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# Interlaboratory Study of Alternate Filter Papers for Use in ASTM E 2187



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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. For decades, a specific type of filter paper has been used as the test substrate, with specified values of mass and moisture uptake used to define acceptable material. There is interest in the availability of alternate makes of filter paper, and an interlaboratory study (ILS) of three of these was performed to identify the potential for any of these to provide test results (a) indistinguishable from the currently specified filter paper and (b) with no degradation of repeatability. Each of the three alternate filter paper types demonstrated test result repeatability similar to that obtained using the current paper. One of the three alternate papers also showed indistinguishable test results for all test cigarettes. However, significant additional testing is 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. In addition, the ILS confirmed test result sensitivity to the upward facing surface of the papers, and led to recommendation that the standard test method be modified so that future tests will be performed with the choice of upward facing paper surface prescribed.

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

#### DISCLAIMER

The research on reduced ignition propensity cigarettes conducted by 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.

## Interlaboratory Study of Alternate Filter Papers for Use in ASTM E 2187

## 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. This Standard is cited in regulations in 49 U.S. States, Canada, Australia, New Zealand, and Finland. The European Union has requested that ISO develop a version of the ASTM Standard that can be cited in a European Standard, and that process is underway. The current version of the ASTM Standard is ASTM E 2187-04.<sup>1</sup>

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 2187-04. Top: full-length burn of a non-filter cigarette; Left: full-length burn of a 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 result 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.<sup>2</sup> 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<sup>1</sup> was used in the test substrates. This was justified on the basis that this was the prevalent filter paper available in the United States.

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 in ASTM E 2187-04.

Since there are now 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. 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  $\pm$  3.3 PFLB when tested on 10 layers of filter paper according to ASTM E 2187-04. Note that to minimize the need for users of SRM 1082 to account for the uncertainty of the certified value of the SRM when assessing their results when using it, 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.

<sup>&</sup>lt;sup>1</sup> Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.

## 2. Preliminary Testing of Alternate Filter Papers

With the advent of pending regulation of cigarette ignition strength using ASTM E 2187, the amount of cigarette testing increased rapidly. This led to a new and significant market for filter paper. Three additional companies expressed interest in being qualified for use in ASTM E 2187: Ahlstrom, Tervakoski, and Tela-Kimberly (Swiss Quality). The first two companies had products that were already commercially available; at the time, it was ascertained that a product from Tela-Kimberly would become available should their prototype be deemed equivalent to the Whatman No. 2 paper. Some cigarette companies and the State of New York expressed interest in having alternate, equivalent brands of filter paper available.

Preliminary data from R.J. Reynolds and Schweitzer-Mauduit, reported at the December 2006 meeting of ASTM E05.15, led to expectation of success for the alternate paper products in matching the performance of the Whatman product. The properties of the paper were reported as similar in mass and smoothness, with some differences in density and thickness. For three commercial, less fire-prone cigarettes and a test cigarette (approximately 50 PFLB on 10 layers of Whatman Certified filter paper), the repeatability and the PFLB values on both 3 and 10 layers of filter paper were encouragingly similar.

One surface of each sheet of the Whatman No. 2 filter paper is rougher than the other. During this preliminary testing, R.J. Reynolds reported small, but significant differences in the PFLB values depending on which side was facing up, and thus was in contact with the cigarette. Testing with the rough side up generated higher PFLB values, presumably because the cigarette contact with the heat sink was less efficient than the cigarette contact with the smooth side of the paper. This suggested that the paper orientation was a variable that should be examined in further studies.

## 3. The Interlaboratory Study (ILS)

With input from discussions with potential participants, the National Institute of Standards and Technology (NIST) prepared a draft plan for an ILS of the three alternate filter papers. A meeting of interested participants was held to review and improve the draft. This led to a final plan dated February 19, 2008.

Nine laboratories agreed to participate in the ILS:

- o R.J. Reynolds Tobacco Co.
- o Lorillard Tobacco Co.
- o Schweitzer-Mauduit International
- Philip Morris USA
- o Arista Laboratories
- o Kidde-Fenwal (for New York State)
- o NIST

- o Labstat
- National Research Council of Canada (NRC-C)

The following is the content of the plan.

#### **GENERAL PRINCIPLES**

The objective of the ILS is to determine whether the use of any of the three additional vendors' filter papers, when meeting the mass specifications in the test method, leads to test results that

(a) are indistinguishable from those obtained using the specified Whatman No. 2 filter paper and

(b) do not degrade the repeatability of the method.

The intent is to assess the similarity of the alternate papers for as many cigarette designs as possible. Assessing interlaboratory reproducibility is not central to satisfying this intent.

The three manufacturers of alternate filter paper are Tervakoski (Finland), Ahlstrom (U.S.), Tela-Kimberly (Swiss Quality) (Switzerland). The first two companies offer commercial products of this nature. The third company would prepare the 150 mm diameter rounds from commercially available strips.

The test method uses 3, 10, and 15 layers of filter paper as substrates. However, it appears that there is no planned use for the 15 layer substrate in routine testing. Thus, the ILS includes testing on just 3 and 10 layers of filter paper.

The ILS will be conducted in three rounds. For each participating laboratory, the total number of determinations would be approximately 1640.

The preferred number of participant laboratories is nine, as is explained in the test plan below. Should the number of participants fall below nine, there are two ways to meet the ILS objective, should the partial results be positive:

- One or more laboratories would test all three alternate filter paper types (preferred).
- A "reserve" laboratory might be available.

Each laboratory will receive 5200 sheets of each of the alternate types of paper it will examine. This is enough for all the tests and the spares for Round 3.

Each laboratory will use no more than two test operators. If two operators are used, one will do all tests on 3 layers of paper and one will do all tests on 10 layers for paper in both rounds 2 and 3. Each operator may use a separate set of test boxes, but the operators will not switch between the sets of boxes. Each laboratory should notify the ILS coordinator of the number of operators that will participate and the number of test boxes each will operate.

#### CONDUCT OF TESTING

**<u>Round 1</u>**: Each laboratory will conduct 40 determinations using a single cigarette and substrate. Total: 40 determinations.

- Purpose: Assure that each laboratory is performing the test properly.
- Each determination will use 10 sheets of mass-selected (or mass-certified) Whatman No. 2 paper.
- Each laboratory will independently obtain its supply of filter paper, recording the batch number.

- The cigarette, supplied by NIST, will be one for which the PFLB in the test is known. NIST will supply the cigarettes at least a few days prior to the visit by the Study Monitor.
- The Study Monitor would observe the testing, direct corrections of procedural errors, and be empowered to disqualify a laboratory should problems persist.
- To speed completion of this preliminary round and to minimize travel, a laboratory could send data indicating staff and facility capability to perform the test well.
  - These data would include (a) at least five tests (40 determinations each) of SRM 1082, to indicate the capability to reproduce a reference value and repeatability of the percent of full length burns (PFLB), and, if the tests of SRM 1082 cigarettes are not subsequent to approximately July 2007, (b) at least five tests (40 determinations each) of some other cigarette to indicate the repeatability required in the Standard. The Study Monitor will not visit laboratories with successful submissions.
  - The Study Monitor will work with the remaining laboratories to schedule observation visits.

**<u>Round 2</u>**: Each laboratory will perform 6 sets of 40 determinations of a single cigarette on 3 and 10 layers of mass-selected (or mass-certified) Whatman No. 2 filter paper. Total: 480 determinations.

- Purpose: Determine that each laboratory is operating with the appropriate precision and check that the cigarette designs chosen by each laboratory do not have extreme ignition strengths on Whatman No. 2 paper.
- Each laboratory will independently obtain its supply of filter paper, recording the batch number. Sufficient paper from the same batch would be purchased to complete Round 3 testing as well.
- Each laboratory will test a different cigarette, recording any identifying batch number.
  - Ideally, these cigarettes would be among those manufactured with a high degree of quality control.
  - The participants will try to select cigarettes with a range of measured ignition strength, keeping in mind that the PFLB on 3 layers will be significantly higher than on 10 layers and that we will avoid PFLB values below 10 and above 90.
  - The cigarettes can be commercial cigarettes or research cigarettes.
  - The identities of any commercial cigarettes will not be shared or retained. To minimize multiple labs accidentally selecting the same cigarette, each laboratory will telephone a central contact person, informing him whether they plan to use a commercial cigarette and, if so, the brand style. Should there be any duplication, the central contact person will request selection of alternate cigarettes.
- Each laboratory will obtain a sufficient number of cigarettes to perform two sets of 40 determinations, one each on 3 layers and 10 layers of filter paper.
  - If both results are not between 10 PFLB and 90 PFLB, then the laboratory will select and test a different cigarette.
  - For its cigarette with test results between 10 PFLB and 90 PFLB, the laboratory will send the results to a central contact person who will forward the results to NIST after removing any laboratory identifiers. NIST will tabulate these results to verify that there is some reasonable distribution of ignition strengths. If not, one or more laboratories will be asked immediately to test a different cigarette type.
- Each laboratory should obtain the equivalent of 10 cartons of cigarettes; these will be sufficient to perform the tests in Round 3 as well. If a laboratory is using a different

cigarette type for each of the two substrates, then the supply should be the equivalent of 5 cartons of each type.

- For each of the two substrates, the test variables will be cigarette carton (if obtained in that manner), and top surface of the paper. (For cigarettes that are already stored in large containers with no obvious way to identify potential production differences among the cigarettes, the only variable is the paper surface.) Each laboratory will perform 20 determinations with one pack of cigarettes and one paper orientation, keeping a record of the carton from which the pack was taken. The testing will continue with packs taken from successive cartons, alternating the paper orientation after each group of 20 determinations. If a determination needs to be repeated (e.g., due to a dropped ash), the cigarette can come from a spare pack and is so denoted. If there are two test operators, one operator would perform the 200 determinations on 3 layers, and the other would perform the 200 determinations.
- NIST will provide a standard Excel template for recording and reporting the test results.
- Each laboratory will be assigned a code number. The Excel data sheets, containing this code number but not the laboratory name, will be sent to the central contact person. He will send the full set of data sheets, with each laboratory identified only by its code number, to NIST for analysis.
- Each laboratory whose standard deviation is significantly different from the expected (binomial) value will be so informed and will be asked to refine its process and re-test.

**<u>Round 3</u>**: Each laboratory will perform 5 sets of 40 determinations on the same cigarette it tested in Round 2, conducting tests on both 3 layers and 10 layers of 3 (of the 4) brands of filter paper, one of which would be Whatman No. 2. Total: 1200 determinations.

- To reduce the number of tests per laboratory, each laboratory would test only 2 of the alternate brands of filter paper. Thus, 6 laboratories would test cigarettes on a particular candidate paper and Whatman No.2 paper. The assigned paper brands will be assigned by NIST.
- Each laboratory is responsible for determining the mass of each batch of filter paper it uses, for compliance with the requirements in ASTM E 2187-04, unless the manufacturer certifies satisfactory values of the mean mass and standard deviation (dry and conditioned).
  - Ideally, the Whatman No. 2 paper will be from the same batch as used in Round 2.
  - The other brands of filter paper will be supplied by the manufacturers.
  - Each laboratory will acquire and retain additional paper of each type used, sufficient to perform 1 set of 5 x 40 determinations on 10 layers and 1 set on 3 layers.
- The cigarettes will be from the same batch(es) as in Round 2 and will be taken from the freezer using the same procedures.
- The data from Round 2 will not be included as part of the results from Round 3.
- NIST will provide a standard template for recording and reporting the test results. The Excel spreadsheet would provide the order of the tests as well, randomizing the order in which different papers are tested in blocks of 20 determinations.

• Each Excel data sheet, containing the laboratory's code letter but not the laboratory name, will be sent to the central contact person. He will send the full set of data sheets, without laboratory identifiers, to NIST for analysis.

#### ANALYSIS OF MULTI-LABORATORY TEST RESULTS

The analysis of the test results would be as follows:

- Six-laboratory comparison of the test result (in PFLB) for each candidate paper relative to the test result for Whatman No. 2 paper. The comparison would be performed for test results on both 3 and 10 layers of filter paper. The precise criterion for an acceptable performance of an alternate paper is to be determined. It will reflect that all six laboratories obtained similar results.
- Six-laboratory comparison of the repeatability for each candidate paper relative to the test result for Whatman No. 2 paper.
  - The comparison would be performed for test results on both 3 and 10 layers of filter paper.
  - Since the "standard" repeatability will have been demonstrated in Round 2, and since the binomial repeatability is known for a given test result, any excessive variation in the Round 3 results would presumably be due to variability in the alternate filter paper.
- Should unexpected results occur in a very few combinations (laboratory/cigarette/paper), a small number of additional tests may be requested to understand the source of the odd results.

#### 4. Results

#### 4.1. Round 1

All nine laboratories submitted satisfactory data for SRM 1082, making unnecessary any visits to the laboratories by the Study Monitor. These results are compiled in Table 1. As noted above, the certified value for SRM 1082 is 12.6 PFLB  $\pm$  3.3 PFLB (mean value and expanded uncertainty).

The results were analyzed using graphics that compared the reported data from each lab with the data predicted for an in-control laboratory that made multiple ignition strength determinations using sets of 40 SRM 1082 cigarettes. These graphics are presented in Figure 2 for the nine participating laboratories. The predicted values of the mean of the determinations are shown on the x-axis and the predicted values of the corresponding standard deviation of the determinations are shown on the y-axis. The predicted points that are most likely to be observed are color-coded blue in each plot, while the points that are less likely to be observed are color-coded red. The total probability associated with the blue points is 95 % and the total probability with the red points is the remaining 5 %. (Each point actually has a different probability of being observed, but the coloring of the points in these plots only divides the data into two classes of points, those that are most likely or least likely to be observed.)

Laboratory	PFLB Values	Mean PFLB	Std. Dev.
Arista	15, 15, 10, 15, 12.5, 12.5, 12.5, 12.5, 17.5, 12.5, 10, 12.5, 15, 12.5, 12.5, 10, 15, 7.5, 10, 15	12.8	2.4
Kidde-Fenwal	5, 12.5, 12.5, 10, 10, 5	9.2	3.4
Labstat	10, 10, 15, 12.5, 15	12.5	2.5
Lorillard	12.5, 20, 20, 12.5, 20, 17.5, 12.5, 10, 10, 17.5	15.3	4.2
NIST	17.5, 15, 17.5, 20, 7.5	15.5	4.8
NRC-C	12, 7.5, 15, 20, 25	16.0	6.8
Philip Morris	5, 5, 7.5, 12.5, 10, 2.5, 5, 15, 12.5, 7.5, 5, 12.5, 25, 5, 7.5, 12.5, 7.5, 10, 20, 22.5, 12.5, 15, 2.5, 15, 10, 7.5, 7.5, 10, 17.5, 5, 15, 20, 15, 17.5, 12.5, 12.5, 12.5, 10, 2.5, 17.5, 10, 12.5, 7.5, 17.5, 10, 10, 12.5	11.2	5.3
R.J. Reynolds	7.5, 27.5, 17.5, 15, 2.5, 12.5, 7.5, 20, 25, 20, 12.5	15.2	7.7
Schweitzer-Mauduit	10, 2.5, 2.5, 7.5, 7.5	6.0	3.3

 Table 1. Round 1 Data – Percent Full-length Burns for Repeat Tests of a Single

 Cigarette on Whatman No. 2 Filter Paper.

Each result actually observed and reported by an ILS laboratory is indicated by the intersection of the cross-hairs drawn over the distribution of predicted data points for SRM 1082. The predicted values are simulated, based on the data and statistical model used for the certification of SRM 1082. Therefore, if the reported results match one of the predicted points from SRM 1082 that is likely to be observed (blue), it suggests the laboratory's results are consistent with the certified value of the SRM. On the other hand, if the mean or standard deviation of the data reported by the laboratory matches a value that is unlikely to be observed (red) or is outside the distribution, it suggests that the laboratory's results may not be consistent with the certified value of SRM 1082.<sup>2</sup> Such cases would generally require further investigation by the laboratory to determine if there are any changes that should be made to its measurement procedures or equipment prior to Round 2 of the ILS.

As shown in Figure 2, for eight of the nine laboratories, the crosshairs intersect in the blue region. One laboratory, Arista, did have data that fell in the extreme portion of the distribution

<sup>&</sup>lt;sup>2</sup> Other features about these results include the crescent patterns visible in the distribution of results and boundary areas where blue and red points in the distribution are intermingled. The crescent patterns are due to the discreteness of the distribution and the fact that the means and standard deviations of the data are not independent of one another for binomial data. The intermingling of the blue and red points along the boundary arises from small random errors in the simulation of the probability distribution for the SRM 1082 data.

of predicted data, indicating potential lack of consistency with the model that was used for the certification of SRM 1082. However, since the Arista mean value was on target and the standard deviation was *low*, no further investigation was deemed necessary.

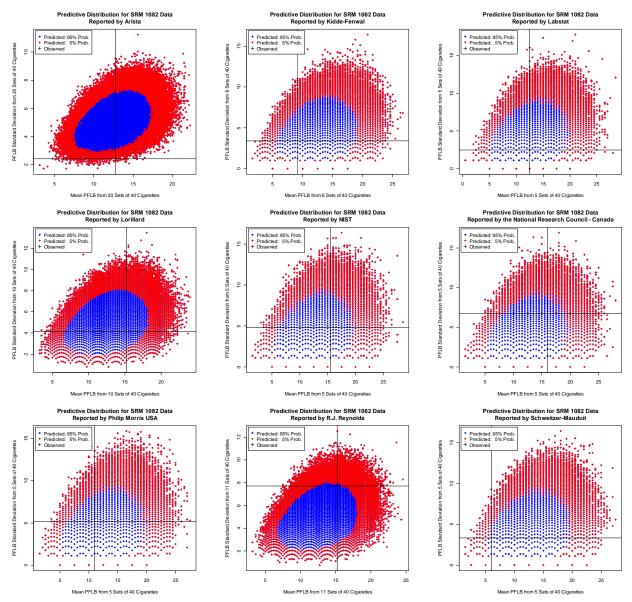


Figure 2. Predicted Distributions of Data and Individual Laboratory Reports of Test Data for SRM 1082 on 10 Layers of Whatman No. 2 Filter Paper.

In addition, it should be noted that it would not be very unusual for this type of result happen by chance, given that multiple laboratories are being simultaneously checked for consistency. For nine laboratories, each in control and checked for consistency at the 95 % probability level, the probability that at least one laboratory's results would fall in the rejection region by chance is bounded between 5 % and 37 %.<sup>3</sup> Given the standard uncertainties that would be observed in each laboratory based on the numbers of sets of 40 determinations made versus the standard uncertainty of the certified value for SRM 1082, the true probability is more likely to be toward

the upper end of this range, indicating that a result of this type would not be unexpected for a group of laboratories that were all in control.

## 4.2 Round 2

Based on preliminary testing (Round 2a), eight of the nine laboratories determined the cigarettes with which they would proceed to Round 2b. Laboratory 9 withdrew from the ILS. Six of the eight remaining laboratories identified a single cigarette that produced no more than 90 PFLB on 3 layers of Whatman No. 2 filter paper and no less than 10 PFLB when tested on 10 layers of Whatman No. 2 filter paper. To be within these test performance ranges, Laboratories 3 and 6 each used two cigarettes, one on each substrate. Laboratories 7 and 8 used the same cigarette.

Using the candidate cigarettes identified in Round 2a, the laboratories then determined the ignition strengths and homogeneity of the cigarettes more thoroughly by running 10 sets of 20 determinations each in Round 2b. Half of the runs were done with rough side of the filter paper facing up, and the other half were done with the smooth side facing up, using a randomized run order to try to eliminate confounding the potential effect of paper surface with any changes in the testing over time. The results of the Round 2b testing are compiled in Table 2.

Lab		10 L	ayers		3 Layers						
#	Rough	Side Up	Smooth	Side Up	Rough	Side Up	Smooth Side Up				
1	15, 10,	$17.0 \pm 7.6$	5, 15, 20,	$16.0\pm6.5$	60, 70, 50,	$64.0\pm11.4$	30, 55, 50,	$56.0\pm18.5$			
	25, 10, 25		20, 20		60, 80		80, 65				
2	20, 10,	$14.0 \pm 4.2$	20, 10, 15,	$12.0 \pm 5.7$	85, 75, 80,	$77 \pm 5.7$	85, 85, 80,	$83 \pm 5.7$			
	15, 10, 15		10, 5		70, 75		90, 75				
3	45, 50,	$45.0\pm3.5$	40, 35, 45,	$38.0\pm5.7$	45, 65, 50,	$50.0 \pm 9.4$	35, 35, 55,	$42.0\pm8.4$			
	45, 45, 40		40, 30		50, 40		45, 40				
4	10, 20,	$17.0 \pm 8.4$	5, 0, 15,	$10.0 \pm 7.1$	65, 80, 60,	$72.0\pm10.4$	75, 60, 55,	$61.0 \pm 9.6$			
	15, 30, 10		15, 15		85, 70		65, 60				
5	15, 30,	$23.0\pm7.6$	25, 25, 35,	$24.0\pm7.4$	70, 95, 90,	$83.0\pm10.4$	100, 85,	$91.0\pm8.9$			
	15, 30, 25		15, 20		85, 75		80, 100, 90				
6	40, 50,	$42.5\pm6.5$	25, 15, 25,	$24.2\pm9.2$	50, 30, 25,	$31.3\pm13.1$	10, 25, 15,	$20.0\pm 6.3$			
	45, 35		25, 15,40		20		20, 25, 25				
7	40, 35,	$25.0\pm14.1$	10, 10, 20,	$17.0 \pm 8.4$	75, 80, 80,	$77.0\pm7.6$	85, 95, 85,	$89.0\pm4.2$			
	10, 30, 10		30, 15		85, 65		90, 90				
8	10, 10,	$13.0\pm4.5$	20, 20, 40,	$22.0\pm10.4$	75, 100,	$82.0\pm7.4$	70, 85, 75,	$79.0\pm6.5$			
	10, 15, 20		15, 15		80, 80, 75		85, 80				

 Table 2. Percent Full-length Burns for Repeat Tests on Whatman No. 2 Filter

 Paper in Round 2.

The PFLB values for all combinations of cigarette and substrate were deemed suitable, i.e., they were between 10 PFLB and 90 PFLB. The repeatability values included the effects of cigarette uniformity, filter paper uniformity, and test operator performance. The data indicated that each combination of these could result in test repeatability that was deemed suitable. This was determined by fitting a binomial model to the results from each laboratory for each paper surface, as described in more detail below, and assessing residuals for consistency with the fitted model.

The data in Table 2 verified that, for the Whatman No. 2 paper, there might be a moderate effect of testing with the rough vs. smooth side of the Whatman paper facing up. The effect was not the same for all the cigarette designs. Therefore, the ILS intent was to weigh both sides equally in Round 3 and more carefully assess the results for the surface effect, as described below.

### 4.3 Round 3

To address the potential sensitivity of the test results to the surface texture of the filter paper in Round 3, half of the Round 3 tests were conducted with each side of the filter paper facing up.

The ILS participants were asked to feel the two sides of the paper from the different manufacturers and to indicate whether they could discern a rough side and a smooth side. The results indicated that the degree of difference in surface roughness varied among the four filter paper brands, and that different people had differing tactile abilities to discern any difference.

Therefore, the following protocol was added to the test procedure:

- Open a container of filter paper with the sheets horizontal.
- Note that the spreadsheet column titled "Filter Paper Surface Facing Up" contains either a "1" or a "2."
- For a determination where the column has a "1," the paper orientation in the determination should be the same as it was in the box.
- For a determination where the column has a "2," the paper orientation in the determination should be turned over, i.e., the up side in the box is the down side in the determination.
- Open each successive container of filter paper in the same manner.

The data from the eight laboratories are compiled in Table 3. In general, the data were wellbehaved. The following highlights some specific anomalies:

- There were a few inconsistencies between the Round 3 Whatman No. 2 data and the data from Round 2b (which involved the same type of cigarette and the same supply of filter paper). These cells in Table 3 are indicated with a pound sign, #.
- There were a few cells in Table 3 where the standard deviation was substantially larger than in the preponderance of cells. These values are marked with an asterisk, \*.

Upon first inspection, it appeared that the data from laboratories 1, 2, 4, 5 were generally selfconsistent, despite occasional large standard deviations (statistical noise). The data from each of the other 4 laboratories have occasional anomalies that do not constitute grounds for proceeding without the full complement of eight data sets. Laboratories 7 and 8, which used the same test cigarette, are in general agreement with each other.

Laboratory 6 deviated slightly from the experiment design in round 3, performing unequal numbers of tests with each side of the filter paper facing up for each type of paper. The distribution of tests with each surface facing up is shown in Table 3. Fortunately, this deviation had a fairly minor effect on the data analysis, increasing the uncertainties of the comparisons between results with different paper surfaces facing up and between different paper brands. The validity of the statistical analysis was not affected, however.

	Whatman No. 2							Ahlstrom								
İ		10 La	ayers		í	3 Layers	5		10 Layers				3 La	yers	yers	
Lab #	Side	1 Up	Side	2 Up	Side	1 Up	Side	2 Up	Side 1 Up Side 2 Up			Side	1 Up	Side 2 Up		
	20		20		65		70		not measured							
	15	19.0	25	15.0	55	60.0	55	59.0								
1	20	±	10	±	60	±	65	±								
	20 20	2.2	10 10	7.1	60 60	3.5	60 45	9.6								
	20		25		85		43 80		15		20		95		90	
	25	15.0	15	18.0	65	81.0	75	77.0	25	25.0	20	22.0	95	93.0	95	96.0
2	0	±	15	±	85	±	60	±	20	±	25	±	95	±	100	±
	15	10.6	20	4.5	90	9.6	85	10.4	25	9.4	20	2.7	85	4.5	100	4.2
	10		15		80		85		40		25		95		95	
	20		50		40		50		30		20		65		75	
	0	7.0	10	32.0	55	49.0	50	49.0	20	19.0	20	13.0	35	62.0	55	68.0
3	0	±	30	±	60	±	70	±	15	±	5	±	70	±	75	±
	5 10	8.4 #	30 40	14.8	55 35	10.8	50 25	16.0 *	25 5	9.6	5 15	7.6	60 80	16.8 *	65 70	8.4
	20	#	40 5		60		23 55	*	5		15		80	*	70	
	0	16.0	10	10.0	55	59.0	75	57.0	not measured							
4	15	±	10	±	85	±	65	±								
	10	12.9	10	3.5	50	15.6	40	13.5								
	35		15		45	*	50									
	35		15		75		95									
	0	23.0	5	22.0	100	91.0	95	92.0								
5	25	±	30	±	95	±	90	±	not measured							
	25	13.5	25	12.0	85	10.8	100	7.6								
	30 45		35 35		100 65		80 50		70		65		60	1	65	
	43 50	47.0	55 50	46.0	50	57.5	50 65	61.0	45	62.5	35	44.0	65	59.0	60	58.3
	50	±	55	±	50	±	55	±	70	±	60	±	50	±	50	±
	40	4.5	45	7.4		10.6	70	8.2	65	11.9	45	20.1	65	6.5		7.6
6	50		45	#			65	#			15	*	55			
							70				35		50			
							55						35			
							55									
	5		15		75		80		10		40		95		95	
	10	8.0	25	10.0	95	75.0	90	79.0	10	23.0	30	21.0	80	88.0	90	95.0
7	10	±	0	±	80	±	70	±	10	±	5	±	90	±	95	±
	10	2.7	5	10.0	60	13.7	80	7.4	55	19.9	5	15.6	85	5.7	95	3.5
	5		5		65		75	#	30	*	25	*	90		100	
	15		5		75		65		10		15		95		75	
	20	14.0	0	2.0	75	77.0	50	61.0	15	11.0	20	15.0	80	79.0	70	75.0
8	5	±	5	±	70	±	70	±	5	±	10	±	60 75	±	70	±
	10 20	6.5	0	2.7	85 80	5.7	65 55	8.2	10	4.2	20	5.0	75 85	12.9	90 70	8.7
	20		0	#	80		55		15		10		85		70	

 Table 3. Percent Full-length Burns for Repeat Tests in Round 3.

	Tela-Kimberly (Swiss Quality)									Tervakoski								
	10 Layers			10 Layers 3 Layers			10 Layers 3 Layers											
Lab #	Side	1 Up	Side	2 Up	Side	1 Up	Side	2 Up	Side	Side 1 Up Side 2 Up		Side 1 Up		Side	2 Up			
	20		10		45		55		15		15		50		60			
	10	14.0	10	15.0	65	59.0	55	51.0	20	19.0	15	16.0	60	56.0	60	65.0		
1	20	±	20	±	65	±	50	±	25	±	20	±	60	±	80	±		
	10	5.5	20	5.0	60	8.2	50	4.2	20	4.2	10	4.2	55	4.2	75	12.2		
	10		15		60		45		15 25		20		55		50			
											25		80		70			
									20	19.0	25	22.0	90	81.0	100	86.0		
2				not me	asured				10	±	25	±	75	±	65	±		
									20	5.5	15	4.5	80	5.5	100	17.1 *		
	25		55		25		40		20		20		80		95	*		
	23 5	19.0	25	34.0	23 40	35.0	40 35	34.0										
3	30	19.0 ±	23 50	54.0 ±	40 30	55.0 ±	30	54.0 ±	not measured									
3	0	15.6	10	18.5	35	- 7.9	35	4.2				not me	asureu					
	35	*	30	*	45	1.5	30	2										
	15		25		60		65		0		10		55		60			
	5	11.0	15	14.0	80	67.0	50	63.0	20	6.0	20	14.0	45	51.0	80	65.0		
4	5	±	20	±	55	±	80	±	5	±	15	±	55	±	65	±		
	5	8.9	10	9.6	60	12.0	55	11.5	5	8.2	15	4.2	45	5.5	65	9.4		
	25		0		80		65		0		10		55		55			
	10		35		75		80		15		25		90		95			
	35	27.0	15	28.0	90	74.0	80	81.0	25	28.0	35	31.0	95	91.0	95	90.0		
5	50	±	20	±	70	±	70	±	25	±	40	±	85	±	90	±		
	20	15.7	30	10.4	65	9.6	75	11.4	40	9.7	15	10.8	90	4.2	95	8.7		
	20	*	40		70		100		35		40		95		75			
	40		50		40		25											
	40	39.0	35	35.0	45	52.0	40	45.0										
6	40	±	50	±	40	±	55	±				not me	easured					
-	25 50	8.9	5	21.2 *	60 75	15.2 *	60	15.8										
	50			*	75	*												
	35				65													
	5	0.0	30	10.0	45	-1.0	95 00	00.0										
_	15	9.0	15	18.0	75 70	71.0	90 80	88.0					-					
7	10	±	10	±	70 75	±	80 85	± 5.7				not me	easured					
	5 10	4.2	10 25	9.1	75 90	16.4 *	85 90	5.7										
	10		23		90	•	90		5		10		85		70			
									0	3.0	10	13.0	85 75	75.0	85	74.0		
Q				not me	asurad				10	5.0 ±	10	±	60	±	75	+.0 ±		
0	8 not measured						0	4.5	20	4.5	75	9.4	70	6.5				
							0		10		80		70					
								-		-		-						

## Table 3. Percent Full-length Burns for Repeat Tests in Round 3 (cont.).

To assess the effects of the paper surface facing up and the candidate papers relative to the Whatman paper, a binomial model was fit to the data using Bayesian statistical methods. The model allowed for different ignition strengths for each laboratory's cigarette on each of three tested paper types and for each of two paper surface facing up. A binomial model was chosen because in past analyses of ignition strength data it has provided a good description of the data obtained in a particular laboratory for a given type of cigarette. Note that in the one case from Round 1 in which the results that were seen had significantly lower variation than would be consistent with a binomial model, the use of the binomial model will tend to overestimate the uncertainties and make it less likely that significant differences will be found between the different paper brands or paper surfaces. Based on the design of the study, paper brands and paper surfaces will only be compared within different combinations of laboratory and cigarette, minimizing the effects of any consistent systematic errors that might impact a particular laboratory's results on the comparison.

The model, given in equation (1) below, places a uniform prior distribution on the true probability, p, of a full-length burn for a given cigarette under a given set of conditions. This prior probability distribution assumes that any value for the ignition strength between 0 and 1 is equally likely, essentially adding no prior information about the ignition strength for any combination of lab/cigarette, paper, or paper surface before observing the data.

$$p_{ijk} \sim Uniform(0,1), \ i = 1,...,8; \ j = 1,2,3; \ k = 1,2$$

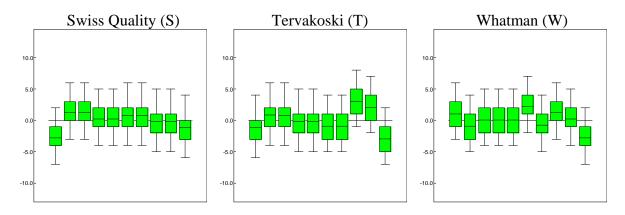
$$I_{ijkl} \sim Bin(20, p_{ijk}), \ l = 1,...,5$$
(1)

*I* is the observed number of full-length burns from a set of 20 sequential tests under the same conditions, *i* indexes the eight laboratories, *j* indexes the three papers each lab measured, *k* indexes the surfaces of paper facing up during each test, and *l* indexes the individual sets of 20 tests.

The same model was separately fit to the data for tests on 3 layers and 10 layers of filter paper. The models were fit using the software package WinBUGS<sup>4</sup> which uses Markov Chain Monte Carlo simulation to obtain posterior probability distributions for each parameter in the model. For this model the parameters of interest were the individual ignition strengths for each combination of cigarette, paper, and surface, differences between those ignition strengths for each surface for each combination of cigarette and paper, and differences between the average ignition strengths for each cigarette and paper versus the corresponding ignition strength on the Whatman paper. The implementation of the model from equation (1) in WinBUGS format and the data used in the WinBUGS analysis are given in Appendix A.

To assess the fit of the model to the data, residuals computed as the difference of the observed number of full-length burns minus the number of full-length burns predicted by the model were compared. Because the analysis was carried out via simulation, a distribution of data predicted by the model was compared to each observation and these distributions were displayed as box plots. A typical set of residual plots for tests made on 3 layers of filter paper in Laboratory 1 is shown in Figure 3. In each plot, the first five boxes are for tests with surface 1 facing upward, and the second five boxes are for tests with surface 2 facing upward.

Since (a) the boxes are centered around the reference line in a random way and (b) none of the distributions is much further away from the reference line than is accounted for by the random variation in the values of full-length burns predicted under the model, the model appears to be consistent with the data. Results for the remaining 7 laboratories are similar. This leads to the conclusion that the data nominally follow a binomial model. This further leads us to conclude that the data are consistent with the requirements for test repeatability in the E2187 standard, which sets the limits for repeatability as a function of ignition strength at limits derived from a binomial model. Similar methods were used to assess the repeatability of the results from Round 2b of the study as noted above and are displayed in Appendix B.



#### Figure 3. Residual plots for Laboratory 1 on 3 layers of filter paper, Round 3 data.

After validation of the binomial model as a reasonable description of the measurement processes in each laboratory, results from the fit of this model were used to compare differences between tests made with the two paper surfaces facing up and among the different brands of papers. Differences in results for tests done with different paper surfaces facing up are shown in Figure 4 and Figure 5. In each of the plots, the uncertainty bars shown are approximate, simultaneous 95 % expanded uncertainties for each type of paper. Red bars indicate differences that are significantly different from zero, and blue bars overlap zero by 2 PFLB or less. The values on the x-axis are the laboratory number followed by the paper designation: Ahlstrom (A), Swiss Quality (S), Tervakoski (T), and Whatman (W).

The results shown in Figure 4 and Figure 5 indicate that there are statistically significant differences, for some cigarette designs for each type of paper, depending on whether the rough side or the smooth side is facing upward.<sup>3</sup>

<sup>&</sup>lt;sup>3</sup> These differences are not simply due to the relatively large number of comparisons being made, leading to the interpretation of random, rare events as significant differences. The uncertainty intervals shown here account for the number of tests done on each combination of paper type and number of layers of paper using the Bonferroni inequality<sup>3</sup>. As a result, each set of intervals for a given combination of paper type and number of layers of paper has a probability of at least 95 % of simultaneously encompassing the true ignition strength differences for all of the cigarette designs tested on that paper.

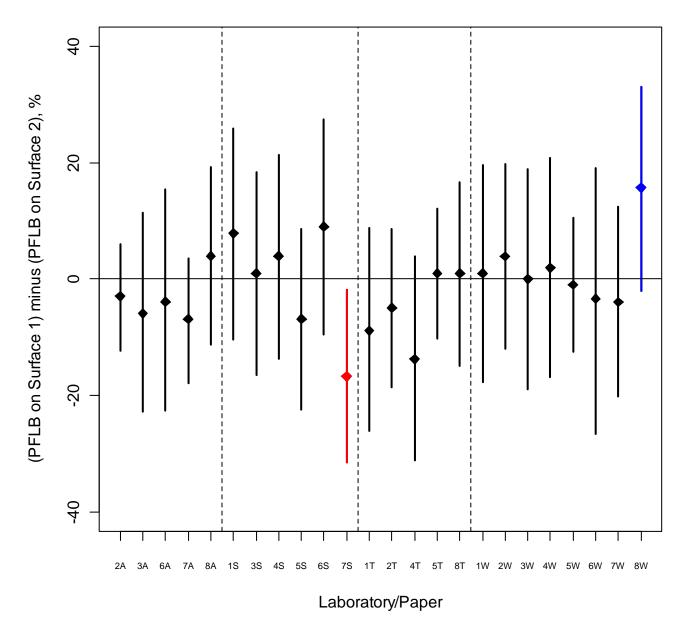


Figure 4. Differences in the Percentage of Full-length Burns with Different Paper Surfaces Facing up for Tests on 3 Layers of Filter Paper.

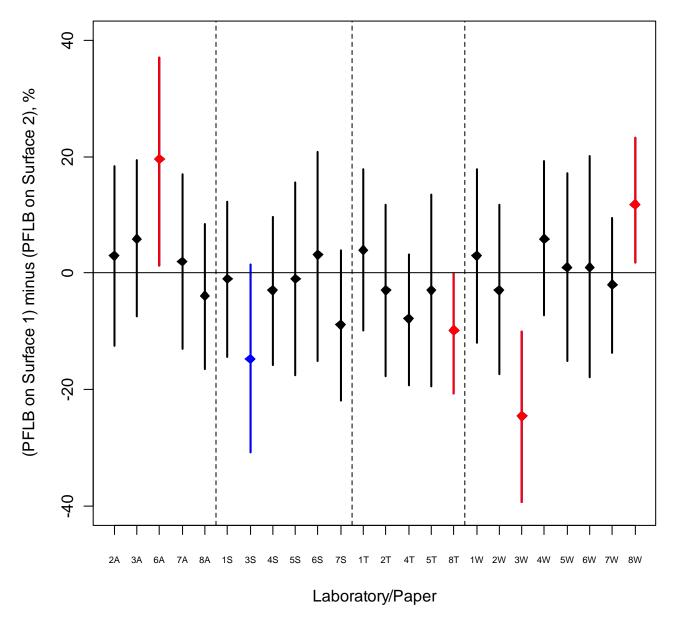


Figure 5. Differences in the Percentage of Full-length Burns with Different Paper Surfaces Facing up for Tests on 10 Layers of Filter Paper.

Figure 6 and Figure 7 show differences between ignition strengths assessed on each brand of paper versus those assessed using the Whatman paper. As above, the uncertainty bars shown are approximate, simultaneous 95 % expanded uncertainties for each difference in ignition strength computed using the Bonferroni inequality to account for multiple comparisons. Red bars indicate differences that are significantly different from zero. The values on the x-axis are the laboratory number followed by the paper designation: Ahlstrom (A), Swiss Quality (S), Tervakoski (T), and Whatman (W).

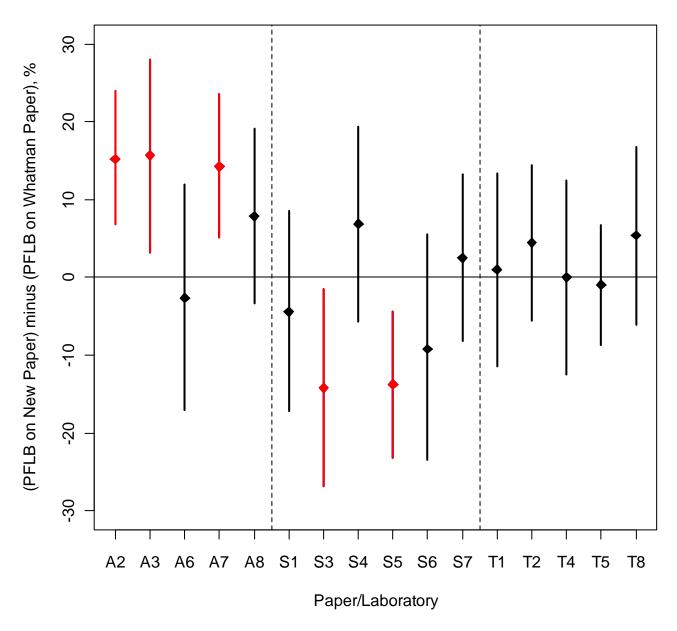
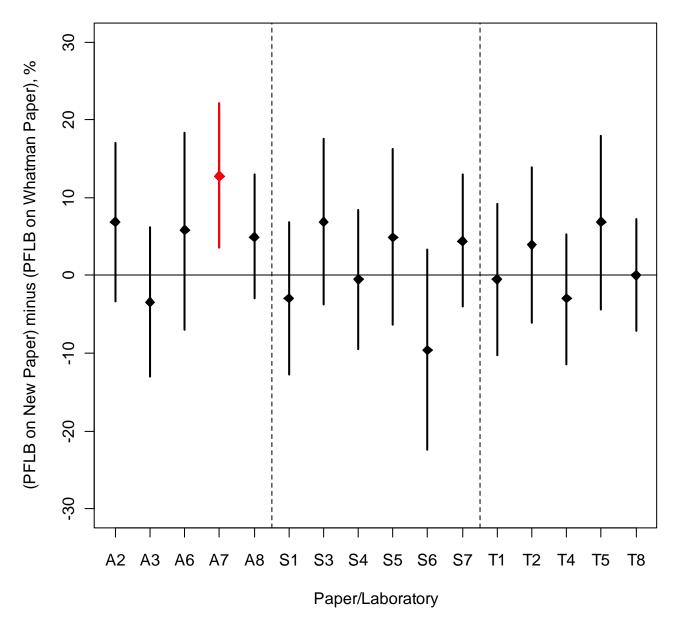


Figure 6. Differences in the Percentage of Full-length Burns between Each Brand of Alternate Paper and Whatman Paper for Tests on 3 Layers of Filter Paper.



## Figure 7. Differences in the Percentage of Full-length Burns between Each Brand of Alternate Paper and Whatman Paper for Tests on 10 Layers of Filter Paper.

Similar to the assessment of the paper surface effect, Figure 6 and Figure 7 indicate that, for more than one cigarette design, there are statistically significant differences between the results of tests conducted using the Ahlstrom and Swiss Quality papers when compared to the corresponding results obtained using Whatman paper. This finding suggests that cigarette tests performed using these batches of the Ahlstrom or Swiss Quality papers would not uniformly produce results consistent with tests performed using Whatman No. 2 paper. In contrast, there is no evidence based on these tests that results obtained using this batch of Tervakoski paper would significantly differ from those made using Whatman paper.

It is noted that these test results are for a single batch of each of the alternate filter paper types. It is possible that significant differences might be seen for future batches of Tervakoski paper. It is also possible that future batches of Ahlstrom or Swiss Quality paper might give results consistent with those obtained using the Whatman paper. However, the fact that there are significant differences (indicated by the red bars) both above and below the zero line for these two types of paper in Figure 6 suggests that simply adjusting the thermal inertia of the papers would not achieve comparability of test results with the Whatman paper.

Decades of testing have indicated a degree of consistency of test results using the Whatman No. 2 paper.<sup>5</sup> For this paper, as manufactured, specification of the paper mass and moisture uptake has led to this consistency. Extensive additional work would be needed to identify which properties control the consistency of each of the other three paper types and what values of these properties are needed to achieve the same test results as the Whatman No. 2 paper. This would then need to be followed by determination of the repeatability of test results for multiple batches of the alternate paper type.

## 5. Conclusions

This interlaboratory study generated valuable information regarding the degree of sensitivity of the test results from ASTM E 2187 to the nature of the test substrate. This information was not substantively diminished by the occasional minor irregularities in the laboratory data. This led to the following conclusions.

1. The standard test method should be modified so that future tests will be performed with the upward facing paper surface prescribed. It might seem logical to perform half of each standard set of 40 determinations with the rough side facing up and half with the smooth side facing up. However, as is manifested in the data set from Laboratory 6, this could result in making the test procedure more difficult to implement. Requiring that all testing be performed with a specified side facing up is more practical.

2. Each of the three alternate filter paper types demonstrated test result repeatability similar to that obtained using Whatman No. 2 paper.

3. Based on the tests of these individual batches of each type of paper, there is evidence to suggest that cigarette tests performed using Ahlstrom or Swiss Quality papers would not uniformly produce results consistent with tests performed using Whatman No. 2 paper. In contrast, these data do not provide evidence that results obtained using this batch of Tervakoski paper would significantly differ from those made using Whatman paper.

4. Significant additional testing would still be needed to have high confidence that testing a variety of cigarettes using any of the three alternate filter paper types would consistently yield test results indistinguishable from the data obtained using Whatman No. 2 paper.

#### 6. Acknowledgements

The authors express their appreciation to Ms. Linda Crumpler, then with RJR Tobacco Company, and Dr. Joe Wanna of Schweitzer-Mauduit International for conducting the preliminary research that preceded this interlaboratory study. The ILS success was the result of active participation of the staff of the nine laboratories listed in Section 3 of this report. Dr. Joseph Senecal of Kidde-Fenwal served as the central contact for transferring the (unattributed) test data from the performing laboratories to NIST.

### 7. Reference

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5. Gann, R.G. and Guthrie, W.F., "Robustness of Measuring the Ignition Strength of Cigarettes with ASTM Method E2187-02b," NIST Technical Note 1454, National Institute of Standards and Technology, Gaithersburg, MD, 2003.

## **Appendix A – Model and Data in WinBUGS Format**

The model used to estimate the ignition strength values for the data from Round 3 is given below. In addition to the quantities defined in equation (1), the quantity sI is the ignition strength predicted using the model; the quantity res is the difference (or residual error) between the observed number of full-length burns and the number predicted using the model; the quantity dp.s is the difference in the ignition strengths between the two paper surfaces for a given combination of lab/cigarette and paper; the quantity dp.p is the difference between the average ignition strength on one of the potential new paper brands versus the Whatman paper.

```
model
{
for(i in 1:48) {p[i]~dunif(0,1)}
for(i in 1:240)
ł
I[i]~dbin(p[set[i]],20)
sI[i]~dbin(p[set[i]],20)
res[i] <- I[i]-sI[i]</pre>
}
for(i in 1:24)
{
dp.s[i] <- p[2*i-1]-p[2*i]
}
for(i in 1:16)
dp.p[i] <- (sum(p[j[i]:(j[i]+1)])-sum(p[k[i]:(k[i]+1)]))/2
}
}
```

The ignition strength data for tests on 3 layers of filter paper is listed below. The data is sorted by laboratory, paper within each lab (alphabetically), and paper surface. The values of the variable set indicate which group of data a particular observation belongs to. For example, the first five values of I, which each have a values of set=1, are from laboratory 1, using Swiss Quality paper, on surface number 1. All of the values with set=2 are from laboratory 1, using Swiss Quality paper, on surface number 2. Set numbers 3 and 4 correspond to laboratory 1 using Tervakoski paper, while set numbers 5 and 6 correspond to laboratory 1 using Whatman paper.

#### list(

set=c( 1, 1, 1, 1, 1, 2, 2, 2, 2, 2, 3, 3, 3, 3, 3,
4, 4, 4, 4, 4, 5, 5, 5, 5, 5, 6, 6, 6, 6, 6,
7, 7, 7, 7, 7, 8, 8, 8, 8, 8, 9, 9, 9, 9, 9,
10,10,10,10,10,11,11,11,11,11,12,12,12,12,12,
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16,16,16,16,16,17,17,17,17,17,18,18,18,18,18,
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22, 22, 22, 22, 22, 23, 23, 23, 23, 23,
25, 25, 25, 25, 25, 26, 26, 26, 26, 26, 27, 27, 27, 27, 27,
28,28,28,28,28,29,29,29,29,29,30,30,30,30,
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46,46,46,46,46,46,47,47,47,47,47,48,48,48,48,48),
I=c( 9,13,13,12,12,11,11,10,10, 9,10,12,12,11,11,
12,12,16,15,10,13,11,12,12,12,14,11,13,12, 9,
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14,17,15,14,14,15,15,14,17,16,13,10,14,13,11),
j=c(7,13,31,37,43, 1,15,19,25,33,39,3, 9,21,27,45),
k=c(11,17,35,41,47, 5,17,23,29,35,41, 5,11,23,29,47)
)

The ignition strength data for tests on 10 layers of filter paper is listed below. The layout of the data is the same as described on the previous page for the tests on 3 layers of paper.

list(

list(	
set=c( 1, 1, 1, 1, 1, 2, 2, 2, 2, 2, 3, 3, 3, 3, 3,	
4, 4, 4, 4, 4, 5, 5, 5, 5, 5, 6, 6, 6, 6, 6,	
7, 7, 7, 7, 7, 8, 8, 8, 8, 8, 9, 9, 9, 9, 9,	
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6, 4, 3, 5, 1, 4, 4, 1, 1, 3, 5, 1, 6, 0, 7,	
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3, 1, 1, 1, 5, 5, 3, 4, 2, 0, 0, 4, 1, 1, 0,	
2, 4, 3, 3, 2, 4, 0, 3, 2, 7, 1, 2, 2, 2, 3,	
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5, 7, 8, 3, 8, 7, 0, 5, 5, 6, 3, 1, 6, 5, 7,	
14, 9,14,13,13, 7,12, 9, 3, 7, 8, 8, 8, 5,10,	
7,10, 7,10, 1, 9,10,10, 8,10, 7,10,11, 9, 9,	
2, 2, 2,11, 6, 8, 6, 1, 1, 5, 1, 3, 2, 1, 2,	
6, 3, 2, 2, 5, 1, 2, 2, 2, 1, 3, 5, 0, 1, 1,	
2, 3, 2, 4, 2, 3, 4, 1, 2, 4, 1, 0, 1, 0, 0),	
j=c(7,13,31,37,43, 1,15,19,25,33,39, 3, 9,21,27,45)	'
k=c(11,17,35,41,47, 5,17,23,29,35,41, 5,11,23,29,47)	
)	

## **Appendix B – Residual Plots**

The plots on the next four pages show the residuals from the models fit to the Round 3 data. The box and whisker structures in each plot show the distribution of residuals with respect to the uncertainty in the fit of the model for each set of 20 observed ignition strength tests that were run during one block of time. In each plot, the first 5 box and whisker structures are for data with paper Side #1 facing up and next five are for data with paper Side #2 facing up.

The residual plots all appear to be consistent with the hypothesized binomial model since the boxes appear to be randomly scattered above and below the zero line and most of the residual distributions overlap the zero line. Changes between ignition strengths with paper side also can be seen in some of these plots and are indicated by a difference in the average length of the first 5 boxes relative to the average length of the second 5 boxes. This change in the variability of the residuals is due to the properties of the binomial distribution and is a reflection of the fact that more data is needed to estimate a proportion near 0.5 to a given relative precision than is needed to estimate proportions nearer to 0 or 1 to the same level of precision.

