NIST Technical Note 1874

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National Institute of Standards and Technology
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U.S. Department of Commerce
Penny Pritzger, Secretary

National Institute of Standards and Technology
Willie May, Under Secretary of Commerce for Standards and Technology and Director

The research on reduced ignition propensity cigarettes conducted by the National Institute of Standards and Technology (NIST) since 1984 was done in the interest of saving lives and protecting property from cigarette-induced fires. In no way does it lessen or negate the health hazards and addictive nature of smoking as determined by the Surgeon General or suggest that NIST and the Department of Commerce condone smoking.

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ABSTRACT

ASTM E 2187 has become the internationally referenced standard for designing and specifying less fire-prone cigarettes. In this test method, a lit cigarette is laid on multiple layers of filter paper, and the observer identifies whether the cigarette burns its full length or not. Historically, a specific brand of filter paper has been used as the test substrate, with specified ranges of mass and moisture uptake necessary to achieve reasonable repeatability and reproducibility of the test results. This approach became less viable when the manufacture of the paper was moved to a different plant, and when an increased demand for the paper resulted in long delays in delivery. This Technical Note describes an interlaboratory study (ILS) of an alternate substrate composed of a thin sheet of full hard 302 stainless steel and a single sheet of filter paper. Seven laboratories examined the effect of filter paper from four manufacturers and steel from three manufacturers on the test results for four cigarette designs. The findings of the ILS were (1) that the ignition propensity results obtained using the steel/paper substrates were not statistically distinguishable from the result obtained for testing on 10 layers of the original filter paper, (2) there were no statistically significant effects of the substrate materials on the test results, for those combinations of cigarette, paper, and steel, and (3) the filter paper manufactured in the new plant gives test results that are more than a factor of two lower than the results from the original plant.

Keywords: ASTM E 2187, cigarette, fire, fire safety, less fire-prone cigarettes, substrate

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1. ASTM E 2187

Since its initial publication in 2002, ASTM E 2187, Standard Test Method for Measuring the Ignition Strength of Cigarettes, has become the basis for designing and specifying less fire-prone cigarettes.[1] This Standard is cited in regulations in the 50 U.S. States, Canada, Australia, New Zealand, and Finland. The European Union requested that ISO develop an identical version (except for format) of the ASTM Standard that could be cited in a European Standard, and that process has resulted in ISO 12863:2010.[2] The current version of the ASTM Standard is ASTM E 2187-09.

In this test method, a cigarette is lit, allowed to burn long enough to "forget" the lighting process (*ca.* 15 mm), and placed on a set number of layers of filter paper. The filter papers act as a heat sink, absorbing energy from the cigarette. A cigarette of high ignition strength continues burning its full length, despite the heat loss to the paper. The coal in a weaker burning cigarette cannot endure the heat loss and still continue burning. The result of a single determination is whether the cigarette burns its full length or not. Typical results are shown in Figure 1.

Figure 1. Photograph of Typical Results of Determinations using ASTM E 2187. Top: full-length burn of a conventional non-filter cigarette; Left: full-length burn of a conventional filter cigarette; Right: ceased burning of a less fire-prone cigarette.

To provide for measurement of a range of ignition strengths, ASTM E 2187 includes three different substrates, providing three different heat sinks. These three consist of 3, 10, or 15 layers of filter paper. To measure the ignition strength of a cigarette type, 40 determinations are performed for the cigarette type on a chosen substrate. The fraction of the 40 determinations that results in full-length burns is recorded. Conventionally, this fraction is converted to a percentage of full-length burns (PFLB) by multiplying by 100 %. All the current regulations require testing on 10 layers of filter paper and require that no more than 10 of the 40 determinations result in a full-length burn; i.e., only cigarettes with measured 25 PFLB or lower can be sold.

This method is derived from the Cigarette Extinction Method, which was developed under the Fire Safe Cigarette Act of 1990.[3] In that developmental effort, and in all the research and testing leading up to the first version of ASTM E 2187, Whatman No. 2 filter paper was used in the test substrates. This was justified on the basis that this was the prevalent filter paper available in the United States, although the paper was manufactured in the United Kingdom (UK).

During initial testing of the method, there were reports of erratic results from some batches of the paper. It was recognized that, since the paper is chemically uniform (i.e., nearly pure cellulose), a chemical difference was not likely to be the source of the odd results. Two significant properties of the paper that affect its effectiveness as a heat sink are the mass and the moisture content. The paper batches on hand were then analyzed for their dry mass and their mass at the conditioning temperature and relative humidity under which the testing was performed. This led to the finding that most of the errant results were from paper specimens whose conditioned and dry masses were outside narrow ranges. These ranges were then added to the requirements in ASTM E 2187 for paper selection. A key outcome of this was the realization that the test results were quite sensitive to the thermal character of the layers of filter paper.

In November 2006, Whatman introduced a Certified Grade of their No. 2 paper. The dry and conditioned masses are provided, and the paper batch is certified to be within the mass ranges required in ASTM E 2187-04.

Meanwhile, because of the compliance requirements for the sale of cigarettes, the manufacturers and regulators expressed the need for some means for assuring that test laboratories were performing the test properly and uniformly. The National Institute of Standards and Technology (NIST) responded to this need with the development of Standard Reference Material 1082 (SRM 1082), Cigarette Ignition Strength Standard. This cigarette is certified to a value and expanded uncertainty of 12.6 PFLB ± 3.3 PFLB when tested on 10 layers of Whatman No. 2 filter paper according to ASTM E 2187-04. The certified value is based on data from three laboratories that each tested between 200 and 800 cigarettes. The use of these large samples sizes results in an uncertainty that is considerably lower than would be expected based on the standard number of cigarettes tested in the field, which is set by ASTM E 2187-04 at 40 cigarettes.

ASTM E 2187-09 contained two key differences from the 2004 version of the Standard. The first was a requirement that at least every 30 days of active testing, a laboratory was to verify the performance of the total test system and operator using a test cigarette whose ignition propensity using ASTM E 2187 had been established. The Standard noted that SRM 1082 was widely used for this purpose.

The second key difference resulted from the observation that the two sides of a sheet of Whatman No. 2 filter paper had differing degrees of roughness. RJR Tobacco found a small, but significant dependence of test performance for an experimental cigarette, depending on which side of the paper was facing upward. Testing with the rough side upward resulted in on the order of 10 % more full-length burns than testing with the smooth side upward.[4] It was hypothesized that the higher PFLB value was due to the less regular thermal contact between the cigarette and the substrate. NIST informally confirmed the nature of this finding. By general agreement, ASTM E 2187-09 required that future testing be conducted with the more conservative orientation, i.e., with the rough side facing upward.

There is an implication for the expected value of the SRM 1082 cigarettes when used to "calibrate" a laboratory's performance using ASTM E 2187-09. The certification of the SRM 1082 cigarettes pre-dated the finding that paper surface orientation affected the test results. Presumably, the paper substrates in the certification testing were approximately randomly distributed with regard to the rough surface orientation, i.e., about half of the cigarettes were tested on substrates with each of the two paper orientations. Thus, the expected test result from ASTM E 2187-09 using SRM 1082 cigarettes should be greater than the certified value by approximately one-half of the difference between tests with the rough side upward and tests with the smooth side upward.

As part of an ILS of alternate filter papers[5] (discussed further in the next section of this report), testing of several cigarette designs on 10 layers of Whatman No. 2 filter paper showed the average test result with the rough side up was approximately 20 % higher (i.e., approximately 1.2 times the smooth side result) than the result with the smooth side up. This would imply that the test result using ASTM E 2187-09 (testing with the rough side always up) would be about 10 % greater than the values obtained from testing with random paper orientation. However, there was a nearly 40 % standard deviation in the rough/smooth ratio; and, in fact, the cigarettes tested by two of the eight ILS laboratories showed *lower* results with the rough side up.

To this point, all of the Whatman No. 2 filter paper had been manufactured at its plant in the United Kingdom (UK).

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2. The Desire for an Alternate Substrate

2.1 Development Efforts

During the period leading up to the publication of ISO 12863, additional cigarette manufacturers began developing prototypes of their cigarettes brands that would meet the regulatory requirement. This led to an increase in the demand for Whatman No. 2 filter paper. By 2011, Whatman (which had become part of GE Healthcare in 2008) moved the production of the paper from its traditional facility in the UK to a new facility in China. Prior to and during the move, there were long delay times in filling orders for the filter paper. There was also some concern that the paper from the two facilities might result in different test results and that future changes in crops and/or processing might lead to additional changes in test results. All of this suggested that additional technical work was needed to ensure test results that were invariant over time.

New projects were initiated to achieve a more stable substrate for the regulatory testing, i.e., addressing the substrate consisting of 10 layers of Whatman No. 2 filter paper.

- An interlaboratory study (ILS) of other commercial brands of filter paper was conducted to determine whether any of them generated test results consistent with the value and reproducibility of the regulatory standard. [5] Eight laboratories participated. Each of the three alternate filter paper types demonstrated test result repeatability similar to that obtained using the Whatman No. 2 (UK) filter paper. One of the three alternate papers also showed test results that were statistically indistinguishable from the results obtained using Whatman paper for all test cigarettes. However, significant additional testing would be required for high confidence that testing a variety of cigarettes using any of the three alternate filter paper types would consistently yield test data indistinguishable from the data obtained using the currently specified paper. That extensive testing has not been pursued.

  During this ILS, the test operators noted that the tactile difference between the two surfaces of the Chinese paper was more difficult to discern that it was for the UK paper. This suggested that, for the Chinese paper, there might be a lesser dependence of test results on surface orientation.

- ISO TC92 SC1 WG15 (Ignition Propensity of Cigarettes), the Working Group charged with developing ISO 12863, commissioned a Task Group to develop a more complete specification for the filter paper. This would enable manufacturers to respond to property requirements that would assuredly lead to consistent test results. This Task Group is currently identifying the pertinent paper properties, the methods of measurement, and the ranges within which the paper would need to be produced.

- NIST developed an alternate substrate that generated a test value and repeatability consistent with the performance of an SRM 1082 cigarette on 10 layers of Whatman No. 2 filter paper manufactured in the UK. [6] This substrate is the subject of this ILS.

2.2 The Proposed Alternate Substrate

The rationale for this substrate focused on two principles:

1. The substrate in ASTM E 2187 serves as a heat sink. A substrate consisting of 10 layers of filter paper is (or is close to being) thermally thick. The bottom sheet of filter paper

does not get very warm, and little heat is lost through the substrate. Thus, the test result is not very sensitive to small changes in the thermal inertia or the thickness of the substrate. The use of a thermally thin substrate is also possible. However, heat losses through such a substrate could be significant. Thus, tight control of the thickness and thermal properties would be important.

NIST examined a large number of thermally thick and thermally thin substrates.[6] These included organic and inorganic materials of varying thicknesses, thermal conductivities, heat capacities, thicknesses, and surface characteristics. Ignition propensity screening with nearly all of the candidates resulted in either no full-length burns or 100 % full-length burns. Of the remaining candidates, some deformed, were brittle, or were expensive. Expensive materials could be considered if they could be used for a sufficient number of tests. This meant, for example, that the material could not change phase and that the surface could be readily cleaned.

2. Water vapor generated by the cigarette should not condense on the substrate surface. As found in the development of a means for measuring the ignition propensity of pre-standard cigarettes[7], this leads to a puddle of water forming on the substrate surface near the cigarette coal. This "drowns" the cigarette smoldering, a phenomenon not related to the real-world ignition scenario. This could be prevented by the substrate surface being too warm for water condensation. The cigarette does not generate sufficient heat for this, and the use of a hot substrate is, again, not a replication of common ignition conditions. An alternative means of preventing the pooling of liquid water is to wick the water away from the vicinity of the coal. This was achieved by placing filter paper on top of a metal substrate.[7]

The NIST experiments led to a thermally thin substrate consisting of a sheet of nominally 0.203 mm (0.008 in.) thick full hard 302 stainless steel shim stock, covered with a single sheet of filter paper (Figure 2). The substrate is supported on an adapter ring that fits into the regular substrate holder (Figure 3).

Figure 2. Photograph of a Cigarette on the Steel and Paper Substrate.

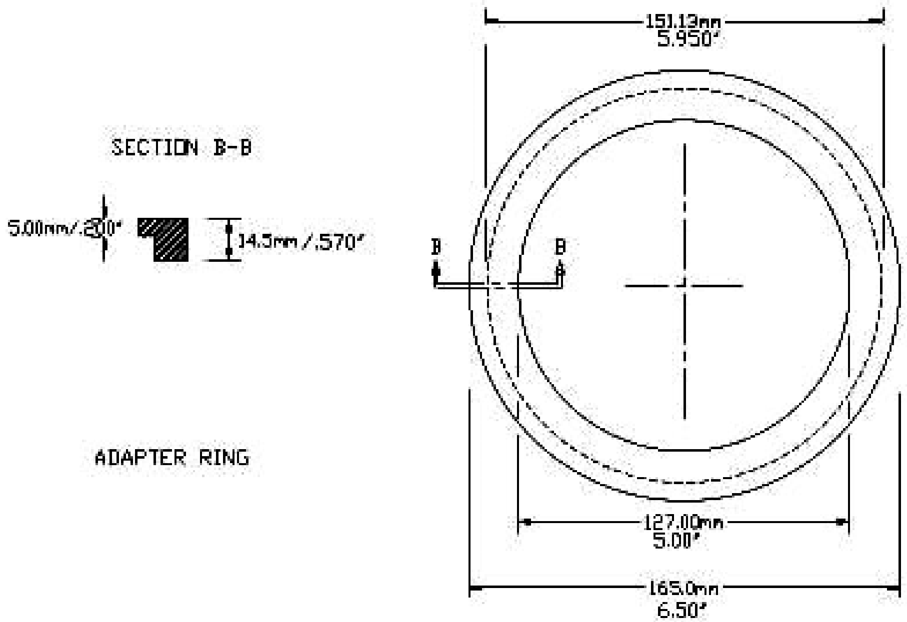

Figure 3. Schematic of the Adapter Ring to Support the Stainless Steel Substrate.

The variability in the thermal inertia of the metal sheet would be proportional to the combined variability in the thickness and in the thermal properties. The shim stock is used for a variety of applications, each of which requires precision in the thickness.[i] The specified range of alloy composition of 302 stainless steel is narrow[8] and the alloy properties are covered in at least 24 Standards[9] so very little variability in the thermal properties was expected. The demand for the shim stock is sufficient that there are multiple manufacturers of the product. As a result, it was anticipated that an inexpensive, uniform, and stable supply of the shim stock would be available, and this was the case.

The ILS of other commercial filter paper brands indicated significant additional testing was needed to establish equivalency to 10 layers of Whatman No. 2 paper. However, the role of the paper in this metal-based substrate is simpler (for example, there are no paper-to-paper interfaces), and the hypothesis was that a paper specification based on mass and moisture content might well suffice.

NIST performed exploratory experiments to scope the ability of this substrate to match the measured tests results obtained with 10 layers of Whatman No. 2 (UK) filter paper.[6] The study included three types of cigarettes and shim stock from three suppliers. Whatman No. 2 filter paper from the UK was used with either the rough or the smooth side upward. Whatman No. 2 filter paper from China and two of the filter paper brands from the prior ILS were tested with the rough side up, if a difference could be discerned.

[i] Manufacturing applications include spacers for machine tool alignment, precision spacing of digital readout glass scales on machine tools, and building up of worn parts. There are also applications in the aerospace industry.

The key findings were:

- For all three cigarette types, one of which was SRM 1082, there was little sensitivity to the source of the metals or the filter paper brand tested with the rough side upward.
- The repeatability of the test results was slightly superior (i.e., the standard deviation was slightly smaller) for the metal/paper substrate, compared with the 10-layer filter paper substrate.
- For SRM 1082 cigarettes, the test result on 10 layers of UK Whatman No. 2 filter paper with the rough side of all sheets facing upward was approximately 15 % higher than the certified value. Given the small number of tests performed, this single-laboratory result was consistent with finding of an approximate 10 % difference derived from the filter paper ILS, as presented above.
- For some cigarette designs, there may be significant differences in test results using papers from the two manufacturing plants.

Based on these findings, the next step was to examine the performance of the alternate substrate with components obtained from different manufacturers and with additional cigarette designs. These data would be used to establish the degree of reproducibility of the results by the participation of multiple laboratories.

3. This Interlaboratory Study (ILS)

3.1 ILS Participants

With input from discussions with potential participants, NIST prepared a draft plan for an ILS of the alternate substrate. Seven laboratories agreed to participate in the ILS:

- Altria Client Services
- Delfort Group
- Lorillard Tobacco Company
- Microbac Laboratories
- National Institute of Standards and Technology
- RJR Tobacco Company
- Schweitzer-Mauduit International

3.2 ILS Plan

Objective: Determine the test value, the repeatability, *r* (the band within which differences among repeat test results from a single laboratory will fall about 95 % of the time), and the reproducibility, *R* (the band within which differences among test results from different laboratories will fall about 95 % of the time) of the ignition propensity of cigarettes determined using a substrate consisting of:

- A single sheet of nominally 0.2 mm (0.008 in.) 302 full hard stainless steel shim stock and
- One layer of filter paper.

The task was to assess *r* and *R* for a variety of cigarette designs, as well as the sensitivity to variability in the substrate materials.

It was unlikely that there was sufficient UK paper to develop a broader evaluation of the performance of cigarettes on the Whatman filter paper from China relative to testing using the UK Whatman filter paper. However, the inclusion of SRM 1082 cigarettes in this ILS provides a link to prior cigarette performance data using batches of the Whatman No. 2 paper manufactured in the UK.

Test Materials:

- NIST provided a test procedure and spreadsheets for recording and transmitting the test data.
- NIST provided pre-cut pieces of full hard 302 stainless steel shim stock. The pieces were nominally 150 mm x 159 mm and were cut from flat (not rolled) stock. The provisional thickness specification was 0.207 mm ± 0.002 mm. The specimens were obtained from vendors whose products came from three different manufacturers. NIST measured the thickness of samples of the three sets of sheets. These were used to arrive at a final specification for the sheet thickness.
- Each participating laboratory was to obtain its own supply of four brands of commercial filter paper: Whatman No. 2 (China), Delfort, Ahlstrom, and Machery-

Nagel. Each laboratory was to verify that their batches of paper all met the following initial specifications (suggested from the prior NIST research):

- Dry mass (15 sheets, 150 mm diameter): (24.8 g ± 0.6) g
- Mass of 15 sheets conditioned at (20 ± 3) °C and (55 ± 5) % relative humidity (the conditions in ASTM E 2187-09): (26.1 ± 0.6) g
- Difference in moisture content: (0.052 ± 0.006) mass fraction

The laboratories were to send the batch numbers and 20 sheets from each batch to NIST. The range of these paper masses and water content were to be considered in arriving at a final specification for the filter paper.

- NIST provided an insert to the ASTM E 2187 substrate holder (Figure 3). This provided support for the metal/paper substrate.
- NIST provided a flat jig plate for use in determining the flatness of the stainless steel sheets and for support while cleaning the sheets between tests. The cast tool and jig plate was made of 6063-T5 aluminum. The dimensions were nominally 300 mm x 300 mm x 12.7 mm. The flatness was 0.38 mm over a source plate with dimensions 2.22 m x 3.66 m. NIST included a tool for determining the extent to which the sheets were not sufficiently flat.
- NIST also provided supplies of three brands of (banded) commercial cigarettes with the pack information removed. The cigarettes were not necessarily from the current marketplace, and their expected test values were between approximately 10 PFLB and 25 PFLB. Each laboratory purchased the needed supply of SRM 1082 cigarettes.

Test Plan:

The ILS was conducted in three rounds. By agreement, the total number of tests per participant was to be approximately 1200. The testing in each laboratory was to be performed by a single test operator throughout the ILS.

Round 1 (200 determinations per laboratory)**:**

- Purpose: Assure that each laboratory and operator was performing ASTM E 2187-09 properly, obtaining the proper value and precision.
- Each laboratory provided data from 5 sets of 40 determinations using SRM 1082 cigarettes on 10 layers of Whatman No. 2 filter paper (China). The filter paper was to be oriented with the rough side facing upward, as in ASTM E 2187-09. The data could be from the laboratory's test archives; if so, the data were to be the most recent obtained by the same test operator who would perform the remainder of the tests for this ILS.

Round 2 (100 to 200 determinations per laboratory)**:**

- Purpose: Identify any needed refinements/clarifications in the test procedure prior to general testing.
- Each laboratory was to perform 5 sets of 20 determinations with SRM 1082 cigarettes using a single substrate, consisting of a stainless steel sheet (Metal 1) and a single piece of Whatman No. 2 filter paper (China), oriented with the rough side facing up, if rough and smooth sides could be discerned.

- As the testing began, it became clear that the mass of the metal rim in ASTM E 2187 was not sufficient to flatten the metal sheet fully. The test procedure was modified to increase the applied mass. This is reflected in the final protocol for Round 2 (and Round 3). (See Appendix A.) All but one of the laboratories that had already performed Round 2 testing repeated those tests with the additional mass.

Round 3 (800 determinations per laboratory)**:**

- Purpose: Obtain test method data for a variety of cigarette types, stainless steel sources, and paper types, including the test values, the repeatability for each laboratory and substrate composition, and the ILS reproducibility for each substrate composition.
- It was decided that, while multiple combinations of cigarette, paper, and steel types needed to be tested, a prime consideration was the assessment of the repeatability and reproducibility of testing with the proposed substrate. This led to maximizing the number of tests per combination, i.e., all laboratories performed the same tests. This was in contrast with a full or fractional factorial design for the ILS, in which more combinations would be examined, but with fewer determinations each.

  Accordingly, each laboratory performed 5 sets of 20 determinations for the following combinations:

	Cigarette Number							
	1 (SRM 1082)		2		3		4	
Substrate Index	B	C	B	C	A	D	A	D
Metal Number	2	3	2	3	1	-	1	-
Paper Number	2	3	2	3	1	4(W)	1	4 (W)

  Note that in Round 1, Cigarette 1 was tested on Substrate D (10 layers of Paper 4, which was Whatman No.2 (China), W). In Round 2, Cigarette 1 was tested on Metal 1 and Paper 4.

- The test protocol for the steel/paper substrate was the final version developed in Round 2. The testing on 10 layers of filter paper was performed according to ASTM E 2187-09.

Data Analysis

The figures of merit in the analysis of the test results were the PFLB value, the repeatability, and the reproducibility for each combination of steel, paper, and cigarette. If the results were acceptably similar in quality to historic test properties with a substrate of 10 layers of filter paper, the characteristics of the steels and papers would provide the basis for specification in a proposed revision of the ASTM Standard.

The analysis of the test results was to include determination of the extent to which each of the following factors affected the test results for the various cigarette designs:

- Source and thickness of the 302 stainless steel,
- Type of filter paper, and
- Laboratory performance.

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4. Test Materials

4.1 Steel

NIST measured the thickness of the full hard 302 stainless steel at 12 locations on each of four sheets from each of the three manufacturers. The measurements were made using a coordinate measuring machine. The precision of the apparatus is ± 0.0003 mm. The results are compiled in Table 1.

Table 1. Thicknesses of 302 Stainless Steel Sheets (dimensions in mm, with a standard deviation less than 0.0003 mm).

Metal No. →	**1**				**2**				**3**			
Specimen →	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**
Measurement Location ↓												
1	0.1992	0.1984	0.2001	0.1989	0.2043	0.2062	0.2027	0.2048	0.1997	0.2016	0.1992	0.2003
2	0.1989	0.2001	0.1978	0.2008	0.2044	0.2044	0.2024	0.2054	0.1979	0.1993	0.1989	0.1999
3	0.1990	0.1987	0.1979	0.1996	0.2041	0.2060	0.2020	0.2045	0.2000	0.1999	0.2000	0.1995
4	0.1993	0.1983	0.1990	0.1992	0.2060	0.2064	0.2118	0.2058	0.2003	0.2001	0.2012	0.1993
5	0.1995	0.1984	0.1991	0.2010	0.2076	0.2054	0.2029	0.2054	0.2009	0.2003	0.2009	0.1995
6	0.1991	0.1991	0.1987	0.1983	0.2061	0.2056	0.2086	0.2049	0.2001	0.2000	0.1995	0.1991
7	0.1989	0.2052	0.1993	0.1999	0.2050	0.2059	0.2043	0.2068	0.1989	0.2022	0.2023	0.2004
8	0.1991	0.2003	0.1984	0.1987	0.2051	0.2055	0.2044	0.2059	0.2000	0.2028	0.2009	0.2010
9	0.2007	0.1994	0.2010	0.1980	0.2072	0.2066	0.2070	0.2060	0.1991	0.2030	0.2001	0.2001
10	0.2000	0.2056	0.1992	0.1991	0.2066	0.2066	0.2058	0.2083	0.2019	0.2007	0.1988	0.2009
11	0.2001	0.1987	0.1983	0.2015	0.2067	0.2051	0.2039	0.2074	0.2001	0.2019	0.1985	0.2000
12	0.2002	0.1998	0.1989	0.1993	0.2058	0.2097	0.2046	0.2065	0.1987	0.2010	0.1987	0.1997
Mean	0.1995	0.2002	0.1990	0.1995	0.2057	0.2061	0.2050	0.2060	0.1998	0.2011	0.1999	0.2000
Std. Dev.	0.0006	0.0025	0.0009	0.0011	0.0012	0.0013	0.0029	0.0011	0.0011	0.0012	0.0012	0.0006
95 % C.I.	0.0004	0.0016	0.0006	0.0007	0.0008	0.0008	0.0018	0.0007	0.0007	0.0008	0.0008	0.0004
Mean/metal				0.1995				0.2057				0.2002
Std. Dev.				0.0015				0.0018				0.0011
95 % C.I.				0.0004				0.0005				0.0003

There are two noteworthy observations.

1. The uniformity of the thickness of each specimen and among the multiple specimens from a single manufacturer is excellent. The 95 % confidence interval for each metal is no more than 1 % of the mean thickness of that sheet.

2. The thicknesses of Metals 1 and 3 are indistinguishable from each other. The thickness of Metal 2 is slightly (approximately 3 %), but statistically significantly, larger.

The shim stock is sold as being nominally 0.008 in. (0.203 mm) thick. If determination of the full-length burn rates were to find no effect of the source of the metal on the test results, a practical specification for the thickness would be (0.203 ± 0.004) mm. This range encompasses all the mean values with their associated confidence bounds in Table 1. It is based on our current knowledge of the different true metal thicknesses, assuming that the differences among the three manufacturers are fixed effects.

4.2 Paper

Each of the participating laboratories obtained their own supplies of the four types of filter paper. They were to send 20 sheets of each type to NIST for weighing. Not all of the participating laboratories did so. Some laboratories provided additional paper, enabling some determination of the variation in mass and moisture content within a batch. The "dry" measurements were performed after the paper had been in an oven at approximately 60 °C for at least 18 hours, the requirement in ASTM E 2187-09. The "conditioned" measurements were performed after the specimens had been in an environment at (54 ± 1) % relative humidity and (23 ± 0.1) °C for at least 18 hours. The data are compiled in Table 2.

With one exception (see below), the mean conditioned masses of all the batches of all four papers were within the specification range in ASTM E2187-09 (26.2 ± 0.5) g. For the cases where the laboratories provided multiple batches of paper specimens, the standard deviations were all below ± 0.2 g, which is also well within the specification range in ASTM E 2187-09.

With the same one exception, the dry masses of all the batches of all the papers were between 24.6 g and 25.7 g, compared to the specified range in ASTM E2187-09 (24.7 ± 0.5) g. For the cases where the laboratories provided multiple batches of paper specimens, the standard deviations were all below ± 0.2 g, which is well within the specification range in ASTM E 2187-09.

Based on historic data and the values from the preliminary NIST research[6], the paper used in this ILS was expected to have a range of moisture content of (0.052 ± 0.006) mass fraction. The actual range was (0.050 ± 0.006) mass fraction.

The one exception was Paper 2 from Lab 4. This laboratory forwarded 10 sets of filter paper sheets for weighing. Both the dry and conditioned masses were 13 % lighter by weight compared to other laboratories' batches of the same filter paper and all batches of the other filter papers. The change in moisture content was normal. The mass variability (dry and conditioned) among the 10 sets was notably higher than the variability in the other papers for which 10 sets were weighed. The nature of the batch number was also different from other batches of the same brand of paper. Laboratory 4 was contacted, and their investigation revealed that the recently purchased paper had been produced approximately 25 years ago with misleading documentation stating it was produced prior to 3/25/11. The data obtained using this paper were not included in further analyses.

Before reaching any broad conclusions about the filter paper, it should be noted that all the Paper 1 used by the seven laboratories came from the same batch. The same was true for Paper 3.

The data indicate that the four paper companies are capable of making paper with dry and conditioned masses within or very close to the narrow ranges prescribed in ASTM E2187-09. Furthermore, the moisture content was also within a narrow range. If systematic testing were to find no effect of the source of the paper on the test results, it would be reasonable to retain the conditioned mass requirement in ASTM E2187-09 of (26.2 ± 0.5) g and a widened dry mass requirement (reflecting the masses found in this ILS) of (24.9 ± 0.7) g, with an additional specification that the moisture content of the conditioned paper be (0.050 ± 0.006) mass fraction. Only 5 of the 66 individual samples did not fall within this range.

Table 2. Masses of 15 Sheets of Filter Papers Used in the ILS of the Alternate Substrate, Dried and Conditioned at 54 % Relative Humidity (mass in g, with a standard deviation of 0.001 g).

	Paper No. 2; Munktell (Ahlstrom)				Paper No. 3; Machery-Nagel				Paper No. 1; Delfort (Lipcan)				Paper No. 4; Whatman No. 2 (China)			
Lab No.	Lot No.*	Cond.	Dry	Moist. %	Lot No.	Cond.	Dry	Moist. %	Lot No.	Cond.	Dry	Moist. %	Lot No.**	Cond.	Dry	Moist. %
1	103285	25.79	24.59	4.9	104390	26.37	25.42	3.7	31036	26.35	25.08	5.1	7648	25.88	24.66	4.9
2	102372	25.98	24.85	4.5	104390	26.54	25.51	4.0	31036	26.35	25.11	4.9	6317	26.04	24.88	4.7
3	n.s.***				n.s.				n.s.				9526	25.88	25.00	3.5
4	10-188	22.74	21.54	5.6	104390	26.47	25.35	4.4	31036	26.25	25.02	4.9	4949	25.94	24.72	4.9
		22.58	21.89	3.2		26.87	25.69	4.6		26.39	25.14	5.0		25.91	24.74	4.7
		22.42	21.26	5.5		26.62	25.43	4.7		26.40	25.14	5.0		25.91	24.75	4.7
		22.70	21.51	5.5		26.63	25.43	4.7		26.19	24.96	4.9		25.87	24.68	4.8
		23.29	22.38	4.1		26.76	25.58	4.6		26.35	25.08	5.1		25.87	24.67	4.9
		23.24	21.97	5.8		26.73	25.53	4.7		26.38	25.13	5.0		25.97	24.74	5.0
		23.15	21.90	5.7		26.77	25.58	4.7		26.64	25.37	5.0		25.95	24.72	5.0
		22.87	21.66	5.6		26.58	25.40	4.6		26.54	25.26	5.1		25.79	24.56	5.0
		23.28	22.06	5.5		26.68	25.52	4.5		26.37	25.10	5.1		25.80	24.57	5.0
		23.34	22.14	5.4		26.76	25.56	4.7		26.23	24.95	5.1		25.93	24.72	4.9
	mean	22.96	21.83	5.2		26.69	25.51	4.6		26.37	25.12	5.0		25.89	24.69	4.9
	std. dev.	0.34	0.34	0.9		0.12	0.10	0.09		0.14	0.13	0.07		0.06	0.07	0.11
	95 % C.I.	0.24	0.24	0.6		0.09	0.07	0.06		0.10	0.09	0.05		0.04	0.05	0.08
5	n.s.	26.50	25.09	5.6	104390	26.76	25.67	4.2	31036	26.57	25.17	5.6	4046	26.14	24.79	5.4
		26.48	25.09	5.5		26.71	25.41	5.1								
		26.59	25.19	5.6		26.93	25.58	5.3								
		26.49	25.10	5.5		26.68	25.54	4.5								
		26.31	24.94	5.5		26.91	25.61	5.1								
		26.56	25.17	5.5		26.86	25.57	5.0								
		26.33	24.97	5.4		26.83	25.39	5.7								
		26.32	24.94	5.5		26.88	25.61	5.0								
		26.43	25.06	5.5		26.67	25.40	5.0								
		26.24	24.86	5.6		26.84	25.45	5.5								
	mean	26.43	25.04	5.5		26.81	25.52	5.0								
	std. dev.	0.12	0.11	0.05		0.10	0.10	0.42								
	95 % C.I.	0.09	0.08	0.04		0.07	0.07	0.30								
6	n.s.				n.s.				31036	26.38	25.06	5.3	8425	26.32	25.12	4.8
									31036	26.43	25.09	5.3	6357	25.77	24.49	5.2
													6357	26.32	25.12	4.8
	mean									26.41	25.08	5.3		26.14	24.91	4.9
	std. dev.									0.04	0.02	0.1		0.32	0.36	0.3
	95 % C.I.													0.79	0.89	0.10
7	n.s.				n.s.				n.s.				n.s.			
All	mean	26.07	25.04	5.0		26.60	25.49	4.3		26.41	25.11	5.2		26.00	24.82	4.7
	std. dev.	0.33	0.26	0.5		0.19	0.05	0.5		0.09	0.04	0.3		0.13	0.13	0.6
	95 % C.I.	0.82	0.65	1.2		0.30	0.08	0.8		0.11	0.05	0.3		0.14	0.14	0.7

* Lab 7 did not submit the requested paper specimens. **All these lot numbers carry the prefix FC00. *** Lot number not supplied

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5. Results

5.1. Round 1

The results from six laboratories are compiled in Table 3. (Lab 3 did not submit data.) Note that, for anonymity, the numbering of the labs has been randomized from the order that was presented in Section 3.1.

Each lab performed five sets of 40 determinations of SRM 1082 on a substrate consisting of 10 sheets of Paper 4 (Whatman No. 2, China), with the rough side facing up. In addition to the numbers of full-length burns observed in each laboratory, Table 3 contains the observed mean and standard deviation for each laboratory (columns 2 and 3), and results from a Bayesian analysis of the data[10,11] using a hierarchical beta-binomial model (columns 4 through 7). The Bayesian model allows for variation between and within laboratories. Results are given for each laboratory separately, as well as for the mean percentage of full-length burns across labs.

The Bayesian model used to describe the data is expressed mathematically as

$$\begin{aligned} p_i &\sim \text{Beta}(\alpha, \beta),\ i = 1, \ldots, n_L \\ B_i &\sim \text{Bin}(p_i, n_{R_i}),\ i = 1, \ldots, n_L \\ \alpha &\sim \text{Exp}(0.003) \\ \beta &\sim \text{Exp}(0.003) \end{aligned} \tag{1}$$

where p_i is the true proportion of full-length burns in the i^{th} lab, n_L is the number of labs ($n_L = 6$ here, since Lab 3 did not submit data in Round 1), α and β are parameters of a Beta probability distribution that describes the true variation in the full-length burn rates between labs, B_i is the total number of full-length burns observed over the 5 sets of 40 runs in the i^{th} lab, and n_{R_i} is the number of runs made in the i^{th} lab ($n_{R_i} = 200$ for all labs except for Lab 5, for which $n_{R_i} = 160$ due to the omission of the outlying result in the fit of the model). The values of B_i are each assumed to follow a binomial probability distribution with parameters p_i and n_{R_i}. The parameters α and β are each given the exponential prior distributions shown, which induces a uniform prior distribution on each value of p_i before the data is taken into account.

This model allows for both between and within lab uncertainty. Within each lab, the results depend mostly on the lab's data alone, but there is some "borrowing of strength" among labs which tends to pull each lab's estimate a bit toward the mean across labs. The mean result also includes uncertainty between and within labs and the between-lab uncertainty plays a more direct role in the uncertainty of the mean.

The model was fit to the data using the open-source software OpenBUGS[12,13], which uses a Markov Chain Monte Carlo simulation to determine posterior probability distributions for the values of each parameter. The results are based on 10000 simulated values, thinning each chain by 400 observations between observations that were kept. Each chain was given a burn-in of 5000 iterations to converge. The convergence of the simulation was assessed by running multiple simulations with different starting values and plotting the results between and within each

Markov chain using the Brooks-Gelman-Rubin plot.[13,14] The ability of the model to adequately describe the structure in the data was assessed using posterior predictive residual plots.

The first observation is that the results from five of the labs are quite self-consistent. The one notable exception is Lab 5, whose data are affected by the second set of 40 determinations, which showed a very high number of full-length burns. If one disregards that set, the Lab 5 results are the values in parentheses, which are consistent with the other laboratories.

Table 3. Round 1 Data: Repeat Tests of SRM 1082 Cigarettes on 10 Sheets of Whatman No. 2 Filter Paper (China) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (40 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	3, 3, 0, 2, 1	4.5	2.9	4.9	1.0	3.1	7.0
2	2, 1, 2, 0, 1	3.0	1.9	4.5	1.0	2.6	6.5
3							
4	0, 2, 1, 1, 2	3.0	1.9	4.5	1.0	2.6	6.5
5	3, 11, 3, 3, 3	11.5 (7.5)	8.0 (0.0)	5.6	1.1	3.7	8.2
6	5, 1, 4, 3, 1	7.0	4.0	5.6	1.1	3.8	8.0
7	3, 0, 1, 1, 5	5.0	4.5	5.0	1.0	3.3	7.1
Overall Mean PFLB and Uncertainties				5.0	0.8	3.6	6.6
				$r = 9.6$ %		$R = 10.1$ %	

The second observation is that the test results from all the laboratories are more than a factor of two below the expected value for SRM 1082 cigarettes tested on 10 layers of (UK) Whatman No. 2 filter paper (12.6 PFLB ± 3.3 PFLB).[15] It is highly unlikely that six test operators from different organizations would *all* obtain deviant results that were similarly that low by chance. It is also highly unlikely that six supplies of SRM 1082 cigarettes, obtained at different times, would all manifest similarly low test results. The working hypothesis is that the recent batches of Whatman No. 2 filter paper from China differ substantially from the historic batches of filter paper made in the UK. This is consistent with the prior indication in Reference 6.

The values of the repeatability and reproducibility limits for a test based on 40 replicate determinations are $r = 9.6$ % and $R = 10.1$ %, respectively. These values are determined using the formulas[ii]

$$r = 2.8\sqrt{m_{\mathrm{PFLB}}\left(100 - m_{\mathrm{PFLB}}\right)/40} \, ,$$

[ii] Originally, the values of repeatability and reproducibility were computed based on 95th percentiles of the predictive distribution for within- and between-lab differences in PFLB rates computed using the Bayesian model. However, on the suggestion of statistician Michael Morton from Altria, that approach was examined further and found to possibly have some issues with low bias for the computed values of *R*. The method used here differs from the originally suggested change only through the addition of a small correction for a high bias in the estimates of *R*. The bias correction enters the formula for *R* through the subtraction of the sample size term 1/C from the term for the prescribed sample size of the test at $n = 40$.

$$R = 2.8\sqrt{s_{\text{PFLB}}^2 + m_{\text{PFLB}}\left(100 - m_{\text{PFLB}}\right)\left(1/40 - 1/C\right)}\text{, and}$$

$$C = \frac{N^2 - \sum_{i=1}^{n_L} n_{R_i}^2}{N\left(n_L - 1\right)}$$

where $2.8 \approx 1.96\sqrt{2}$ is the multiplier used to determine *r* or *R* at the approximate 95 % confidence level, m_{PFLB} is the mean of the labs' overall PFLB rates[iii], and 40 is the sample size specified for determining the PFLB rate in E2187. The value s_{PFLB}^2 is the sample variance of the labs' overall PFLB rates[iii], n_{R_i} is the total number of cigarettes tested at each lab[iii], $N = \sum_{i=1}^{n_L} n_{R_i}$ is the total number of cigarettes tested across all labs[iii], and n_L is the number of labs reporting results[iii].

The fact that the values of *r* and *R* are essentially the same indicates that the variability among labs is significantly smaller than the intralaboratory variability, which is scaled to a sample size of 40 tests. These values of r and R are consistent with the values currently published in ASTM E 2187 for a cigarette with a true PFLB level similar to that observed here.

In summary, the *r* and *R* values indicate that the labs were performing the tests consistently, while the very low mean value relative to the certified value for SRM 1082 indicates that something non-procedural, like the nature of the substrate, has changed. This analysis was deemed sufficient to support a decision to proceed to Round 2 even though there were data from only six of the seven labs.

5.2 Round 2

The results from six laboratories are compiled in Table 4 based on a fit of the Bayesian model described in Section 5.1, except with $n_{R_i} = 100$ for all labs. (Lab 3 reported data for Round 2, but the data were not able to be used because the data were collected prior to the revision of the test procedures to minimize the effect of curvature of the sheet steel portion of the substrate on the results.) Each lab performed five sets of 20 determinations of SRM 1082 on a substrate consisting of a sheet of Metal 1 and one sheet of Paper 4 (Whatman No. 2, China), with the rough side facing up.

The values of the repeatability and reproducibility limits for a test based on 40 replicate determinations are $r = 15.1\ \%$ and $R = 18.2\ \%$, respectively. These values are determined using the formulas given near the end of Section 5.1. The fact that the two values are close again indicates that the variability among labs is significantly smaller than the intralaboratory variability in this case, though there may be a small amount of between-laboratory variability. These values of r and R are essentially consistent with the values currently published in ASTM E 2187, considering the previously discussed effect of testing with the rough side facing upward.

[iii] for a given combination of cigarette and substrate

Table 4. Round 2 Data: Repeat Tests of SRM 1082 Cigarettes on Metal 1 plus One Sheet of Whatman No. 2 Filter Paper (China) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	1, 5, 3, 4, 5	18.0	1.7	14.6	2.2	10.8	19.5
2	4, 2, 3, 2, 1	12.0	1.1	13.2	2.0	9.3	17.3
3							
4	3, 6, 2, 3, 2	16.0	1.6	14.1	2.1	10.4	18.5
5	3, 2, 1, 2, 4	12.0	1.1	13.2	2.0	9.3	17.4
6	0, 1, 2, 1, 1	5.0	0.7	11.7	2.3	6.9	15.8
7	1, 8, 3, 4, 2	18.0	2.7	14.6	2.2	10.8	19.3
Overall Mean PFLB and Uncertainties				13.6	2.1	10.6	17.0
				$r = 15.1$ %		$R = 18.2$ %	

The values of the repeatability and reproducibility limits for a test based on 40 replicate determinations are $r = 15.1$ % and $R = 18.2$ %, respectively. These values are determined using the formulas given near the end of Section 5.1. The fact that the two values are close again indicates that the variability among labs is significantly smaller than the intralaboratory variability in this case, though there may be a small amount of between-laboratory variability. These values of r and R are essentially consistent with the values currently published in ASTM E 2187.

In summary, the mean value and the 95 % expanded uncertainty interval for this combination of paper and metal are statistically indistinguishable from the certified values for the SRM 1082 cigarettes. The consistent r and R values indicate that the alternate substrate is not introducing a notable increase in the variability of test performance. The low values of the standard deviations in the posterior PFLB mean values further indicated that the six labs were following the revised (for the alternate substrate) test procedure similarly and repeatably. For these reasons, the ILS proceeded to Round 3.

5.3 Round 3

Each lab performed five sets of 20 determinations for each of four cigarettes on two substrates, as noted above in Section 3.2. Wherever discernable, the test was run with the rough side of the paper up for each substrate. The results from the seven laboratories are compiled in Table 5 through Table 12 based on fits of the Bayesian model and the other methods described in Section 5.1, except with $n_{R_i} = 100$ for all labs. Table 13 summarizes the results (columns 4 through 7) for all three rounds of the ILS.

Table 5. Round 3 Data: Repeat Tests of SRM 1082 Cigarettes on Substrate B (Metal 2 plus One Sheet of Filter Paper 2) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs*)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	2, 1, 3, 1, 4	11.0	6.5	12.1	3.0	6.8	18.5
2	2, 1, 1, 4, 5	13.0	9.1	13.6	3.1	8.2	20.1
3	3, 6, 5, 5, 4	23.0	5.7	21.1	3.7	14.3	28.8
4							
5	3, 5, 2, 4, 1	15.0	7.9	15.1	3.2	9.3	21.8
6	1, 0, 0, 0, 1	2.0	2.7	5.4	2.6	1.4	11.5
7	11, 3, 6, 6, 2	30.4	15.1	27.1	4.8	18.5	37.0
Overall Mean PFLB and Uncertainties				15.7	3.3	9.9	23.1
*For Lab 7, the run sizes are 20, 20, 20, 20, 9				$r = 16.2$ %		$R = 31.1$ %	

Table 6. Round 3 Data: Repeat Tests of SRM 1082 Cigarettes on Substrate C (Metal 3 plus One Sheet of Filter Paper 3) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	0, 1, 2, 2, 0	5.0	5.0	6.9	2.5	2.7	12.5
2	2, 3, 1, 1, 2	9.0	4.2	10.2	2.9	5.3	16.5
3	6, 6, 5, 7, 6	30.0	3.5	28.3	4.3	20.3	37.1
4	5, 4, 4, 6, 10	29.0	12.4	27.5	4.3	19.6	36.1
5	7, 7, 5, 8, 2	29.0	11.9	27.4	4.3	19.4	36.0
6	0, 0, 2, 1, 0	3.0	4.5	5.1	2.3	1.6	10.4
7	5, 5, 4, 5, 6	25.0	3.5	23.9	4.0	16.6	32.3
Overall Mean PFLB and Uncertainties				17.8	4.1	10.6	27.1
				$r = 17.2$ %		$R = 36.9$ %	

Table 7. Round 3 Data: Repeat Tests of Cigarette 2 on Substrate B (Metal 2 plus One Sheet of Filter Paper 2) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	2, 1, 0, 0, 1	4.0	4.2	4.6	1.1	2.6	7.1
2	0, 1, 2, 0, 3	6.0	6.5	4.9	1.2	2.9	7.6
3	0, 1, 1, 0, 0	2.0	2.7	4.3	1.2	2.1	6.7
4							
5	0, 1, 0, 0, 2	3.0	4.5	4.4	1.2	2.4	6.9
6	1, 1, 1, 1, 3	7.0	4.5	5.1	1.3	3.0	7.9
7	2, 0, 0, 1, 2	5.0	5.0	4.8	1.2	2.7	6.7
Overall Mean PFLB and Uncertainties				4.7	1.0	3.0	6.7
				$r = 9.6$ %		$R = 8.7$ %	

Table 8. Round 3 Data: Repeat Tests of Cigarette 2 on Substrate C (Metal 3 plus One Sheet of Filter Paper 3) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	0, 0, 1, 0, 1	2.0	2.7	2.5	0.8	1.1	4.4
2	0, 0, 1, 1, 0	2.0	2.7	2.5	0.9	1.1	4.4
3	0, 3, 0, 0, 1	4.0	6.5	2.9	0.9	1.4	4.9
4	0, 1, 1, 0, 0	2.0	2.7	2.5	0.9	1.1	4.4
5	0, 1, 2, 1, 1	5.0	3.5	3.0	1.0	1.5	5.3
6	0, 0, 1, 0, 1	2.0	2.7	2.5	0.9	1.1	4.4
7	0, 0, 0, 0, 0	0.0	0.0	2.1	0.8	0.6	3.9
Overall Mean PFLB and Uncertainties				2.6	0.7	1.5	4.1
				$r = 6.8$ %		$R = 7.0$ %	

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Table 9. Round 3 Data: Repeat Tests of Cigarette 3 on Substrate A (Metal 1 plus One Sheet of Filter Paper 1) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	0, 0, 0, 1, 1	2.0	2.7	4.1	1.7	1.1	7.6
2	2, 1, 1, 1, 3	8.0	4.5	7.0	2.0	3.8	11.6
3	0, 0, 1, 0, 2	3.0	4.5	4.6	1.7	1.6	8.0
4	1, 2, 1, 1, 5	10.0	8.7	7.9	2.2	4.5	13.1
5	1, 2, 4, 3, 4	14.0	6.5	9.9	2.8	5.5	16.3
6	0, 1, 1, 1, 3	6.0	5.5	6.0	1.8	3.0	9.9
7	0, 0, 0, 0, 0	0.0	0.0	3.1	1.8	0.2	6.8
Overall Mean PFLB and Uncertainties				6.0	1.5	3.4	9.1
				$r = 10.6$ %		$R = 16.0$ %	

Table 10. Round 3 Data: Repeat Tests of Cigarette 3 on Substrate D (10 Layers of Whatman Filter Paper (China)) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	0, 1, 2, 0, 1	4.0	4.2	7.3	1.6	4.2	10.4
2	3, 3, 2, 2, 3	13.0	2.7	9.3	1.8	6.3	13.4
3	0, 1, 1, 0, 4	6.0	8.2	7.8	1.6	4.8	11.0
4	4, 1, 1, 0, 1	7.0	7.6	8.0	1.6	5.1	11.3
5	1, 0, 3, 2, 4	10.0	7.9	8.6	1.6	5.8	12.2
6	1, 0, 1, 1, 1	4.0	2.2	7.3	1.6	4.1	10.6
7	3, 2, 2, 3, 3	13.0	2.7	9.3	1.8	6.3	13.4
Overall Mean PFLB and Uncertainties				8.2	1.2	6.0	10.8
				$r = 12.1$ %		$R = 14.4$ %	

Table 11. Round 3 Data: Repeat Tests of Cigarette 4 on Substrate 1 (Metal 1 plus One Sheet of Filter Paper 1) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev. PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	0, 0, 0, 0, 0	0.0	0.0	1.7	1.4	0.0	5.0
2	0, 2, 1, 1, 0	4.0	4.2	4.7	1.9	1.6	9.2
3	3, 0, 0, 1, 0	4.0	6.5	4.7	1.9	1.7	9.1
4	2, 2, 4, 2, 4	14.0	5.5	12.5	3.1	7.1	19.1
5	4, 3, 3, 3, 8	21.0	10.8	18.0	3.8	11.2	26.0
6	2, 2, 2, 3, 1	10.0	3.5	9.4	2.7	4.9	15.3
7	0, 1, 0, 1, 0	2.0	2.7	3.2	1.7	0.7	7.2
Overall Mean PFLB and Uncertainties				7.3	2.4	3.5	12.7
				$r = 11.9$ %		$R = 23.0$ %	

Table 12. Round 3 Data: Repeat Tests of Cigarette 4 on Substrate 4 (10 Layers of Whatman Filter Paper (China)) with Associated Repeatability and Reproducibility Limits.

Lab	Full-length Burns (20 Runs)	Observed Mean PFLB	Observed Std. Dev PFLB	Posterior PFLB Mean	Std. Dev. Posterior PFLB Mean	95 % PFLB Lower Bound	95 % PFLB Upper Bound
1	0, 2, 3, 0, 0	5.0	7.1	9.9	1.9	5.9	13.5
2	3, 4, 4, 2, 4	17.0	4.5	12.6	2.1	9.1	17.3
3	2, 3, 4, 1, 2	12.0	5.7	11.4	1.8	8.1	15.3
4	2, 0, 3, 4, 2	11.0	7.4	11.2	1.8	7.8	14.9
5	5, 3, 1, 4, 4	17.0	7.6	12.5	2.1	9.1	17.2
6	2, 2, 3, 1, 1	9.0	4.2	10.8	1.8	7.3	14.5
7	1, 2, 1, 3, 0	7.0	5.7	10.3	1.9	6.5	13.9
Overall Mean PFLB and Uncertainties				11.2	1.4	8.7	14.1
				$r = 13.9$ %		$R = 16.9$ %	

Table 13. Summary of Ignition Propensity Data for the Three ILS Rounds.

	Round									
	1	2	3							
Cig. No.→	1	1	1		2		3		4	
Substrate	D	E	B	C	B	C	A	D	A	D
Metal No.	-	1	2	3	2	3	1	-	1	-
Paper No.	4	4	2	3	2	3	1	4	1	4
Mean PFLB	5.0	13.6	15.7	17.8	4.7	2.6	6.0	8.2	7.3	11.2
Std. Dev.	0.8	2.1	3.3	4.1	1.0	0.7	1.5	1.2	2.4	1.4
Lower C.I.	3.6	10.6	9.9	10.6	3.0	1.5	3.4	6.0	3.5	8.7
Upper C.I.	6.6	17.0	23.1	27.1	6.7	4.1	9.1	10.8	12.7	14.1
r*	9.6 %	15.1 %	16.2 %	17.2 %	9.6 %	6.8 %	10.6 %	12.1 %	11.9 %	13.9 %
R*	10.1 %	18.2 %	31.1 %	36.9 %	8.7 %	7.0 %	16.0 %	14.4 %	23.0 %	16.9 %
Table No.	3	4	5	6	7	8	9	10	11	12

* The values in ASTM E 2187 are approximately 13 % and 16 % for a cigarette with a true PFLB rate of 10 % or 90 %.

Cigarette 1 is SRM 1082; Paper 4 is Whatman No. 2 (China).

6. Substrate Performance

6.1 Test Precision

There are some general observations regarding the repeatability and reproducibility values in Table 13.

- The *r* values in all three rounds were generally consistent with each other and with the repeatability limits in ASTM E 2187. The tests involving Cigarette 1 might show modestly higher intralaboratory variability than the overall average, and the tests involving Cigarette 2 might show slightly lower intralaboratory variability.
- The between-laboratory variability, *R*, for the two sets of Cigarette 1 tests in Round 3 (columns 4 and 5) was substantially larger than for any of the other data sets. To a lesser degree, the *R* value for Cigarette 4 on Substrate A was also high.
- Each *R* value in the other columns was essentially consistent with the *r* value in the same column, with the possibility of a small amount of extra random variation being present between labs over that observed within labs.

Table 14 summarizes the nature of the PFLB values for each of the labs for each combination of cigarette and substrate. An “H” indicates that the lab’s results were above the upper bound of the 95 % expanded uncertainty interval for that cigarette/substrate combination. An “L” indicates that the lab’s results were below the lower bound of the 95 % expanded uncertainty interval for that combination. The absence of a character indicates that the lab’s results were within the 95 % expanded uncertainty interval for that combination. Note that the purpose of the 95 % uncertainty intervals for each mean is not to capture the individual lab results. This characterization of the individual laboratory results is simply meant to highlight the nature and persistence of any laboratory effects across the different rounds of testing.

Examining the rows in the table, three of the labs (2, 3, 6, and 7) were balanced in that their results were high about as often as they were low. Lab 1 generated results that were generally low, while results from Labs 4 and 5 were often high. Overall, this is not a problematic distribution since the results from most labs do not seem to be consistently high or low.

Looking at the columns of results in the table, four of the ten have 2 or 3 L’s or H’s, while the remaining six have 4 or more L’s or H’s. This split is controlled by clustering in the results. When the results cluster around two or three well-separated values, then the center of the uncertainty interval, and sometimes even the uncertainty bounds, fall between the clusters of points. This means that a relatively large number of data points will fall outside the interval. As noted above, however, these uncertainty intervals are not designed to capture a specified proportion of individual results, so this behavior does not seem problematic either. Clusters can, and will, occur by chance in the data naturally and this effect is likely to be a byproduct of that random behavior.

Table 14. Relative Results for All Three ILS Rounds for the Seven Laboratories.

	Round									
	1	2	3							
Cig. No.→	1	1	1		2		3		4	
Substrate	D	E	B	C	B	C	A	D	A	D
Metal No.	-	1	2	3	2	3	1	-	1	-
Paper No.	4	4	2	3	2	3	1	4	1	4
Lab 1		H		L			L	L	L	L
Lab 2	L			L			L	H		H
Lab 3				H	L			L		
Lab 4	L			H			H		H	
Lab 5	H			H		H	H		H	H
Lab 6	H	L	L	L	H			L		
Lab 7		H	H			L	L	H	L	L

6.2 Ignition Propensity of SRM 1082

The first objective of the alternate substrate was that testing on it produces ignition propensity values that were statistically indistinguishable from those obtained from testing on 10 layers of Whatman No.2 (UK) filter paper. Figure 4 shows the difference between the measured PFLB value and the certified value for SRM 1082 for testing on four substrates: Round 1 (Substrate D), Round 2 (Substrate E), and Round 3 (Substrates B and C). The uncertainty bars account for the uncertainty in both the ILS measurements and the certified ignition propensity of SRM 1082. The overlap of the uncertainties with zero on the ordinate for the left three data points indicated that the differences between the ILS values and the certified values are not statistically significant for the ILS testing on three different metal/paper substrates in Rounds 2 and 3. By contrast, the mean value from Round 1 (testing on 10 layers of Whatman No. 2 (China) filter paper is quite different from the certified value. (Recall that in the certification testing, the upward facing surface of the filter paper was not specified. Thus, the ordinate values in Figure 4 might be slightly high, which would improve the agreement between the results from the metal/paper substrates and the result from 10 layers of Whatman No. 2 (UK) filter paper.)

These results support the hypothesis that testing using the alternate substrate produced ignition propensity values that were not significantly different from those obtained from testing on 10 layers of Whatman No.2 (UK) filter paper. The result from Round 1 suggests that the Whatman No. 2 (China) filter paper manufactured during the period when the ILS labs procured their supplies performed differently from the Whatman No. 2 paper previously manufactured in the UK.

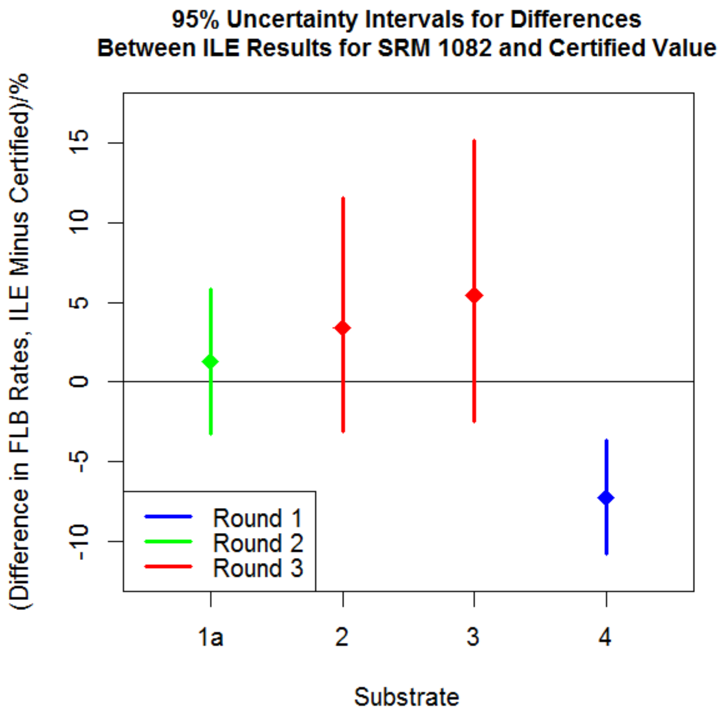

Figure 4: Differences between Mean Results for SRM 1082 on the Different Substrates Used in This ILS and Its Certified Value (Based on Tests on 10 Layers of Whatman #2 (UK) Filter Paper.

6.3 Dependence on the Substrate Materials

The presentation in Figure 4 indicates that testing of SRM 1082 cigarettes on substrates composed of three different metal/paper combinations generated FLB data that are not significantly different from each other. To help ensure that these results are robust across different brand styles of cigarettes, comparisons were made with three additional cigarettes as well (Cigarettes 2, 3, and 4). There were differences in the between-lab variability in Round 3, so a paired test was used to compare substrates. Given the binomial nature of the measurements the appropriate paired test is a nonparametric sign test.[16]

This test involves comparison of the PFLB values from each of the seven labs for a particular cigarette on two different substrates. The test statistic is the number of paired differences greater than zero. (The statistic also could be based, equivalently, on the number of paired differences less than zero.) Under the null hypothesis of no difference in FLB rates, the negative and positive values should be evenly distributed, with approximately half being above zero and half being below zero. A significant difference in the FLB rates on the two substrates being compared is indicated by extreme numbers of positive (or negative) paired differences. For seven labs, the rejection region (RR) for the test can be set as either:

RR = {0,7} (confidence level 98 %), or

RR = {0,1,6,7} (confidence level 87.5 %)

For six labs, the rejection region (RR) for the test can be set as either:

RR = {0,6} (confidence level 96.9 %), or

RR = {0,1,5,6} (confidence level 78.1 %)

These comparisons are depicted in Figure 5 for four combinations of cigarette and substrate:

- Cigarette 1 on Substrates B and C
- Cigarette 2 on Substrates B and C
- Cigarette 3 on Substrates A and D
- Cigarette 4 on Substrates A and D

As shown in Figure 5, in each of the four comparisons, either three or four of the points are above the abscissa and the remaining points are below the abscissa. The positive and negative groupings are as close to being even as possible, given the odd number of laboratories. Thus, there is no statistical evidence of difference between the substrates based on this test.

One potential disadvantage of nonparametric tests like the paired sign test is lack of efficiency for detecting differences relative to parametric tests. In this case, however, the absolute power of this sign test, shown in Figure 6, seems adequate to detect meaningful differences in the full-length burn rates of a cigarette on different substrates. The power curve shown in Figure 6 is based on the mean PFLB values for cigarette 1 on substrate B taken from Table 5. As an example of the power of this test with this configuration of full-length burn rates, the probability of detecting a 10 % difference between the substrates is about $p_{\text{detect}} = 0.80$.

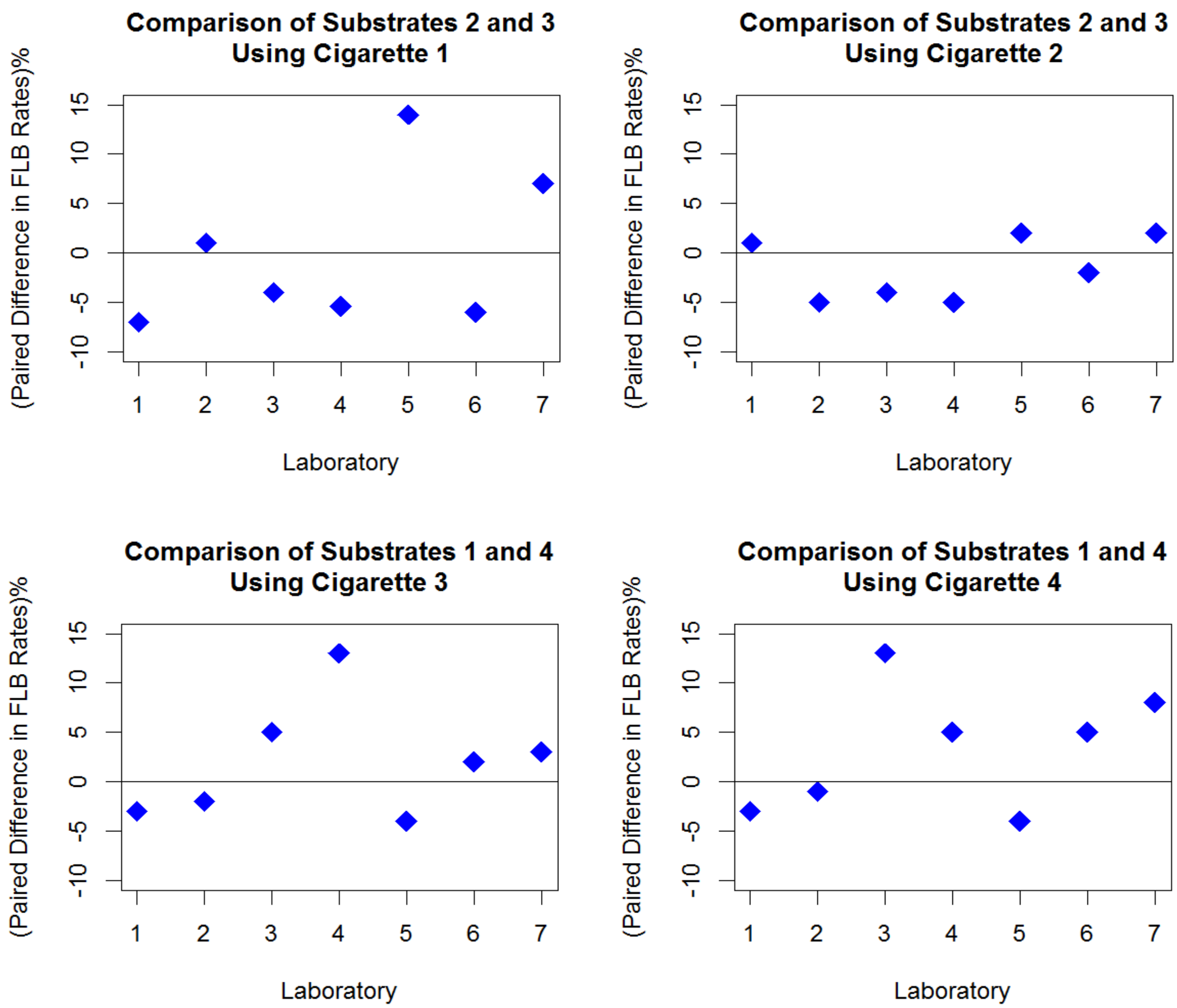

Figure 5. Pairwise Comparison of PFLB Values from Seven Laboratories for a Particular Cigarette on Two Substrates. Top left: Cigarette 1 on Substrates B and C; top right: Cigarette 2 on Substrates B and C; bottom left: Cigarette 3 on Substrates A and D; bottom right: Cigarette 4 on Substrates A and D.

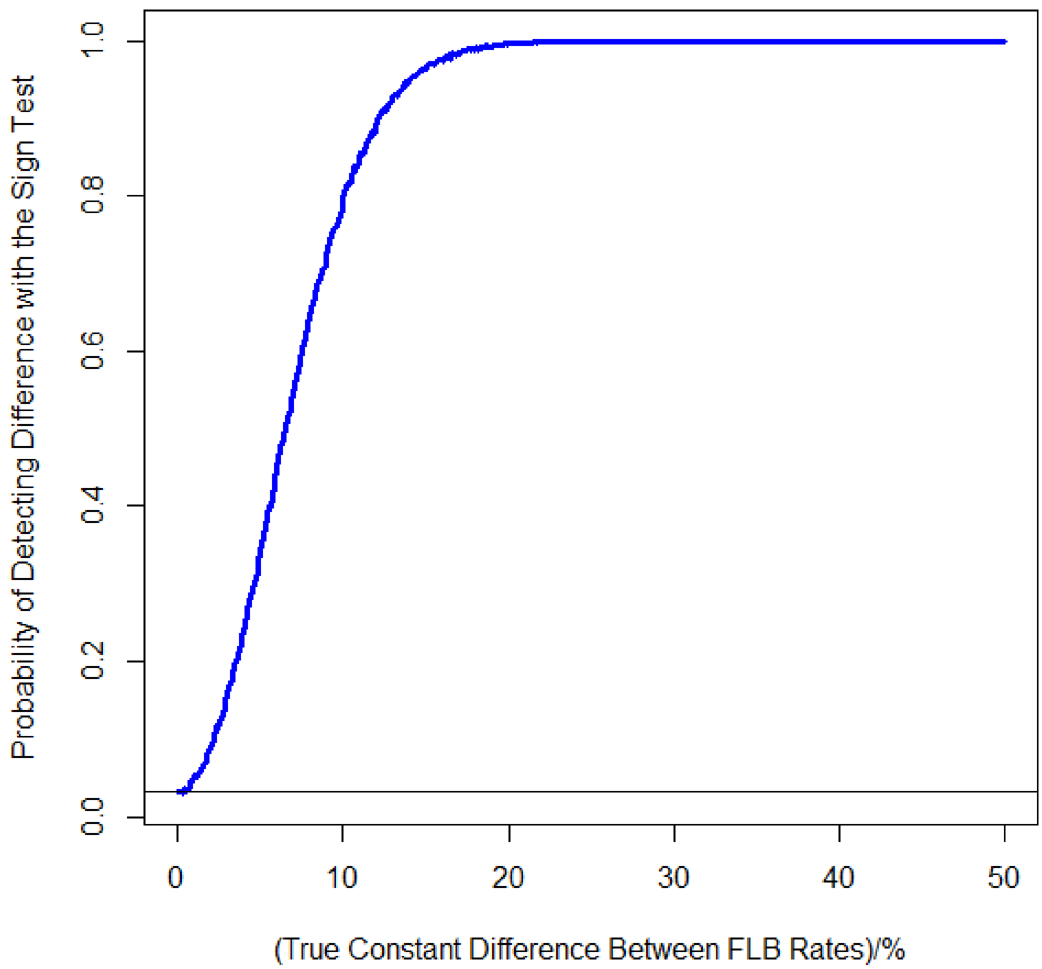

Figure 6. Power curve for the sign test computed via Monte Carlo simulation. The FLB rates used in the computation were based on the mean values for cigarette 1 on substrate B from Table 5.

7. Conclusions

- The ignition propensity results obtained using the steel/paper substrates were not statistically distinguishable from the result obtained for testing on 10 layers of Whatman No. 2 (UK) filter paper. Combining the results from columns 2, 3, and 4 in Table 13, the mean value and its standard error are (15.9 ± 4.7) PFLB. The expanded uncertainty bounds at the 95 % probability level are (8.6, 26.9) PFLB. The certified values for SRM 1082 (tested on 10 layers of Whatman No.2 (UK) filter paper) are (12.6 ± 3.3) PFLB.
- Recall that in the certification testing, the upward facing surface of the filter paper was not specified. Had the testing been conducted with the rough side of the paper always facing upward, the PFLB mean value would have been slightly higher. This would modestly improve the agreement between the results from the metal/paper substrates and the result from 10 layers of Whatman No. 2 (UK) filter paper.
- There were no statistically significant effects of the substrate materials on the test results, for those combinations of cigarette, paper, and steel that were tested. As a result, the proposed specifications for the substrate materials are:
    - Paper
        - Mass of 15 sheets, dried at (60 ± 2) °C for at least 16 h: (24.9 ± 0.7) g. The standard deviation of five such samples is to be no more than 0.3 g.
        - Mass of 15 sheets, conditioned at (23 ± 3) °C and (55 ± 5) % relative humidity for at least 24 h: (26.2 ± 0.5) g. The standard deviation of five such samples is to be no more than 0.3 g.
        - Moisture content of the conditioned paper, relative to the dried paper: (0.050 ± 0.006) mass fraction.
    - Steel
        - Full hard 302 stainless steel shim stock
        - Thickness: (0.203 ± 0.004) mm
- The batches of Whatman No.2 (China) filter paper in this ILS appear to be different from the paper formerly manufactured in the UK. The mean test value and standard deviation for SRM 1082 cigarettes from the six reporting labs in this ILS was (5.0 ± 0.8) PFLB, compared to the certified value based on tests on 10 layers of Whatman No. 2 (UK) filter paper of (12.6 ± 3.3) PFLB. It is not yet known whether this low value represents all Whatman No. 2 (China) filter paper or just the batches manufactured at the time of this ILS.

8. Acknowledgements

The ILS success was the result of the active participation of the staff of the seven laboratories listed in Section 3.1 of this report. The participants appreciate the effort of the NIST Fabrication Technology Group for making the thickness measurements of the 302 stainless steel shim stock.

9. References

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Appendix A. ILS Modifications to the Test Procedure in ASTM E 2187-09

In the following, new text is shown in **boldface**.

Add a new Section 7.4.2:

An adapter ring to support the stainless steel substrate shall be made of polymethylmethacrylate (PMMA), or a similarly rigid material, dimensioned as follows. The outer diameter shall be 165 ± 1 mm (6.50 ± 0.04 in.), the inner diameter shall be 126 ± 1 mm (4.98 ± 0.04 in.), and the height shall be 15.5 ± 1 mm (0.61 ± 0.04 in.). A recess in the bottom, 10.0 ± 1 mm (0.40 ± 0.04 in.) deep, shall decrease the outer diameter to 150 ± 1 mm (5.90 ± 0.04 in.). The top surface of the adapter shall be flat to within ± 0.03 mm (0.001 in.).

Modify Section 7.5 to read as follows:

7.5 *Metal Rim*—A circular brass or other dense metal rim, shown in Fig. 2, shall be used to hold the sheets of filter paper flat against each other. The outside diameter of the rim shall be 150 ± 2 mm (5.90 ± 0.08 in.). The inner diameter shall be 130 ± 2 mm (5.1 ± 0.08 in.). The thickness shall be 6.4 ± 1 mm (0.25 ± 0.04 in.). **The mass of the rim shall be 302 g ± 5 g.** The rim surface shall be flat and smooth. A pair of parallel metal pins, each approximately 1 mm in diameter and 8.1 ± 0.05 mm (0.32 ± 0.02 in.) apart, shall protrude 17 ± 4 mm (0.65 ± 0.15 in.) toward the center of the rim. The pins are to be spaced to keep the non-ignited end of a conventional 25 mm circumference cigarette from rolling, but without pressuring the cigarette. If cigarettes of significantly different diameter are to be tested, other pairs of pins, appropriately spaced, shall be inserted into the rim. **When using the stainless steel substrate, an evenly distributed weight shall be used to provide a sufficient load for flattening any residual curvature in the stainless steel plate. This weight shall have a mass equivalent to the metal rim described in Section 7.5, and shall be placed on top of the metal rim.**

NOTE: It is suggested to use an object identical to the metal rim in Section 7.5. The metal pins are not necessary.

Modify Section 9.1 to read as follows:

9.1 Cigarette test specimens and **the** filter paper **and stainless steel** substrate **materials** are sensitive to contamination. Test cigarettes shall be handled only by the last nominal 25 mm (1 in.) of the end of the cigarette that is not to be lit. The sheets of filter paper **and the stainless steel sheets** shall not be handled in the vicinity where the cigarette will contact the paper **and steel** during a test. In all cases, the materials shall be handled with dry hands only.

NOTE 3—The use of clean, dry, non-powdered surgical gloves can mitigate incidental contamination of the test materials while maintaining operator dexterity.

Modify with re-numbering, Section 9.3 as follows:

9.3 ***Substrates***

9.3.1 The **filter paper** substrates consist of nominal 150 mm (6 in.) diameter circles of Whatman #2 ash-free cellulosic filter paper. Substrates are formed by placing multiple layers of filter paper into the holder assembly, then placing the metal rim on top to ensure good contact between the layers. All sheets of filter paper shall be oriented with the rough surface facing upward, **where discernible**.

9.3.2 The paper/steel substrate consists of a single sheet of nominal 150 mm (6 in.) diameter circle of filter paper centered on top of a nominal 159 mm (6.25 in.) x 150 mm (6.0 in.) rectangle of 302 stainless steel shim stock. The substrate is formed by centering the 302 stainless steel sheet on the adapter ring, centering a sheet of filter paper on top of the steel, centering the metal rim on top of the filter paper, and then placing the metal rim weight on top. The steel sheet shall be oriented concave down, when discernible. The sheet of filter paper shall be oriented with the rough surface facing upward, when discernible.

NOTE 4—In an interlaboratory study of alternate filter papers for use in ASTM E2187, data have indicated that for some cigarette designs, slightly different test values could be obtained depending on whether the rough surface or the smooth surface of the Whatman No. 2 filter paper was facing upward.

9.4 Filter Paper

9.4.1 For paper from a manufacturer's batch to be used in **the filter paper substrates**, the mean mass of 15 sheets of the conditioned filter paper shall be 26.1 ± 0.5 g. This shall be determined by weighing five samples of 15 sheets, each sample being from a different box from the manufacturer's batch. The standard deviation of the five samples shall be no more than 0.3 g. **For paper from a manufacturer's batch to be used in the stainless steel/filter paper substrates, the mean mass of 15 sheets of the conditioned filter paper shall be 26.1 ± 0.6 g, determined in the same manner.**

9.4.2 For paper from a manufacturer's batch to be used in **the filter paper substrates**, the mean mass of 15 sheets of the dried filter paper shall be 24.7 ± 0.5 g. This shall be determined by weighing five samples of 15 sheets, each sample being from a different box from the manufacturer's batch. Each set of 15 sheets shall have been stored at 60 ± 2°C for at least 16 h, placed in a sealed plastic bag upon removal from the oven, cooled to 23 ± 3°C, and weighed within 3 min of opening the bag. The standard deviation of the five samples shall be no more than 0.3 g**. For paper from a manufacturer's batch to be used in the stainless steel/filter paper substrate, the mean mass of 15 sheets of the conditioned filter paper shall be 24.8 ± 0.6 g, determined in the same manner.**

9.5 Stainless Steel

The 302 stainless steel layer shall consist of a nominal 159 mm x 150 mm (6.25 in. x 6 in.) rectangle of 302 stainless steel shim stock. The piece shall be flat, not from a roll. The thickness shall be 0.207 mm ± 0.002 mm (0.00800 in. ± 0.00008 in.) thick. The flatness shall be determined according to Section 11.3.

Re-number Section 10, adding a new Section 10.2 as follows:

10.2 The stainless steel sheets shall be individually (i.e., not stacked) conditioned at a temperature of 23 °C ± 3 °C (73 °F ± 5 °F) for at least 2 h prior to testing.

Modify Section 11 as follows:

11. Procedure

11.1 Turn on the exhaust system designated for removal of test combustion products 30 min prior to beginning testing.

11.2 Ensure that the filter paper holder**, with the adapter ring for the stainless steel substrate,** is in the test chamber at the geometric center of its bottom. Cover the chimney on the test chamber.

11.**3 If using filter paper substrates, c**onduct the test beginning with 15 layers of filter paper except as indicated in Annex A1. Select the number of layers of filter paper for the scheduled determinations using the procedure in Annex A1. All sheets of filter paper shall be oriented with the rough surface facing upward. **If using the stainless steel/filter paper substrate, ensure that the stainless steel sheet is sufficiently flat as follows. Place the sheet concave downward on a surface that is flat to within 0.025 mm (0.001 in.) over an area of at least 200 mm x 200 mm (7.9 in. x 7.9 in.). The sheet is not sufficiently flat if a 2 mm diameter precision ground steel rod, in contact with the flat surface, can be inserted between the sheet and the flat surface at any point on the perimeter of the sheet.**

NOTE: Some suggested flat surfaces are granite plates of at least 25 mm (1 in.) thickness or aluminum tool and jig plates of at least 19 mm (0.75 in.) thickness.

11.3.1 If the relative humidity and temperature in the test room cannot be maintained within the specified ranges, the **substrate materials** and cigarettes shall be sealed in plastic bags in the conditioning room and transported. Care shall be taken to ensure that test materials are protected from physical damage during transport and prior to use.

11.3.2 Immediately before testing **with a filter paper substrate**, place the proper number of filter papers on the filter paper holder and place the metal test rim on top. Discard filter papers that will not lay flat**. When testing using the stainless steel/filter paper substrate, place the steel sheet and the filter paper on the adapter ring and place the metal rim and the circular weight on top.**

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11.5.3 If the cigarette self-extinguishes while in the cigarette holder, terminate the determination and record the results as a self-extinguishment, noting that this occurred in the holder. This attempt shall count as a valid determination. The test operator shall be permitted to re-use this **substrate**. However, if the room is not at the standard conditioning temperature and humidity (see 7.1), the **substrate materials shall first** be reconditioned in a constant humidity box (see 7.2).

11.5.4 When the cigarette has burned to the 15 mm mark, simultaneously cover the chimney and open the chamber door, gently remove the cigarette from the holder, and move the holder to the front corner of the test chamber.

11.5.5 Gently lay the cigarette with the ash still attached onto the top of the filter paper so that the non-ignited end is placed between the appropriately sized cigarette anti-roll parallel metal pins. (Fig. 2). The cigarette paper seam shall be turned up. Do not drop the cigarette onto the **substrate** and do not press the coal into the **substrate**. If the ash falls off during any part of the transport or positioning process, terminate the determination and begin again; do not count the attempt.

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11.10 **The stainless steel substrate shall be cleaned after each determination. Lay the stainless steel substrate on the clean, flat surface used in Section 11.3. Using a soft laboratory wipe that has been wetted with ethanol or isopropanol, gently wipe the top surface of the stainless steel to remove any char residue or other contaminants. After carefully removing the stainless steel substrate from the flat surface, wipe the flat surface to remove any residual contamination. Ensure that the stainless steel substrate is dry before proceeding with the next determination.**