TECHNICAL REPORT



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

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Foreword

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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*.

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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 <u>www.iso.org/members.html</u>.

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].

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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 11 en STANDARD PREVIEW

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

ISO and IEC maintain terminological 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

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https://standards.iteh.ai/catalog/standards/sist/561dcf88-0f3a-4158-8424-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

<u>Table 1</u> 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 Teh S	ΓΑΝΤ (190±20) μmp ΓVΤ	ISO 534 ^c	
	5 oz cylinder, 1 inch² orifice plate, 100 ml air volume.		
^b Measurement pressure: 1,47 kPa.	Measurement pressure: 1,47 kPa. (standards.iteh.ai)		
Measurement pressure: 53 kPa. Area of clamping zone: 200 mm ² .			

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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. **KD PKEVIE**

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 <u>7.2</u> and <u>7.3</u>.

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 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. 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.