and

— how well the substrates matched the target specifications and tolerances.

In order to answer these questions, the data provided were first plotted with the ranges. For some of the target specifications the standard deviations were very large, which resulted in a very wide range of seemingly acceptable values. The analysis in this subclause demonstrates whether these values are significantly different from the specifications and whether these parameters are in control.

The results from this analysis can be found in 7.2 and 7.3.

The mean ±3 standard deviation plots based on the specifications (7.2) showed that virtually every sample tested could be considered acceptable. The only group of samples that could not be considered acceptable based on this analysis were most samples tested for dried weight from lab. 9. From these plots, some outliers can be observed. Therefore, before interpreting the results of the ANOVA analyses (7.3, to check the differences between the substrates and labs), the residual plots were checked. Based on these residual plots, further outliers were removed. The new data set (with these outliers removed) was used to perform further statistical analysis.

Control charts (7.4) of the different parameters were created. These indicated which laboratories were outliers. It is to be noted that the control chart for air permeability differs from the other control charts as there were more than 100 observations per laboratory; it was therefore categorized by both laboratory and substrate. This resulted in much tighter control limits.

The following points were removed as outliers:

— Weight (15 cond. sheets) — last Whatman sample from Lab 9 (29,481 6 g)

— Weight (15 dried sheets) — second Hahnemuehle sample from lab 8 (27,514 0 g)

— Basis Weight — Second Macherey-Nagel sample from lab 4 (90 gsm)

— Thickness — Thirteenth Macherey-Nagel sample from Lab 2 (223 µm)

For conditioned weight, no laboratories were identified as outliers and control limits were [25,90; 26,44], with a mean of 26,17. For dried weight, lab. 9 was a clear outlier (sample 8 on the x-axis). The control charts showed that lab. 7 was not within the control limits, based on the control limits of [24,93; 25,46] with a mean of 25,2. For Roughness (RS), the control charts showed that the process was not entirely within the control limits, and labs. 1, 3 and 5 were outside of the control limits of [1 842,9; 2 060,3] with a mean of 1 951,6. For Roughness (SS), the control charts showed that lab. 5 was far lower than the other labs. The control limits were [1 390,6; 1 570,3] with a mean of 1 480,5. For Thickness, the control charts showed that the process was not entirely within the control limits. The results for lab. 7 were far below the other laboratories (see sample 9 in the graph). The control limits for thickness were [186,05; 192,37] with a mean of 189,21. The results for Air permeability were hard to place into control limits. This process was not in control, as labs 1 and 9 were completely outside of the control limits. It was recommended to investigate this further, as the results were very variable between laboratories.

After the above initial analysis, the analysis was re-run but with the removal of the outlying substrates and/or labs. This analysis is presented below in the section ANOVA outputs (7.3). The results show significant differences between substrates and laboratories.

For Thickness (after removal of lab. 7 as this showed extremely low results, as shown in the control charts), labs. 5 and 8 reported significantly higher results than lab. 1. Macherey-Nagel had significantly higher results than the other substrates, while Hahnemuehle had significantly lower results for Thickness than the other substrates. Whatman and Tervakovski were not significantly different.

For Roughness (smooth side), no differences were observed between the laboratories. However, significant differences were observed between the substrates, with Macherey-Nagel showing the highest values and Tervakovski showing the lowest values. No significant difference was found between Hahnemuehle and Whatman.

For Roughness (rough side) differences were observed between laboratoies and substrates. The highest values were observed for the Whatman and Macherey-Nagel substrates, whereas the Tervakovski samples showed significantly lower values. Hahnemuehle was between the two groups of substrates and was significantly different from both. Lab. 5 showed the lowest results, while labs. 2 and 4 showed significantly higher results than lab. 5, but lower results than labs. 1 and 3.

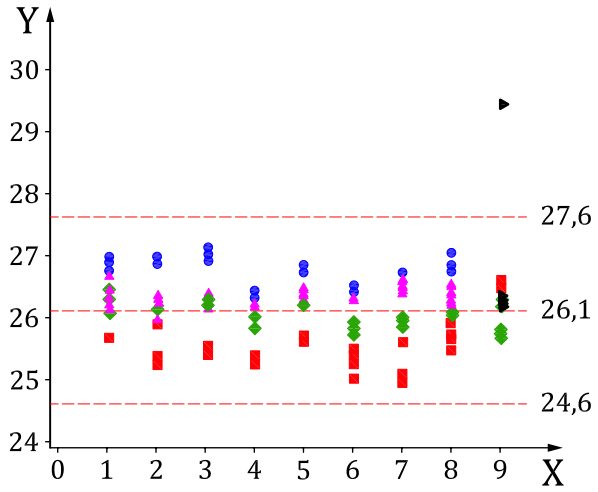
For Air permeability, significant differences were observed between all laboratories and all substrates. In terms of the substrates, the highest values were observed for Hahnemuehle, and the lowest values for Whatman. In terms of the laboratories, the highest values were reported by lab. 1, and the lowest by lab. 9.

For Dried weight, significant differences were observed between the laboratories and substrates. The Hahnemuehle substrate had the highest values, and was significantly higher than Whatman, Tervakovski and Macherey-Nagel, with Macherey-Nagel also being significantly lower than Whatman and Tervakovski. Lab. 8 had significantly higher values for Dried weight than labs. 4, 2 and 7. Lab 7 showed significantly lower values of Dried weight than labs. 8, 1 and 3.

For Conditioned weight, significant differences were observed between all substrates and some of the laboratories. For the substrates, Hahnemuehle had the highest values of conditioned weight, followed by Whatman, ahead of Tervakovski, with Macherey-Nagel showing the lowest values of conditioned weight. Lab. 1 was significantly higher in Conditioned weight than labs. 7, 6 and 4. Additionally, labs. 6 and 4 reported significantly lower values of Conditioned weight than labs 1, 5 and 3.

7.2 Mean ±3 standard deviation plots based on specifications

Figure 1 shows a scatterplot of Weight vs. Recoded Laboratory, using 15 conditioned sheets.

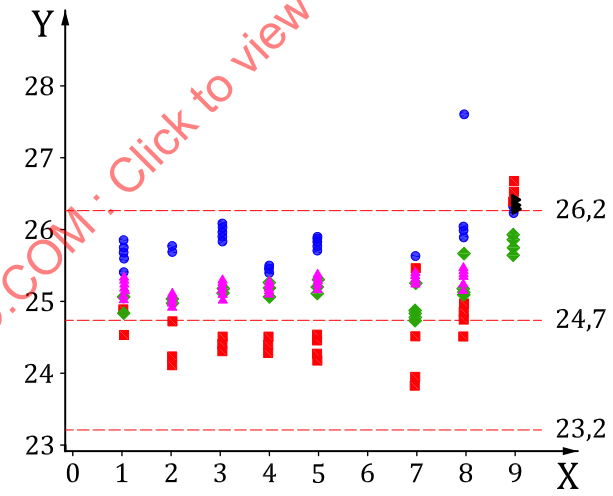


Key

X	recoded laboratory	◆	Tervakoski (Lip-Can)
Y	weight (15 cond. sheets) (g)	▲	Whatman
●	Hahnemühle	▶	Whatman
■	Macherey-Nagel		

Figure 1 — Scatterplot of Weight (15 Cond. Sheets) (g) vs. Recoded Laboratory

Figure 2 shows a scatterplot of Weight vs. Recoded Laboratory, using 15 dried sheets.

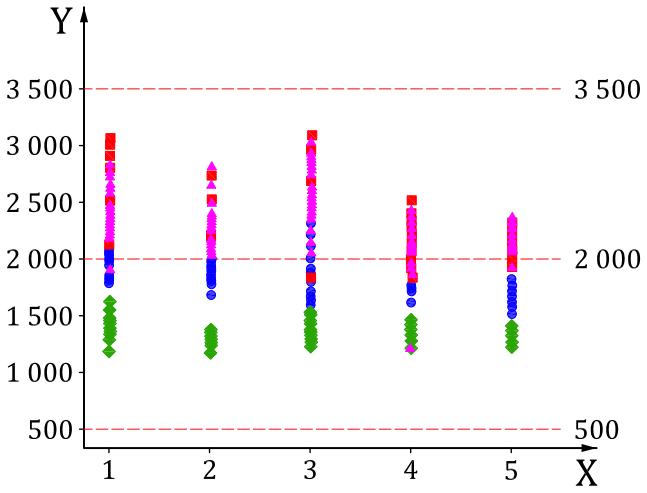


Key

X	recoded laboratory	◆	Tervakoski (Lip-Can)
Y	weight (15 dried sheets) (g)	▲	Whatman
●	Hahnemühle	▶	Whatman
■	Macherey-Nagel		

Figure 2 — Scatterplot of Weight (15 Dried Sheets) (g) vs. Recoded Laboratory

Figure 3 shows a scatterplot of Roughness (RS) vs. Recoded Laboratory.

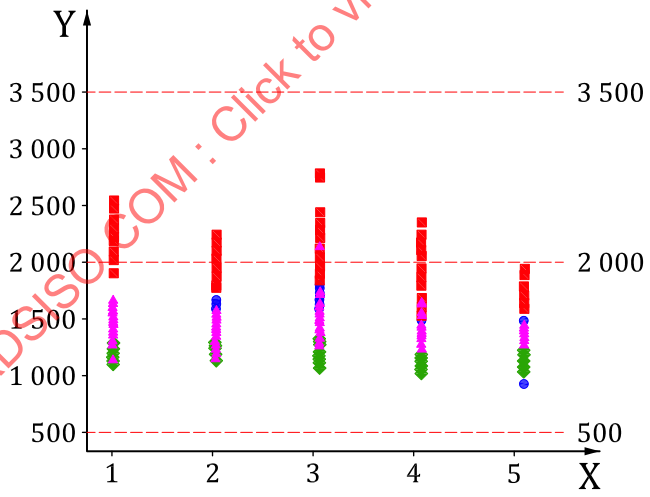


Key

X	recoded laboratory	◆	Tervakoski (Lip-Can)
Y	roughness (RS) (ml/min)	▲	Whatman
●	Hahnemühle	▶	Whatman
■	Macherey-Nagel		

Figure 3 — Scatterplot of Roughness (RS) (ml/min) vs Recoded Laboratory

Figure 4 shows a scatterplot of Roughness (SS) vs. Recoded Laboratory.

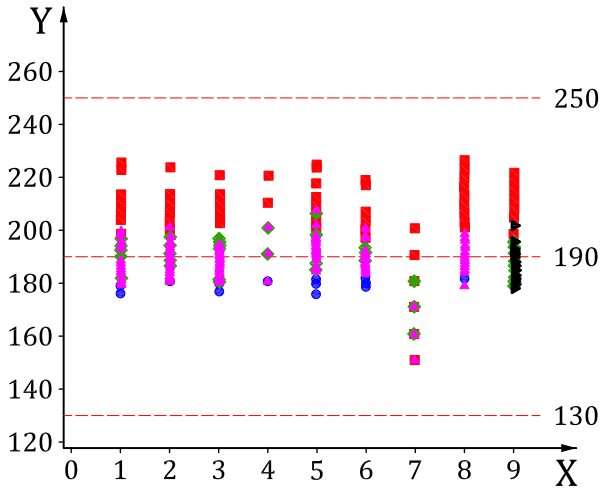


Key

X	recoded laboratory	◆	Tervakoski (Lip-Can)
Y	roughness (SS) (ml/min)	▲	Whatman
●	Hahnemühle	▶	Whatman
■	Macherey-Nagel		

Figure 4 — Scatterplot of Roughness (SS) (ml/min) vs Recoded Laboratory

Figure 5 shows a scatterplot of Thickness vs. Recoded Laboratory.

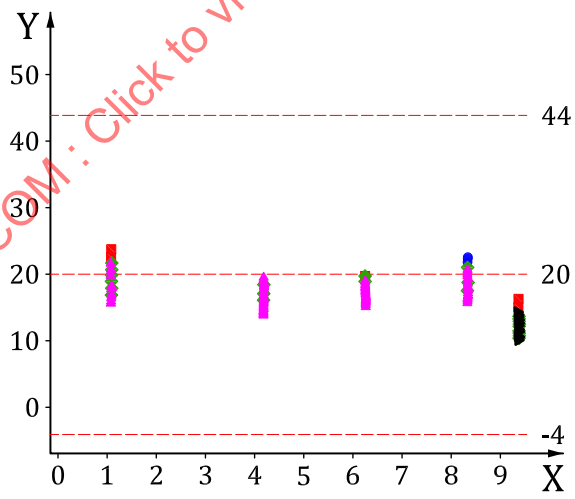


Key

X	recoded laboratory	◆	Tervakoski (Lip-Can)
Y	thickness (µm)	▲	Whatman
●	Hahnemühle	▶	Whatman
■	Macherey-Nagel		

Figure 5 — Scatterplot of Thickness (µm) vs Recoded Laboratory

Figure 6 shows a scatterplot of Air Permeability vs. Recoded Laboratory.



Key

X	recoded laboratory	◆	Tervakoski (Lip-Can)
Y	air permeability (s)	▲	Whatman
●	Hahnemühle	▶	Whatman
■	Macherey-Nagel		

Figure 6 — Scatterplot of Air Permeability (s) vs Recoded Laboratory

7.3 ANOVA outputs

Tables 2 to 13 show comparisons of various test results. It is noted that means that do not share a grouping letter are significantly different.

Table 2 — Comparison for Thickness (µm), Tukey Pairwise Comparisons: Substrate

Grouping information using the Tukey Method and 95 % Confidence			
Substrate	**N**	**Mean**	**Grouping**
Macherey-Nagel	150	207,589	A
Tervakoski (Lip-Can)	150	188,822	B
Whatman	150	188,449	B
Hahnemuehle	150	184,246	C

Table 3 — Comparison for Thickness (µm), Tukey Pairwise Comparisons: Laboratory

Grouping information using the Tukey Method and 95 % Confidence				
Laboratory	**N**	**Mean**	**Grouping**	
5	80	194,050	A	
8	80	193,548	A	
3	80	192,388	A	B
4	80	192,375	A	B
9	80	192,150	A	B
2	80	191,738	A	B
6	40	191,125	A	B
1	80	190,838		B

Table 4 — Comparison for Roughness (SS) (ml/min), Tukey Pairwise Comparisons: Substrate

Grouping information using the Tukey Method and 95 % Confidence				
Substrate	**N**	**Mean**	**Grouping**	
Macherey-Nagel	80	2 054,39	A	
Hahnemuehle	79	1 436,05		B
Whatman	80	1 397,46		B
Tervakoski (Lip-Can)	80	1 194,71		C

Table 5 — Comparison for Roughness (SS) (ml/min), Tukey Pairwise Comparisons: Laboratory

Grouping information using the Tukey Method and 95 % Confidence			
Laboratory	**N**	**Mean**	**Grouping**
1	80	1 550,16	A
3	80	1 549,44	A
4	79	1 493,61	A
2	80	1 489,40	A

Table 6 — Comparison for Roughness (RS) (ml/min), Tukey Pairwise Comparisons: Substrate

Grouping information using the Tukey Method and 95 % Confidence			
Substrate	**N**	**Mean**	**Grouping**
Macherey-Nagel	99	2 348,67	A
Whatman	99	2 292,47	A

Table 6 *(continued)*

Grouping information using the Tukey Method and 95 % Confidence			
Substrate	**N**	**Mean**	**Grouping**
Hahnemuehle	100	1 848,99	B
Tervakoski (Lip-Can)	100	1 324,42	C

Table 7 — Comparison for Roughness (RS) (ml/min), Tukey Pairwise Comparisons: Laboratory

Grouping information using the Tukey Method and 95 % Confidence			
Laboratory	**N**	**Mean**	**Grouping**
3	79	2 122,13	A
1	80	2 088,20	A
2	80	1 925,43	B
4	79	1 856,19	B
5	80	1 776,25	C

Table 8 — Comparison for Air Permeability (s), Tukey Pairwise Comparisons: Substrate

Grouping information using the Tukey Method and 95 % Confidence			
Substrate	**N**	**Mean**	**Grouping**
Hahnemuehle	150	18,407 3	A
Macherey-Nagel	150	17,853 3	B
Tervakoski (Lip-Can)	149	16,896 1	C
Whatman	150	16,143 0	D

Table 9 — Comparison for Air Permeability (s), Tukey Pairwise Comparisons: Laboratory

Grouping information using the Tukey Method and 95 % Confidence			
Laboratory	**N**	**Mean**	**Grouping**
1	120	20,208 3	A
8	120	19,100 0	B
6	119	17,439 3	C
4	120	16,837 9	D
9	120	13,039 2	E

Table 10 — Comparison for Weight (15 Dried Sheets) (g), Tukey Pairwise Comparisons: Substrate

Grouping information using the Tukey Method and 95 % Confidence			
Substrate	**N**	**Mean**	**Grouping**
Hahnemuehle	34	25,617 5	A
Whatman	35	25,137 2	B
Tervakoski (Lip-Can)	34	25,009 2	B
Macherey-Nagel	35	24,462 4	C

Table 11 — Comparison for Weight (15 Dried Sheets) (g), Tukey Pairwise Comparisons: Laboratory

Grouping information using the Tukey Method and 95 % Confidence					
Laboratory	**N**	**Mean**	**Grouping**		
8	19	25,241 9	A		
1	20	25,137 5	A	B	
3	20	25,102 3	A	B	
5	20	25,094 4	A	B	C
4	19	24,986 9		B	C
2	20	24,941 8		B	C
7	20	24,891 1			C

Table 12 — Comparison for Weight (15 Cond. Sheets) (g), Tukey Pairwise Comparisons: Substrate

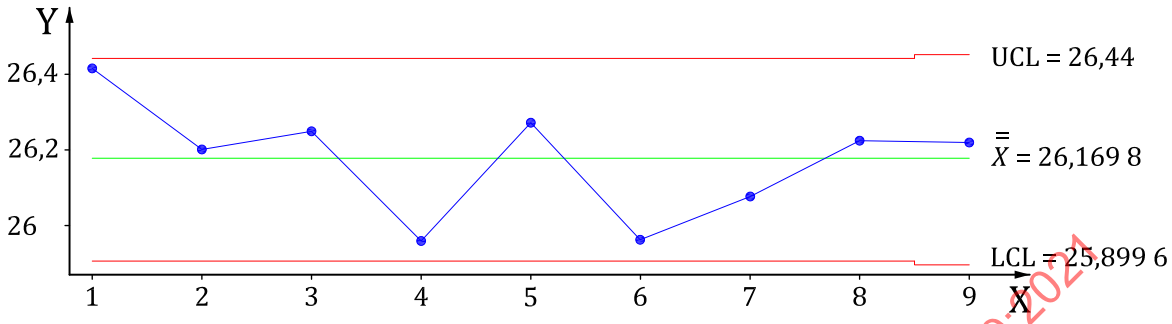
Grouping information using the Tukey Method and 95 % Confidence						
Substrate	**N**	**Mean**	**Grouping**			
Hahnemuehle	45	26,647 1	A			
Whatman	44	26,309 2		B		
Tervakoski (Lip-Can)	45	26,069 2			C	
Macherey-Nagel	45	25,657 9				D

Table 13 — Comparison for Weight (15 Cond. Sheets) (g), Tukey Pairwise Comparisons: Laboratory

Grouping information using the Tukey Method and 95 % Confidence					
Laboratory	**N**	**Mean**	**Grouping**		
1	20	26,401 0	A		
5	20	26,266 3	A	B	
3	20	26,238 9	A	B	
8	20	26,221 4	A	B	C
9	19	26,221 3	A	B	C
2	20	26,192 4	A	B	C
7	20	26,075 7		B	C
6	20	25,963 6			C
4	20	25,957 2			C

7.4 Control Charts

Figure 7 shows an Xbar-R chart of weight for 15 conditioned sheets.

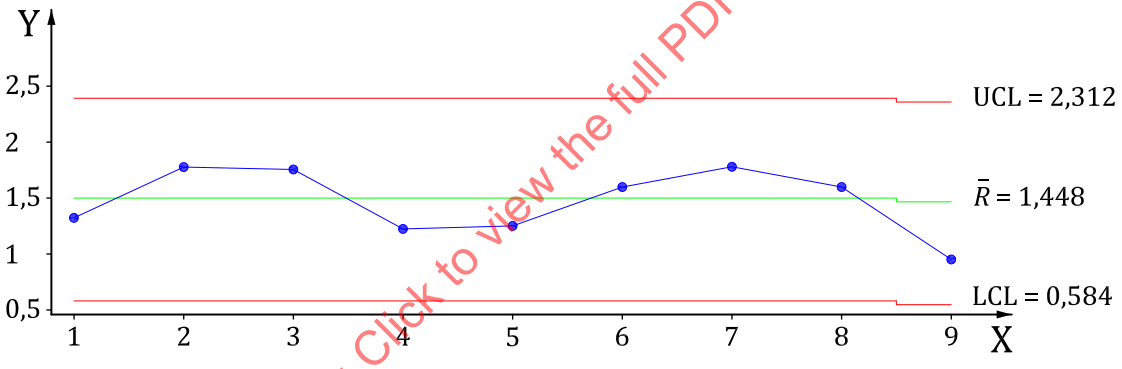


Key

X sample

Y sample mean

a) Chart showing sample mean (average sample mean, $\overline{\overline{X}}$)



Key

X sample

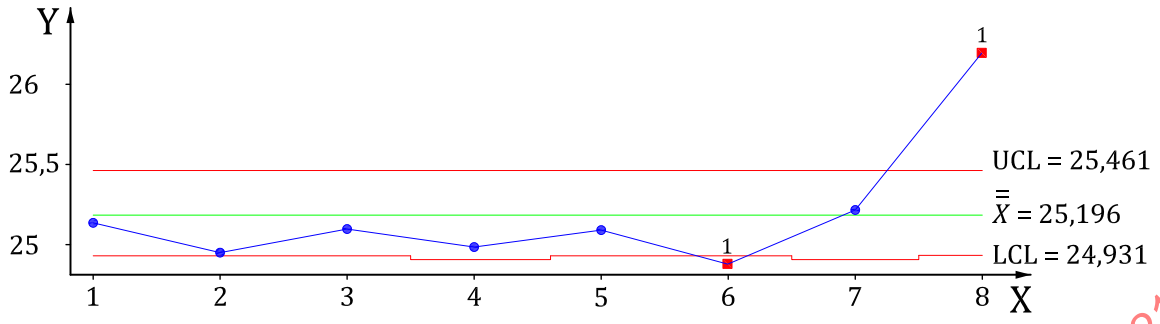
Y sample range

b) Chart showing sample range (average sample range, $\overline{R}$)

NOTE Tests were performed with unequal sample sizes.

Figure 7 — Xbar-R Chart of Weight (15 Cond. Sheets) (g)

Figure 8 shows an Xbar-R chart of weight for 15 dried sheets.

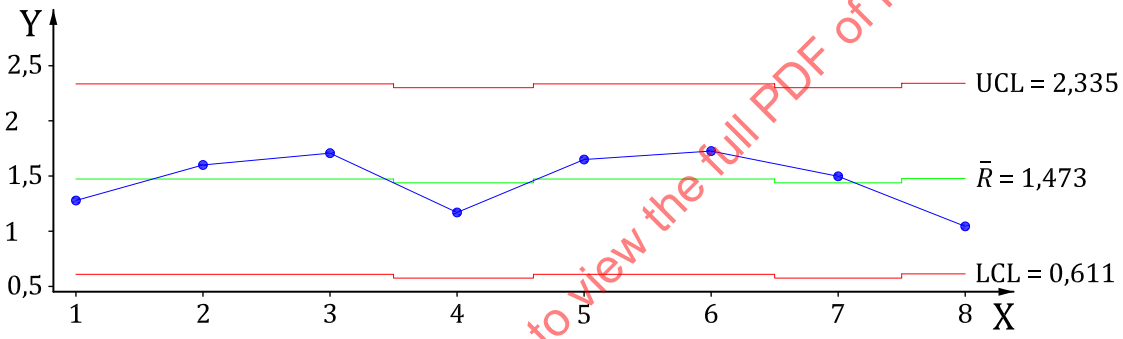


Key

X sample

Y sample mean

a) Chart showing sample mean (average sample mean, $\bar{\bar{X}}$)



Key

X sample

Y sample range

b) Chart showing sample range (average sample range, $\bar{R}$)

NOTE Tests were performed with unequal sample sizes.

Figure 8 — Xbar-R Chart of Weight (15 Dried Sheets) (g)