C. Test Procedures

The cigarettes were tested nominally using the two procedures described above. However, it was expected that many of these packings would be of very high ignition propensity. Therefore, the full complement of replicates (48 or 16) was performed first for the duck #4 and 15-layer substrates. If 48 ignitions in the Mock-Up Ignition Method were observed, then 8 replicates were performed on the remaining substrates. (This is more than the minimum of six called for in Section II.A., since it is based on an earlier estimate of the reproducibility of the test methods.) Also, for certain of the top fourteen packings (4 through 9 in Table 32), additional tests were run on Duck #6 before the decision was made to limit the testing to eight replicates. If 16 full-length burns in the Extinction Method were noted, then no further testing was performed.

One of the packings in the group of six selected as less ignition prone (packing C in Table 32) had a tendency to self-extinguish during the vertical free-burn period prior to placement on the substrate. Thus, on duck #6, 13 of the 35 extinguishments occurred during this vertical free-burn. When an additional 24 replicates were run with this duck using a horizontal cigarette orientation during the free-burn interval, 12 caused ignition and 12 self-extinguished on the mock-up. This increase in ignition rate from 27% to 50% is comparable to the repeatability of the method (Table 24) and was not considered sufficient to revise the test procedure. Note, however, that this cigarette was run only with a horizontal free-burn for the tests on duck #10 and the three filter paper substrates.

D. Analysis of Data

Table 32 shows that the top 14 best-selling packings behaved in a virtually identical manner, with packing #7 exhibiting 2 self-extinctions on the duck #4. Both test methods indicate they are strong igniters; neither method reveals any differentiation among these packings. Reference to the interlaboratory study results for the Mock-Up Ignition Method (Figure 4) indicates that all of these cigarettes are stronger igniters than the 2 strongest experimental cigarettes (503 and 501) used in that study.

The 6 packings chosen as likely to be of lesser ignition propensity did in fact show this tendency to varying degrees. Both methods reveal the same qualitative picture: a monotonic increase in the number of ignitions or full-length burns as one moves toward the lighter fabrics or fewer filter layers. Of particular note is that 4 of these packings (A, C, E, F) showed few or no ignitions in 48 replicates on the #4 duck. Compared to the Mock-Up Ignition Method, the Extinction Method does not seem to pick up the reduced ignition propensity of one of these (packing E) and also does not distinguish as strongly the performance of cigarettes A and D from the 14 best-selling packings. Packing C shows a persisting tendency toward a lesser ignition propensity, even on Duck #10; the Extinction Method does not show this on the 10- or 3-layer substrates. These observations are consistent with those in the interlaboratory study, which indicated that the Mock-Up Method is capable of better distinction among cigarettes in the upper/middle part of the ignition propensity range.

Table 32. Results of Commercial Cigarette Testing

	Mock-Up Ignition Test Method			Cigarette Ex	tinction Test	Method
Cigarette	Duck #49	Duck #6	Duck #10	15 Layers ¹⁰	10 Layers	3 Layers
1	48/0/0	8/0/0	8/0/0	16/0		
2	48/0/0	8/0/0	8/0/0	16/0		
3	48/0/0	8/0/0	8/0/0	16/0		1
4	48/0/0	12/0/0	8/0/0	16/0		
5	48/0/0	12/0/0	8/0/0	16/0		
6	48/0/0	12/0/0	8/0/0	16/0		
7	46/0/2	16/0/0	8/0/0	16/0		
8	48/0/0	16/0/0	8/0/0	16/0		
9	48/0/0	16/0/0	8/0/0	16/0		
10	48/0/0	8/0/0	8/0/0	16/0		<u> </u>
11	48/0/0	8/0/0	8/0/0	16/0		
12	48/0/0	8/0/0	8/0/0	16/0		
13	48/0/0	8/0/0	8/0/0	16/0		
14	48/0/0	8/0/0	8/0/0	16/0		
A	2/5/41	44/0/4	8/0/0	6/10	15/1	6/0
В	35/1/12	44/0/4	8/0/0	15/1	6/0	6/0
С	0/0/48	13/0/35	4/0/4	2/14	8/8	16/0
D	22/1/25	35/0/13	8/0/0	14/2	15/1	6/0
E	0/31/17	46/0/2	8/0/0	15/1	16/0	6/0
F	0/6/42	38/0/10	8/0/0	3/13	8/8	16/0

Table 33 provides a further check of consistency between the two methods, and thus further affirmation that the measured cigarette performance is consistent across diverse substrates. Here, the ignition strengths of the five cigarettes from the interlaboratory study and five of the second group of commercial cigarettes are tabulated. [The fourteen best-selling commercial packings showed nominally 100% ignitions on all six substrates and thus the data are not informative.] Cigarette C is omitted because the testing was performed using two different pre-burn procedures. The rows are in order of decreasing average of the six values in the row; the columns are arranged similarly. The averages from the interlaboratory study are for 432 replicates (9 labs \times 48 each) for the cotton duck substrates and 144 replicates (9 \times 16) for the filter paper substrates. The number of replicates for the commercial cigarettes are far fewer and shown in Table 32.

Results for the Mock-Up Ignition Method are shown in the sequence: Ignitions/Non-Ignitions/Self-Extinctions.

¹⁰ Results for the Cigarette Extinction Method are shown in the order: Full Burn/Partial Burn.

Table 33. Percent Ignitions or Full Length Burns on Test Method Substrates

SUBSTRATE → CIGARETTE ↓	3 Layers	Duck #10	10 Layers	Duck #6	15 Layers	Duck #4
В	100	100	100	92	94	73
503	100	100	100	100	100	53
501	100	100	100	100	100	11
D	100	100	94	73	88	46
E	100	100	100	96	94	0
531	99	98	94	95	88	0
A	100	100	94	92	38	4
F	100	100	100	79	19	0
529	57	30	6	8	2	0
530	6	3	0	0	0	0

There is a generally consistent decrease in ignition strength from the top left corner of the matrix to the lower right corner, especially considering the reproducibility of the data established in the interlaboratory study. Perhaps the largest single departure from the general pattern in the Table is for either cigarette 501 or cigarette D tested on the duck #4 substrate. However, these cigarette ignition propensities are quite comparable to each other. The two duck #4 values are within the established interlaboratory reproducibility of each other, and both cigarettes yield similar results on all the other substrates.

As in the TSG studies [3], it is of interest to determine whether reduced ignition propensity necessarily results in increased yields of undesirable smoke components. The mean values and standard deviations for the two sets of commercial cigarettes tested here are shown in Table 34. The entries in Table 34 were compiled from data contained in reference [34]. These data were generated by the Tobacco Institute Testing Laboratory. The results show that reduced ignition propensity has been achieved with no significant increase in these three smoke components.

Table 34. Averaged Smoke Component Yields from Commercial Cigarettes (mg per cigarette)

Cigarettes	Tar (mg)	Nicotine (mg)	Carbon Monoxide (mg)
1-14	14.4 ± 4.2	1.04 ± 0.27	13.7 ± 2.2
A-F	11.7 ± 4.8	0.98 ± 0.38	12.5 ± 6.2

V. CONCLUSIONS AND RECOMMENDATIONS

The research funded under the Cigarette Safety Act of 1984 (P.L.98-567) and the Fire Safe Cigarette Act of 1990 (P.L. 101-352) has led to the development of two test methods for measuring the ignition propensity of cigarettes.

- The Mock-Up Ignition Test Method uses substrates physically similar to upholstered furniture and mattresses: a layer of fabric over padding. The measure of cigarette performance is ignition or non-ignition of the substrate.
- The Cigarette Extinction Test Method replaces the fabric/padding assembly with multiple layers of common filter paper. The measure of performance is full-length burning or self-extinguishment of the cigarette.

The 14 best-selling commercial cigarette packings and six other commercial packings were examined using the two methods. Both methods showed reduced ignition propensities for five of the six specialty cigarettes relative to the best sellers.

For a product standard at present, there is a preference for using the Mock-Up Ignition Test Method because it is capable of better discrimination among cigarettes of high/moderate ignition propensity. However, routine measurement of the relative ignition propensity of cigarettes is feasible using either of the two methods.

Improved cigarette performance under both methods has been linked with reduced ignition behavior in full-scale chairs constructed using fabrics that differ substantially from the materials in the test methods. It is reasonable to assume that this implies an analogous benefit of reduced ignitability is to be found in the real world population of upholstered furniture. However, the *precise* incremental life and property savings that would accrue from the use of the test methods described here in conjunction with a particular test criterion has not been established.

Both methods have been subjected to interlaboratory study. The resulting reproducibilities were comparable to each other and comparable or superior to most currently-used standard fire tests.

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APPENDIX A

U.S. DEPARTMENT OF COMMERCE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY Gaithersburg, MD 20899

Report of Test FR 3984

May 1991

"Reevaluation of Experimental Cigarettes used in the Cigarette Safety Act of 1984"

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Introduction

Under P.L. 101-352, the Fire-Safe Cigarette Act of 1990, one of the tasks assigned to NIST was to develop a valid test method for the ignition propensity of cigarettes. In a previous study [A-1], under the Cigarette Safety Act of 1984, 32 experimental cigarettes that vary systematically in five design parameters were extensively studied to determine their ignition propensity on soft furnishings. The evaluation was done with bench-scale mock-ups and on full-scale furniture. Analysis of the test results indicated an agreement between the bench-scale and full-scale tests. Since these experimental cigarettes are well characterized, it is desirable to use these cigarettes in the new study. However, these cigarettes have been stored in freezers since the completion of the first study. To determine if the cigarettes have changed during storage, a reevaluation was undertaken using fabric and padding materials retained from the first study.

Statistical Selection of Cigarettes

The original characterization of these cigarettes involved testing on four substrates, which in turn were composed of combinations of 3 fabrics and two paddings. Due to the limited availability of one of the fabrics (California standard), it was impossible to repeat the entire experimental series using all 32 types of cigarettes on all 4 substrates. There was only enough material available to perform ignition tests for 8 of the 32 cigarettes on all 4 substrates, using 5 replicates for each case.

The selection of 8 cigarettes from the available 32 amounts to choosing exactly a 1/4 fraction of the available cigarette types. In making the selection, we attempted to achieve two objectives:

- (1) to choose cigarettes whose ignition propensities evenly span the entire range of ignition rates observed in the previous testing, and
- (2) to choose cigarettes in a <u>balanced</u> fashion so that each of the five design factors that define the cigarette types would be equally represented among the 8 selected cigarettes.

The statistical theory of fractional factorial experimental design [A-2] can be used to satisfy the second objective. In particular, a 1/4 fraction of the (full) 2⁵ factorial design that defines the 32 cigarettes would consist of 8 cigarettes for which:

- 4 have Burley tobacco, and 4 have Flue-cured;
- 4 have Expanded tobacco, and 4 are Not expanded;
- 4 have paper of Low permeability, and 4 have High permeability;
- 4 have Citrate, and 4 have No citrate; and
- 4 are 21 cm in circumference, and 4 are 25 cm.

In addition, in a fractional factorial experimental design, a second level of balance would be achieved. For example, among the 4 cigarettes with high citrate, 2 would be 21 cm in circumference and 2 would be 25 cm; similarly, the 4 low citrate cigarettes would have 2 at 21 cm and 2 at 25 cm circumference. In an analogous way, each <u>pair</u> of factors would exhibit this kind of balance, with the result that each level of one factor would be combined with each level of the other factor in an equal number of cases.

There are many ways that this kind of fully balanced fractional factorial selection could be made from the 32 cigarette types available. It was initially hoped that one or more of these fractional factorial selections would yield a set of 8 cigarettes that would also satisfy the first objective of uniformly spanning the ignition rates that had been obtained in the previous experiment. Ultimately we found that it was not possible to achieve both of the stated objectives exactly, and so a compromise set of 8 cigarettes was found that was imperfectly, but nearly, balanced and which does exhibit quite uniform coverage of the ignition rates. It was felt that for the purposes of this reevaluation experiment, the need to use cigarettes that uniformly represent the full range of previously observed ignition rates was more important than achieving a perfectly balanced fractional factorial arrangement.

Table A-1 displays the extent to which balance in the above sense was achieved in the final compromise set of cigarettes chosen.

Table A-1. Selection of Cigarettes for Reevaluation Study: Balance on Cigarette Design Factors and Coverage of Levels of Previous Numbers of Ignitions

Cigarette Number	Tobacco Type	Packing Density	Paper Permeability	Citrate	Circumference	Previous Number of Ignitions
106	В	E	L	N	21	1
130	F	Е	L	N	25	4
108	В	E	Н	N	21	7
129	F	E	L	С	25	10
101	В	N	L	С	21	13
131	F	E	Н	С	25	15
103	В	N	Н	С	21	17
120	В	N	Н	N	25	20
Balance Achieved	5 B 3 F	5 E 3 N	4 L 4 H	4 N 4 C	4 21mm 4 25mm	

Results and Conclusions

The eight statistically-selected cigarettes were tested for their ignition propensity on the same substrates and in the same manner as the previous study. In addition to the storage factor, two other differences were a change in the canopy hood used and the technician who performed the tests.

The results of the testing are shown in Table A-2 below.

Table A-2. Reevaluation of Eight of the Thirty-two Experimental Cigarettes

		Number of Ignitions								
	CA, Unc.				SPL/PU ^a Unc./flat		Denim/PU Crev./cov.		Total	
Cig. Id.	Prev.	Now	Prev.	Now	Prev.	Now	Prev.	Now	Prev.	Now
106 BELN21	0	0	1	1	0	0	0	0	1	1
130 FELN25	3	4	1	1	0	2	0	5	4	12
108 BEHN21	3	2	4	3	0	0	0	1	7	6
129 FELC25	5	5	3	5	2	5	0	1	10	16
101 BNLC21	3	4	5	5	5	5	0	5	13	19
131 FEHC25	5	3	5	5	5	5	0	0	15	13
103 BNHC21	5	4	5	5	5	5	2	5	17	19
120 BNHN25	5	5	5	5	5	5	5	5	20	20
Totals	29	27	29	30	22	27	7	22	87	106

Maximum number of ignitions per cigarette is 20, per substrate 40.

CA/CB California test fabric/cotton batting.

SPL/PU 100% cotton Splendor fabric/polyurethane 2045.

Denim/PU 100% cotton Denim fabric/polyurethane 2045.

Unc./flat Uncovered cigarette on a flat mockup.

Crev./cov. Covered cigarette on a nat mockup.

a Cigarette with filter with one half of the tobacco column

removed before lighting.

If no real change in ignition propensity occurred, then the numbers of ignitions in the "Previous" and "Now" columns of Table A-2 should be the same, except for statistical fluctuations.

For each of the 8 cigarette types, and for each of the 4 substrates shown in Table A-2, we calculated the difference between the number of ignitions in the current study ("Now"), minus the number of ignitions in the "Previous" study. If these differences represent only statistical fluctuations, then they would form a statistical population centered near zero. The Wilcoxon Signed Rank Test [A-3] was adopted as a formal statistical test procedure to evaluate whether the observed differences indicate a change in ignition propensity or only random noise. This is a non-parametric test procedure that is valid for use with data that do not follow the commonly assumed Gaussian distribution. Validity for non-Gaussian data was an important consideration because the difference data from this experiment clearly exhibit a non-Gaussian pattern of variation.

The results of the Wilcoxon Signed Rank Test are that the observed differences in numbers of ignitions show a statistically significant tendency (p = 0.04) toward increased ignitions after the storage period. Inspection of Table A-2 shows that the increased ignitions come almost exclusively from the denim substrate, which suggests the possibility that the statistically significant difference is due entirely to the denim substrate. This is consistent with the observation of Rogers and Hayes [A-4] that unless denim is stored free of finishing materials in the dark and in a temperature controlled environment, it will deteriorate with time.

To evaluate the hypothesis of no change in ignition propensity for the non-denim substrates, the Wilcoxon Signed Rank Test was recomputed using only the other three substrates (CA/CB, SPL/PU, and SPL/PU-half cigarette). In this case, the differences in ignition numbers were not significantly different from zero (p = 0.47). That is, the data for the three non-denim substrates are wholly consistent with the hypothesis of no change in ignition propensities of the experimental cigarettes, compared with the previous study.

It was noted that cigarette number 129 showed noticeable increases in the number of ignitions for both of the conditions involving the SPL/PU substrate. This suggests the possibility of a real change in ignition propensity for this particular combination of cigarette and substrate. In pursuing this observation, it is pertinent to note that cigarette 129 showed a relatively small increase in ignitions on the denim substrate both in comparison to its increase for the two SPL/PU substrates and also in comparison to the increases for other cigarettes on the denim substrate. Thus any physical explanation of a change in ignition propensity for cigarette 129 would seem to call for a unique cigarette-substrate interaction on SPL/PU. The reevaluation experiment was designed as an overall test for possible changes in the experimental cigarettes. It was not designed to generate sufficient data to evaluate unique effects for each cigarette and substrate combination. As it happens, the largest single observed difference (2 ignitions in the previous study versus 5 ignitions now) is not significant at the standard 5% level of significance (p = 0.08). Here, the significance calculation was obtained using Fisher's Exact Test for a 2×2 contingency table [A-5]. Based on all these considerations, it does not seem profitable to pursue further the observed increase in ignitions for cigarette 129 on SPL/PU.

Summary

Overall, we interpret the results of this study as showing that the ignition propensities of the experimental cigarettes have not changed during storage but that the denim substrate has changed in ignitability.

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APPENDIX B

MOCK-UP IGNITION TEST METHOD PROCEDURE

Scope:

The purpose of this test procedure is to provide a method for measuring the propensity of cigarettes to ignite specified types of substrate assemblies.

Apparatus and Equipment:

- 1. Test chambers of the design shown in Figure B-1 shall be constructed for testing the cigarette/substrate combinations.
- 2. A vacuum draw apparatus consisting of the following type shall be used for igniting the test cigarettes: See Figure B-2.

The vacuum draw apparatus is composed of a cigarette holder, particulate filter, rotameter, trap, pressure gauge, shut-off valve and a vacuum pump. The cigarette holder (a plastic drying tube) shall have a flexible diaphragm with a hole cut to a size appropriate for holding a cigarette and making a tight seal around the filter end. The holder must be mounted in a secure fastener which will hold the cigarette firmly in a horizontal position for lighting. The particulate filter, composed of a plastic drying tube 150 mm long and 20 mm in diameter filled with glass wool, shall be adequate to remove smoke from the combustion gases to prevent contamination of all downstream assemblies. The filter shall be changed regularly to insure that gas flow is unobstructed and that contamination is not allowed to pass this point. A rotameter capable of measuring 1000 ml/min, air, shall be used for adjusting flow through the vacuum draw system. A 250 ml impinger trap is used in the system to dampen flow variations. A vacuum gauge capable of measuring to at least 800 mm Hg shall be positioned before the pump in order to track apparatus operating pressure. The electric pump shall be capable of producing a vacuum of 500 mm Hg with a flow rate of at least 23 1/min. A Cole-Parmer Air Cadet pump model 7530-40 or equivalent is found to be suitable.

- 3. An environmental conditioning room or chamber shall be maintained which provides area adequate for conditioning cigarettes and test substrate materials. This room shall be capable of maintaining a relative humidity of $55 \pm 5 \%$ and a temperature of $23 \pm 3 \degree C$.
- 4. A constant humidity box of a size to hold 4 to 6 substrate assemblies and more than one 250 ml beaker of conditioned cigarettes is necessary in test rooms where humidity and temperature control is difficult. A shallow tray with a 15 mm deep layer of saturated solution of sodium bisulfate and water has been found to provide the appropriate conditioning environment. (NOTE: This solution is highly corrosive.)

- 5. The conditioning room and test rooms shall be monitored by a recording hygrothermograph (Cole-Parmer Model 8368-00 or equivalent). A Vista Scientific Corporation, battery operated psychrometer or equivalent is used to measure relative humidity in the constant humidity box and to calibrate the hygrothermograph.
- 6. Chemical or canopy hoods are needed for removing combustion products from the test room. Air flows through these hoods shall be at a level which is necessary to remove cigarette and substrate combustion products while not being high enough to influence combustion processes in the test chambers.
- 7. Clean plastic or rubber gloves shall be provided for test operators when handling test substrate materials and cigarettes.
- 8. A laboratory balance capable of weighing to 0.001 g with a repeatability of ± 0.003 g is required for weighing cigarette specimens.
- 9. A butane gas lighter capable of producing a stable luminous flame no longer than 20 mm is required for igniting test cigarettes.
- 10. Water spray bottles or 25 mm diameter wax candles may be used for extinguishing smoldering substrate assemblies. Appropriate fire-proof waste containers shall be used for disposal of the ignition test materials.

Calibrations:

The following are guidelines for basic calibrations of test equipment used in this standard. Time intervals stated in this method for calibrations are considered to be the minimum. Calibrations of equipment shall be carried out at any time when equipment or test conditions indicate that evaluation and recalibration may be necessary.

1. The ignition test chambers shall be checked before use to insure that the front door seals properly and that air movement in the test area does not introduce transient air movement in the test chambers. Door seals are checked visually to insure that they are closed flush against the chamber's side wall and the latching device secures the door tightly. All construction seams shall be inspected to insure that they are air tight and no cracks shall be visible on any surface of the test chamber. If leaks are detected, measures shall be taken to insure that these areas are again made air tight.

Stability of air inside of test chamber shall be determined by making a substrate mock-up and placing it and a lighted cigarette on it in the test position. Observe air movement in the chamber to insure that smoke being emitted by the cigarette is rising vertically and is not showing turbulence within 150 mm above the lit end of the cigarette. If turbulence is noted: 1) the test chamber shall be checked for leaks, 2) the test chamber location shall be evaluated for excess air flow in the laboratory, and 3) air flow rate of the exhaust system shall be evaluated as the source of disturbance. Air flow in the test chamber shall be maintained to

produce a near laminar vertical smoke stream to a minimum distance of 150 mm above the cigarette.

- 2. The vacuum draw apparatus for igniting cigarettes shall be calibrated each week before the beginning of testing. A soap film, bubble flow meter shall be connected to the inlet of the vacuum apparatus at the cigarette attachment point. The vacuum pump is started and adjusted to provide an air flow of 1000 ± 50 ml/min. This flow and range of variation shall be noted and recorded in the laboratory's calibration records for the vacuum system's rotameter.
- 3. The humidity and temperature sensors used to record environmental conditions in the conditioning room/chamber and test room shall be checked for accuracy each week. This shall be accomplished by comparing their results with a calibrated psychrometer and thermometer. A record of calibration adjustments shall be kept for each hygrothermograph used in the conditioning and test rooms.
- 4. The laboratory balance shall be calibrated each week. The calibration is checked by leveling the balance and weighing a calibrated 1.00 g mass. If the balance is found not to be within specifications, make adjustments as needed to bring the device into compliance. Record the results in your laboratory's calibration files.

Test Specimens and Substrates:

Cigarette test specimens and the test substrates are sensitive to contamination. At all times when these materials are handled, clean plastic or rubber gloves shall be worn.

- 1. Cigarette test specimens shall be protected from physical or environmental damage while in storage. It is important that the specimens not be crushed or deformed in any manner. Measures shall be taken to insure that the specimens are not contaminated by foreign materials while in storage and they shall be protected from degradation by insects. If the specimens are to be stored for more than one week, they shall be placed in a freezer reserved for the sole protection of the cigarette specimens.
- 2. Suitable constituent materials for the mock-ups are only those specified in the main body of this report. Bulk materials must be cut to size and then randomized in such a manner as to ensure that samples from any part of the material batch are equally likely to be incorporated in any given mock-up assembly.
- 3. Substrate materials consist of 200 x 200 mm (8 x 8 in) cotton duck fabric swatches and 200 x 200 x 50 mm (8 x 8 x 2 in) polyurethane foam cushions. The substrates are formed by squarely laying the cotton duck fabric flat on top of the foam cushion. Substrate construction may vary by placing various membranes or films, of equal dimensions as the fabric (except thickness), squarely between the cotton duck and foam cushion. All wrinkles shall be smoothed to produce a level test assembly with good contact between the layers.

Conditioning:

- 1. Cigarettes and substrate materials are conditioned at $55 \pm 5\%$ RH and 23 ± 3 °C for 24 hours prior to ignition testing. While conditioning, the cigarettes are contained vertically, with filter up, in clean 250 ml polyethylene or glass beakers with a maximum of 20 cigarettes per beaker to assure free air access to the specimens.
- 2. The substrates, composed of fabric, foam and possibly film materials, are positioned to allow air to circulate around the sides and top of the material. The substrates shall be placed on clean, dry surfaces while being conditioned.
- 3. If the laboratory conditioning room cannot meet the required environmental conditions, a controlled humidity box may be used. Humidity is maintained by the addition of water and chemicals to a holding tray located inside the box. Air in the box must be circulated by means of a small fan in one corner. If a relative humidity box is required, the RH and temperature must be measured with a calibrated wet/dry bulb hygrometer. A battery-driven fan instrument such as a Vista Scientific Corp. psychrometer, or equivalent, has been found to be suitable. Humidity measurements are made in the morning and evening of test day and recorded.

Safety:

- 1. Exhaust systems should be checked daily to insure that they are working. All products of combustion should be removed from the laboratory work area.
- 2. Personnel shall be instructed on general emergency procedures in the laboratory and on procedures to handle an uncontrolled fire.
- 3. Appropriate fire extinguishment equipment shall be provided in each fire test laboratory to extinguish test specimens and to suppress a small fire which may exceed normal controlled limits.
- 4. An appropriate waste container shall be used in each fire test laboratory for safe disposal of specimens and test assemblies after being exposed to heat and fire.

Test Procedure:

- 1. Turn on the exhaust system designated for removal of test combustion products 30 min prior to beginning ignition testing.
- 2. Do not start additional test preparations until relative humidity and temperature in the test room are stabilized within the following respective ranges 55 ± 5 % RH and 23 ± 3 °C. Record the relative humidity and temperature in your laboratory log.
- 3. Wear clean, plastic or rubber gloves when handling cigarettes and substrates.

- 4. Adjust the ignition source, a disposable butane gas lighter, to provide a flame no less than 15 mm in length and no longer than 20 mm.
- 5. If the testing room is separated from the conditioning room, individual substrate assemblies shall be placed into plastic bags and sealed in the conditioning room. They may then be transported to the test room. Test cigarettes may also be transported to the test room in sealed plastic bags.

If the relative humidity and temperature in the test room cannot be maintained at the specified test conditions, the substrates shall be placed in the above mentioned humidity box prior to testing.

- 6. Immediately before selection and ignition of a cigarette for testing, a substrate assembly is removed from its conditioned environment and placed inside the test chamber at the geometric center of its bottom. The square brass frame is placed onto the substrate to maintain fabric flatness. Make sure that the fabric is flat against the foam surface by lightly smoothing with a gloved finger. Do not use fabrics that will not lay flat against the foam.
- 7. Without delay, remove a cigarette from the humidity box. Weigh the cigarette and record the results. Discard the specimen if its weight is more than two standard deviations of the mean obtained from weighing 50 random selected cigarettes of the same design and similarly conditioned. If the cigarette weight is within specifications, a mark is made on the cigarette's paper seam 15 mm from the tobacco end. The mark is made with a #2 or softer graphite pencil. The cigarette filter is inserted into the vacuum draw apparatus rubber diaphragm and held in a horizontal position. Start the vacuum draw apparatus, and make sure the center of the rotameter's indicator ball is within ± 50 ml/min of 1000 ml/min. Immediately ignite the cigarette with a preadjusted butane lighter.

The ignition flame is held to the tobacco end of the cigarette for three seconds to achieve uniform ignition. The lit cigarette is then removed from the diaphragm, held vertically, coal up, under a 600 ml beaker and moved to the test chamber. With the chimney on the test chamber covered, the door is opened, and the lit cigarette is placed vertically, filter down, into a holder located on the center of the substrate assembly. Simultaneously, the door is closed gently and the chimney cover removed. Smoke from the cigarette should pass directly out of the chamber stack. If the cigarette should self-extinguish while in the cigarette holder and before removal to be placed onto the test substrate, terminate the test and identify the results as a self-extinguishment. When the cigarette has burned to the 15 mm mark, simultaneously cover the chimney and carefully open the chamber door. With care, the cigarette is removed from the holder, and the holder is placed in a front corner of the test chamber. The cigarette, with ash still attached, is gently placed onto the top of the substrate assembly so that the coal is located at the geometric center of the surface and the cigarette angled 45° to the fabric warp (i.e., diagonally). See Figure B-3. The cigarette paper seam shall be turned up. Do not drop the cigarette onto the substrate, and do not press the coal onto the substrate. Without delay, simultaneously remove the chimney cover, and gently close the door. A stopwatch is started. If the ash falls off during any part of the transport or positioning process, immediately extinguish the cigarette and begin with paragraph 7 in this section.

- 8. The burning cigarette and substrate are observed. The smoke plume near the cigarette should remain undisturbed; if not, note your observations on the test sheet. A time record is kept from when the cigarette is placed onto the substrate assembly until either:
 - (1) self extinguishment (the coal goes out before the tobacco column is consumed),
 - (2) non-ignition (the tobacco column burns to the end without causing the substrate to smolder),
 - (3) borderline ignition (ignition appears, but a char does not propagate more than 10 mm, then goes out, while the tobacco column burns to completion), or
 - (4) ignition (the substrate starts to smolder, and the char propagates away from the burning tobacco column by at least 10 mm).

The first three of these observations are considered to be non-ignitions. The fourth is the only outcome that is considered to be an ignition.

It may be helpful to darken the room intermittently to observe if ignition is occurring. Record ignition time on the test sheet. After recording the ignition time, observe the substrate for 2 to 3 minutes to insure that smoldering has begun. Observations are also made to document the area of fabric char expansion to at least a 10 mm point from where ignition occurred.

- 9. Smoldering substrates are extinguished with water from a squirt bottle or by prodding the smoldering area with a candle. The time at which extinguishment occurs is recorded. It should be noted that candles are generally used when specimens are to be retained for further study. If candles are used for extinguishing, be sure that all combustion processes have stopped before storing the test assembly. All substrates used in testing are stored in metal containers until they are disposed of in a safe and acceptable manner as determined by the testing facility.
- 10. After a test is completed, leave the test chamber door open to allow air to circulate throughout its volume. After the chamber has been cleared, preparations may begin for additional testing.

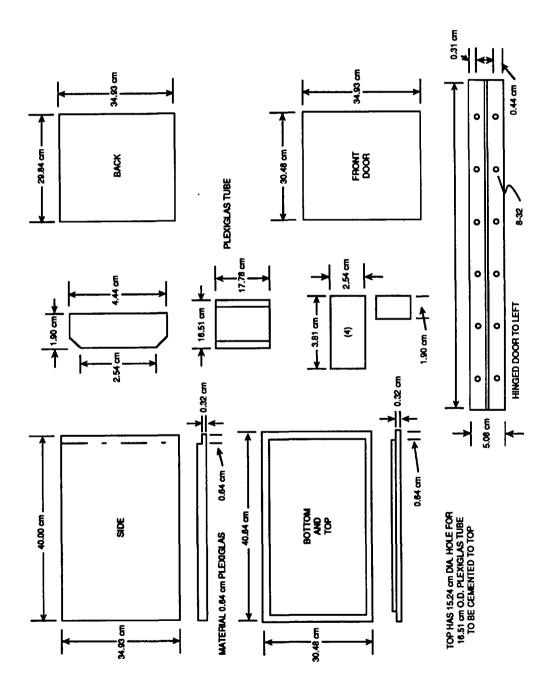


Figure B-1. Schematic of Test Chamber Components.

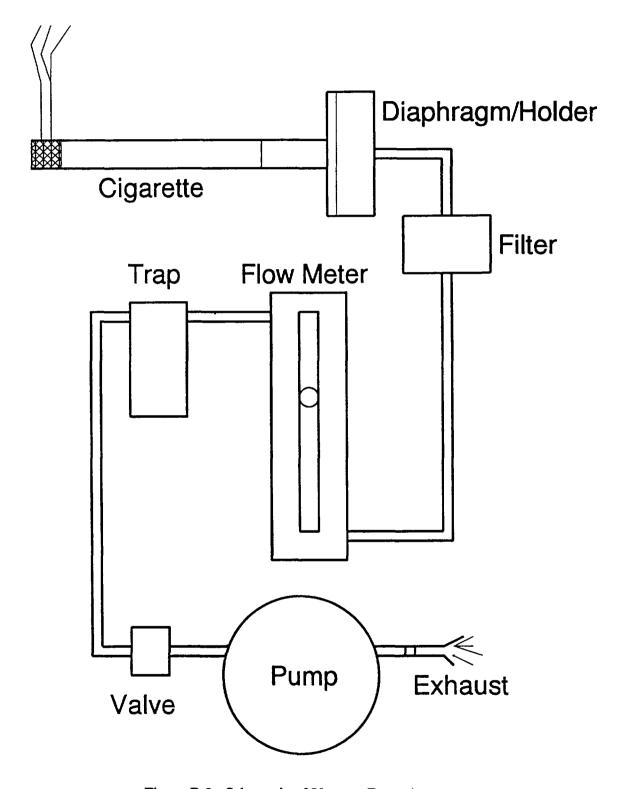


Figure B-2. Schematic of Vacuum Draw Apparatus.

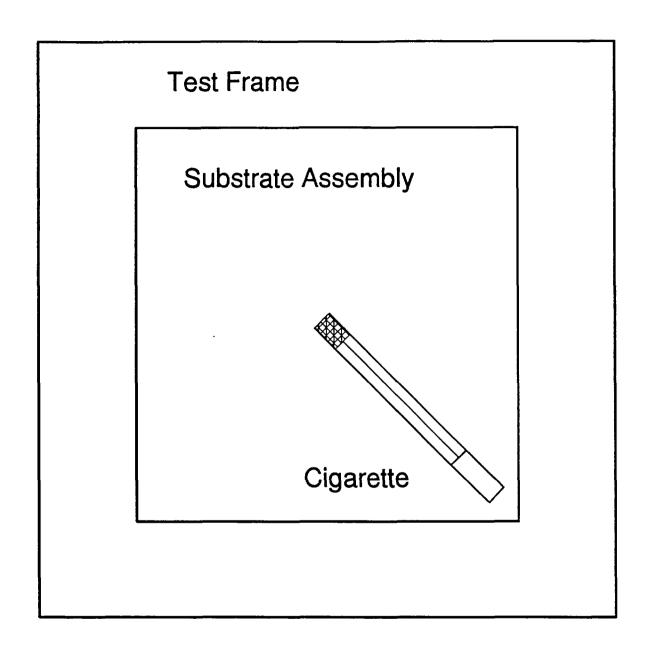


Figure B-3. Location of Cigarette on Mock-Up Method Substrate Assembly.

APPENDIX C

ESTIMATE OF OXYGEN SUPPLY PATHS TO CIGARETTE COAL ATOP A FLAT UPHOLSTERY SUBSTRATE

The physical system considered here is a lighted cigarette smoldering atop a flat mock-up consisting of a fabric over a polyurethane foam. The issue of concern is the relative importance of oxygen supply to the cigarette coal via two paths: (1) from ambient air above the fabric plane and (2) from ambient air contained within the polyurethane foam below the fabric plane. If the coal receives an appreciable portion of its oxygen supply from below the fabric plane then the ability of oxygen to penetrate the fabric will be important to the behavior of that coal.

This system is three-dimensional and time-dependent; this precludes any exact approach to assessment of the oxygen supply pathways of interest. It will be necessary to make estimates based on the information available.

The total oxygen demand of the smoldering coal is easily calculated from its mass burning rate and the overall stoichiometry of the smolder process. The mass burning rate of the TSG cigarettes in free burn varied in the range 5.7 - 15.5x10⁻⁴ g/s [C-1]. A cigarette studied by Baker [C-2] appears to fall close to the middle of this range (taken here as $10x10^{-4}$ g/s), using the smolder velocity he reports and an estimated packing density of 0.3 g/cm³. The TSG study showed that the smolder velocity of a cigarette placed on a horizontal substrate of the type considered here will slow down about 20-25%. Thus the oxygen demand is slowed proportionately. For Baker's cigarette we thus estimate the mass burning rate on the substrate to be approximately 7.5 - 8.0x10⁻⁴ g/s. The stoichiometry of free burn cigarettes has been measured by Muramatsu for a variety of tobacco blends; the results fall in the range 1.4 - 1.9x10⁻² moles of oxygen per gram of tobacco consumed [C-3]; this converts to a range of 0.45 - 0.61 grams oxygen/ gram of tobacco. Then the oxygen demand for the coal smoldering on the substrate is estimated to be in the range 3.4 - 4.9x10⁻⁴ grams of oxygen/second.

Baker measured the oxygen concentration profiles around his cigarette coal in free burn [C-2]. These profiles can be used to estimate the amount of oxygen diffusing from the air above the fabric plane to the coal. These profiles will be changed by the proximity of the substrate; to a first approximation all oxygen which Baker's profiles would show reaching the coal from below (in free burn) is blocked by the presence of the substrate. In addition, the profiles are such that the gradient of oxygen above the coal is substantially less than elsewhere because of the rising smoke plume. Thus, the significant diffusive routes through the oxygen boundary layer on the coal are from the sides and from the front. Inspection of the oxygen profiles presented in reference F-2 suggests that an average oxygen diffusion length on the sides of the coal is 2.8 mm for a distance from 4 mm behind the paper burn line to 4 mm beyond it. Then the mass of oxygen diffusing through these boundary layer profiles is calculated as follows.

 $m_{SD} = A_{SD} \rho D(\Delta Y_{OS} / \Delta x)$

where m_{SD} is the mass of oxygen entering the coal by diffusion from the sides; A_{SD} is the cigarette surface area through which this oxygen enters (1/4 of the periphery and an 8 mm length yields an area of 1.0 cm²); ρ is the gas density in the boundary layer; D is the diffusivity of oxygen in the boundary layer; ΔY is the oxygen mass fraction change across the boundary layer; Δx is the boundary layer thickness. Using a mean boundary layer temperature of 150 °C, one makes the following estimate of m_{SD} :

$$m_{SD} \approx (1.0)(7.9 \times 10^{-4})(0.41)(0.2/0.28) = 2.3 \times 10^{-4} \text{ g O}_2/\text{sec}$$

The oxygen entering the front of the coal is more difficult to estimate from Baker's profiles; here it is taken to be comparable to the rate above but modified by the lesser area for diffusion from that direction (0.5 cm^2) , the cross-sectional area of the cigarette). This means that the above value is multiplied by 1.5 to get an estimate of the total oxygen inflow from the ambient air above the fabric plane (designated m_{AFP}):

$$m_{AFP} \approx 3.4 \times 10^{-4} \text{ g O}_2/\text{sec}$$

It should be noted that this number is comparable to the estimate above of the total oxygen demand of the coal.

Consider next the issue of oxygen supply to the coal from below the fabric plane. There is essentially no pressure force to cause air to flow upward from the cells of the polyurethane foam, through the fabric and into the cigarette coal. The air within the foam cells is being heated from above; only the finite spatial extent of the heated zone in the lateral (fabric plane) direction implies the application of a weak buoyancy force acting over the very short height of the thermal layer in the foam. This is opposed by the flow resistance of the foam and the fabric. Diffusion of oxygen is less inhibited in the foam because its very open cell structure poses little blockage. Thus oxygen diffusion out of the foam is the main process of interest here. We have no information on the diffusion of oxygen through the less porous structure of a fabric so it will not be included here; the result will be an overestimate of the supply of oxygen able to diffuse out of the foam and to the cigarette coal.

The relaxation time of the diffusion process in the foam is short compared to the time required for the coal to move a distance equal to its own length. This means that the oxygen profiles in the foam will be little affected by the fact that the cigarette coal is trying to move slowly over the top of the fabric. Then these profiles can be accurately estimated using the solution to a simple transient problem: diffusion into a sink of finite radius from a semi-infinite medium. This problem is treated in reference C-4; the solution for the spherically-symmetric oxygen concentration profiles as a function of distance from the sink (the cigarette coal) and time (here, time since the coal made contact with the substrate) is as follows:

$$\frac{(C-C_0)}{(C_1-C_0)} = (\frac{a}{r}) \ erfc(\frac{(r-a)}{2(Dt)^{1/2}})$$

Here C is the mass concentration of oxygen at radius r at time t; C_0 is the initial concentration of oxygen in the foam (essentially same as ambient); C_1 is the concentration maintained from time zero

onward at the surface of the oxygen sink (here $C_1 = 0$); a is the radius of the oxygen sink (taken here as 0.35 cm, comparable to the radius of the cigarette); erfc indicates that the error function complement is to be evaluated for the quantity in the brackets.

The oxygen flux of interest is that into the sink (coal) at r = a. This is given by the following:

$$m_{RFP} = -2\pi a^2 D \left(\frac{\partial C}{\partial r}\right)_{r=a}$$

Here the derivative is found from the equation above to yield the following:

$$m_{BFP} = 2\pi a^2 D(C_1 - C_0)[(\frac{1}{a}) + \frac{1}{(\pi Dt)^{1/2}}]$$

Using a value of oxygen diffusivity of 0.15 cm²/s in the much cooler and slightly obstructed space filled by the foam, one finds that this reduces to:

$$m_{BFP} = 3.15 \times 10^{-5} [2.86 + \frac{1.46}{t^{1/2}}]$$

This can then be used to calculate the diffusion flux from the foam space to the coal as a function of time after placement of the cigarette on the substrate. See Table C-1 below.

Table C-1. Calculated Mass Flux of Oxygen from Foam to Coal

Time (sec)	m _{BFP} (g O ₂ /sec)
5	1.1x10 ⁻⁴
10	1.0x10 ⁻⁴
30	9.8x10 ⁻⁵
100	9.5x10 ⁻⁵
300	9.3x10 ⁻⁵

Since this starts as an infinite diffusive flux at time zero, it is apparent that in only a few seconds it settles down to a nearly constant, low oxygen supply rate. Recall that this does not account for the diffusive resistance of the fabric for which we have no estimate. The actual flux to the coal is thus

expected to be smaller than that above, which is already only about 1/4 of the oxygen supply available from above the fabric plane. The implication is that changes in fabric diffusion resistance with changes in fabric structure or weight can be expected to be secondary in their impact on the behavior of the cigarette coal.

Ignition of the fabric itself, below the cigarette coal, probably relies significantly on the above oxygen flux, at least until the reaction zone can spread outward slightly to the point of getting more oxygen from above the fabric plane. Variations in fabric diffusion resistance would not affect this appreciably; the above estimate does not depend on fabric diffusion resistance.

REFERENCES

- [C-1] Gann, R., Harris, R., Krasny, J., Levine, R., Mitler, H. and Ohlemiller, T, "The Effect of Cigarette Characteristics on the Ignition of Soft Furnishings," NBS Technical Note 1241, U.S. National Bureau of Standards, Gaithersburg MD, 1987.
- [C-2] Baker, R., Beitr. z. Tabakforsch. Int'l, 11, 1982, p.181.
- [C-3] Muramatsu, M., Umemura, S., and Okada, T., Nippon Kagaku Kaishi, 10, 1978, p.1441.
- [C-4] Crank, J., The Mathematics of Diffusion, Oxford at Clarendon Press, London, (1956) p. 98.

APPENDIX D

METAL ION CONTENT OF FABRICS: TEST METHOD AND RESULTS

The test method for alkali metal and alkaline earth ions in the cotton duck fabrics consisted of an acid extraction process followed by analysis of the resultant solution by ion chromatography.

Extraction:

- 1. 100 ml of 4.6 mM HNO₃ poured into 125 ml plastic bottles.
- 2. 2.5 x 2.5 cm fabric samples immersed.
- 3. Overnight extraction with agitation at ambient temperature.
- 4. 5.0 or 10.0 ml aliquot diluted to 50 ml in at least 18.3 megohm-cm water for ion chromatography analysis.

Analysis:

1. Method C-207 from the following source:

Heckenburg, A., Alden, P., Wildman, B., Krol, J., Romano, J. Jackson, P., Jandik, P. and Jones, W., <u>Waters Innovative Methods for Ion Analysis</u>, Millipore Corporation, Manual #22340, 1989

2. Conditions:

Instrument: Waters ILC-1 Ion/Liquid Chromatograph.

Eluent: 0.1 mM EDTA/3.0 mM HNO₃.

Column: IC-Pak C M/D.

Flow rate: 1.0 ml/min.

Injection: 100 µL.

Standard: 1.0 ppm of Na⁺, K⁺, Mg⁺², and Ca⁺².

Detection: Conductivity.

The results for samples from the three cotton ducks used in this study are shown in the following tables.

Table D-1. Cation Content of #4 Cotton Duck

		Cation Content (ppm)						
Sample ID	Na ⁺	K+	Mg ⁺²	Ca ⁺²				
4-46-10-1	<10	4608	612	698				
4-46-40-2	<10	4688	622	713				
4-46-72-2	<20	4429	586	663				
4-48-10-5	<10	4273	580	681				
4-48-44-1	<10	4201	577	680				
4-48-78-3	<10	4254	588	689				
4-50-1-1	<20	4475	574	660				
4-50-30-1	<10	4372	554	555				
4-50-75-1	<10	4535	579	652				
4-50-112-5	<20	4525	561	562				
4-52-1-3	<10	4600	579	595				
4-52-36-5	<20	4646	579	616				
4-52-68-2	<10	4457	571	586				
4-52-72-3	<10	4372	515	501				
4-52-106-2	<10	4654	587	575				
4-54-1-2	<20	4587	555	545				
4-54-1-3	<10	4477	550	591				
4-54-31-2	35	4463	562	581				
4-54-66-3	21	4544	563	549				
4-54-77-3	22	4569	560	581				
4-56-35-6	<20	4527	565	565				
4-56-66-1	<10	4447	566	583				
4-56-90-3	<10	4550	566	545				
4-56-121-5	<10	4514	559	563				

Table D-2. Cation Content of #6 Cotton Duck

		Cation Con	tent (ppm)	
Sample ID	Na ⁺	K+	Mg ⁺²	Ca ⁺²
6-65-4-1	<10	5571	655	766
6-65-30-2	<10	5423	633	733
6-65-60-3	<10	5659	658	755
6-65-90-6	<20	5905	649	743
6-65-121-5	<20	5775	669	745
6-67-1-43	25	5872	675	753
6-67-32-2	<10	6029	651	743
6-67-60-5	29	5846	661	734
6-67-90-4	32	5766	642	692
6-67-121-2	<10	5986	652	712
6-69-36-4	<10	4443	565	557
6-69-61-1	44	4514	579	584
6-69-95-6	<20	4950	596	622
6-69-120-3	22	4383	551	535
6-71-26-62	<10	5797	640	723
6-71-64-4	<10	5813	642	710
6-71-90-2	29	5765	644	685
6-71-131-1	<20	5591	604	640
6-73-34-5	<20	4276	562	640
6-73-60-4	<10	4501	588	662
6-73-91-6	<10	4540	583	649

Table D-3. Cation Content of #10 Cotton Duck

	Cation Content (ppm)						
Sample ID	Na ⁺	K+	Mg ⁺²	Ca ⁺²			
10-57-4-3	<10	4474	610	716			
10-57-34-1	<10	4551	614	704			
10-57-65-2	<10	4471	608	736			
10-57-91-4	<10	4463	609	705			
10-57-121-3	<10	4562	617	717			
10-57-152-3	47	4326	593	683			
10-57-182-3	<10	4510	618	709			
10-57-182-3	<10	4312	593	682			
10-57-216-3	49	4384	602	719			
10-57-243-4	22	4393	601	710			
10-58-33-3	58	4194	573	684			
10-58-61-2	28	4172	576	689			
10-58-61-2	51	4318	594	715			
10-59-2-23	<10	4342	612	718			
10-59-32-4	<20	4369	605	695			
10-59-61-3	<10	4522	616	739			
10-59-90-2	<20	4465	611	707			
10-59-123-1	<10	4348	585	661			
10-59-149-1	<10	4481	613	694			
10-59-180-3	<10	4274	581	690			
10-59-216-1	<20	4559	622	698			
10-59-241-1	<10	4435	600	680			

Table D-3. (cont.) Cation Content of #10 Cotton Duck

	Cation Content (ppm)						
Sample ID	Na ⁺	K ⁺	Mg ⁺²	Ca ⁺²			
10-61-32-4	57	4064	577	669			
10-61-59-3	<10	4190	579	660			
10-61-86-1	<10	4225	595	667			
10-61-124-3	<10	4274	605	665			
10-61-156-2	<10	4365	594	666			
10-63-31-43	121	4081	575	678			
10-63-63-33	74	4155	542	605			
10-63-96-33	95 _	4110	593	671			
10-63-123-4	95	4110	593	671			
10-63-161-1	55	4206	584	687			

APPENDIX E

CIGARETTE EXTINCTION TEST METHOD PROCEDURE

Scope:

The purpose of this test procedure is to provide a method for measuring the propensity of cigarettes to ignite specified types of substrate assemblies.

Apparatus and Equipment:

- 1. Test chambers of the design shown in Figure B-1 shall be constructed for testing the cigarette/substrate combinations.
- 2. A support holder for the substrate-cigarette assembly shall be constructed; see Figure E-1.
- 3. A vacuum draw apparatus consisting of the following type shall be used for igniting the test cigarettes: See Figure B-2 of Appendix B.

The vacuum draw apparatus is composed of a cigarette holder, particulate filter, rotameter, trap, pressure gauge, shut-off valve, and a vacuum pump. The cigarette holder (a plastic drying tube) shall have a flexible diaphragm with a hole cut to a size appropriate for holding a cigarette and making a tight seal around the filter end. The holder must be mounted in a secure fastener which will hold the cigarette firmly in a horizontal position for lighting. The particulate filter, composed of a plastic drying tube 150 mm long and 20 mm in diameter filled with glass wool, shall be adequate to remove smoke from the combustion gases to prevent contamination of all down stream assemblies. The filter shall be changed regularly to insure that gas flow is unobstructed and that contamination is not allowed to pass this point. A rotameter capable of measuring 1000 ml/min, air, shall be used for adjusting flow through the vacuum draw system. A 250 ml impinger trap is used in the system to dampen flow variations. A vacuum gauge capable of measuring to at least 800 mm Hg shall be positioned before the pump in order to track apparatus operating pressure. The electric pump shall be capable of producing a vacuum of 500 mm Hg with a flow rate of at least 23 l/min. A Cole-Parmer Air Cadet pump model 7530-40 or equivalent is found to be suitable.

- 4. An environmental conditioning room or chamber shall be maintained which provides an area adequate for conditioning cigarettes and test substrate materials. This room shall be capable of maintaining a relative humidity of $55 \pm 5\%$ and a temperature of 23 ± 3 °C.
- 5. A constant humidity box of a size to hold two to three boxes of filter paper and more than one 250 ml beaker of conditioned cigarettes is necessary in the test room if humidity and temperature control are difficult.

- 6. The conditioning room and test rooms shall be monitored by a recording hygrothermograph (Cole-Parmer Model 8368-00 or equivalent). A Vista Scientific Corporation battery-operated psychrometer or equivalent is used to measure relative humidity in the constant humidity box and to calibrate the hygrothermograph.
- 7. Chemical or canopy hoods are needed for removing combustion products from the test room. Air flows through these hoods shall be at a level which is necessary to remove cigarette and substrate combustion products while not being high enough to influence combustion processes in the test chambers.
- 8. Clean plastic or rubber gloves shall be provided for test operators when handling test substrate materials and cigarettes.
- 9. A laboratory balance capable of weighing to 0.001 g with a repeatability of \pm 0.003 g is required for weighing cigarette specimens.
- 10. A butane gas lighter capable of producing a stable luminous flame no longer than 20 mm is required for igniting test cigarettes.
- 11. An appropriate fire-proof waste container shall be used for disposal of the ignition test materials.

Calibrations:

The following are guidelines for basic calibrations of test equipment used in this standard. Time intervals stated in this method for calibrations are considered to be minimum. Calibrations of equipment shall be carried out at any time when equipment or test conditions indicate that evaluation and recalibration may be necessary.

1. The ignition test chamber shall be checked before use to insure that the front door seals properly and that air movement in the test area does not introduce transient air movement in the test chamber. Door seals are checked visually to insure that they are closed flush against the chamber's side wall and the latching device secures the door tightly. All construction seams shall be inspected to insure that they are air tight and no cracks shall be visible on any surface of the test chamber. If leaks are detected, measures shall be taken to insure that these areas are again made air tight.

Stability of air inside of the test chamber shall be determined by mounting a single filter paper in the substrate assembly holder and placing it together with a lighted cigarette in the test position within the test chamber. Observe air movement in the chamber to insure that smoke being emitted by the cigarette is rising vertically and is not showing turbulence within 150 mm above the lit end of the cigarette. If turbulence is noted: 1) the test chamber shall be checked for leaks; 2) the test chamber location shall be evaluated for excess air flow in the laboratory; 3) air flow of the exhaust system shall be evaluated as the source of disturbance. Air flow in the test chamber shall be maintained to produce a near laminar vertical smoke stream to a minimum distance of 150 mm above the cigarette.

- 2. The vacuum draw apparatus for igniting cigarettes shall be calibrated each week before the beginning of testing. A soap film bubble flowmeter shall be connected to the inlet of the vacuum apparatus at the cigarette attachment point. The vacuum pump is started and adjusted to provide an air flow of 1000 ± 50 ml/min. This flow and range of variation shall be noted and recorded in the laboratory's calibration records for the vacuum system's rotameter.
- 3. The humidity and temperature sensors used to record environmental conditions in the conditioning room/chamber and test room shall be checked for accuracy each week. This shall be accomplished by comparing their readings with a calibrated psychrometer and thermometer. A record of calibration adjustments shall be kept for each hygrothermograph used in the conditioning and test rooms.
- 4. The laboratory balance shall be calibrated each week. The calibration is checked by leveling the balance and weighing a calibrated 1.00 g mass. If the balance is found not to be within specifications, make adjustments as needed to bring the device into compliance. Record the results in your laboratory's calibration files.

Test Specimens and Substrates:

Cigarette test specimens and the test substrates are sensitive to contamination. At all times when these materials are handled, clean plastic or rubber gloves shall be worn.

- 1. Cigarette test specimens shall be protected from physical or environmental damage while in storage. It is important that the specimens not be crushed or deformed in any manner. Measures shall be taken to insure that specimens are not contaminated by foreign materials while in storage, and they shall be protected from degradation by insects. If the specimens are to be stored for more than one week, they shall be placed in a freezer reserved for the sole protection of the cigarette specimens.
- 2. Substrate materials consist of 150 mm diameter circles of Whatman #2 filter paper. Substrates are formed by placing multiple layers of filter paper into the holder assembly. The filter paper should be placed in the holder assembly with the brass ring on top of the specified number of filter papers to ensure good contact between the layers.

Conditioning:

1. Cigarettes and substrate materials are conditioned at 55 ± 5 % RH and 23 ± 3 °C for at least 24 hours prior to ignition testing. While conditioning, the cigarettes are contained vertically, with filter up, in clean 250 ml polyethylene or glass beakers with a maximum of 20 cigarettes per beaker to assure free air access to the specimens.

- 2. The substrate filter paper sheets are supplied in boxes of 100 sheets. These boxes shall be opened and placed in the conditioning room along with the cigarettes. There is no need to remove the sheets from the box as long as the top of the box is completely opened. The boxes are positioned to allow air circulation around each box. Each box of filter papers should be conditioned for a minimum of one week prior to use.
- 3. If the laboratory conditioning room cannot meet the required environmental conditions, a controlled humidity box may be used. Humidity is maintained by the addition of water and chemicals to a holding tray located inside the box. Air in the box must be circulated by means of a small fan in one corner. If a relative humidity measurement is required, the RH and temperature must be measured with a calibrated wet/dry bulb hygrometer. A battery-driven fan instrument such as a Vista Scientific Corp. psychrometer, or equivalent, has been found to be suitable. Humidity measurements are made in the morning and evening of each test day and recorded.

Safety:

- 1. Exhaust systems should be checked daily to insure that they are working. All products of combustion should be removed from the laboratory work area.
- 2. Personnel shall be instructed on general emergency procedures in the laboratory and on procedures to handle an uncontrolled fire.
- 3. Appropriate fire extinguishing equipment shall be provided in each fire test laboratory to extinguish test specimens and to suppress a small fire which may exceed normal controlled limits.
- 4. An appropriate waste container shall be used in each fire test laboratory for safe disposal of specimens and test assemblies after being exposed to heat and fire.

Test Procedure:

- 1. Turn on the exhaust system designated for removal of test combustion products 30 min prior to beginning ignition testing.
- 2. Do not start test preparations until relative humidity and temperature in the test room are stabilized within the following respective ranges 55 ± 5 % RH and 23 ± 3 °C. Record the relative humidity and temperature in your laboratory log.
- 3. Wear clean, plastic or rubber gloves when handling cigarettes and substrates.
- 4. Adjust the ignition source, a disposable butane gas lighter, to provide a flame no less than 15 mm in length and no longer than 20 mm.

5. If the relative humidity and temperature in the test room cannot be maintained at the specified test conditions, the filter papers shall be placed into plastic bags and sealed in the conditioning room. They may then be transported to the test room. Test cigarettes may also be transported to the test room in sealed plastic bags.

If the relative humidity and temperature in the test room cannot be maintained at the specified test conditions, the filter papers shall be placed in the above mentioned humidity box prior to testing.

- 6. Immediately before selection and ignition of a cigarette for testing, a substrate assembly is removed from its conditioned environment and placed inside the test chamber at the geometric center of its bottom. The brass ring is placed onto the filter paper substrate. Do not use filter papers that will not lay flat in the holder assembly.
- 7. Without delay, remove a cigarette from the humidity box. Weigh the cigarette and record the results. Discard the specimen if its weight is more than two standard deviations of the mean obtained from weighing 50 randomly selected cigarettes of the same design and similarly conditioned. If the cigarette weight is within specifications, a mark is made on the cigarette's paper seam 15 mm from the tobacco end. The mark is made with a #2 or softer graphite pencil. The cigarette filter is inserted into the vacuum draw apparatus rubber diaphragm and held in a horizontal position. Start the vacuum draw apparatus, and make sure the center of the rotameter's indicator ball is within ± 50 ml/min of 1000 ml/min. Immediately ignite the cigarette with a preadjusted butane lighter.

The ignition flame is held to the tobacco end of the cigarette for three seconds to achieve uniform ignition. The lit cigarette is then removed from the diaphragm, held vertically, coal up, under a 600 ml beaker and moved to the test chamber. With the chimney on the test chamber covered, the door is opened, and the lit cigarette is placed vertically, filter down, into a holder located on the center of the substrate assembly. Simultaneously, the door is closed gently and the chimney cover removed. Smoke from the cigarette should pass directly out of the chamber stack. If the cigarette should self-extinguish while in the cigarette holder and before removal to be placed onto the test substrate, terminate the test and identify the results as a self-extinguishment. When the cigarette has burned to the 15 mm mark, simultaneously, cover the chimney and carefully open the chamber door. With care, the cigarette is removed from the holder, and the holder is placed in the front corner of the test chamber. The cigarette, with the ash still attached is gently placed onto the top of the substrate assembly so that the filter end of the cigarette is placed between the appropriately sized cigarette antiroll fingers; see Figure E-2. The cigarette paper seam shall be turned up. Do not drop the cigarette onto the substrate and do not press the coal onto the substrate. Without delay, simultaneously remove the chimney cover and gently close the door. A stopwatch is started. If the ash falls off during any part of the transport or positioning process, immediately extinguish the cigarette and begin with paragraph 7 in this section.

- 8. The burning cigarette and substrate are observed. The smoke plume near the cigarette should remain undisturbed; if not, note your observations on the test sheet. A time record is kept from when the cigarette is placed onto the substrate assembly until either:
 - (1) self-extinguishment (the coal goes out before the tobacco column is consumed),
 - (2) total burn (the tobacco column burns to the end).

It may be helpful to darken the room intermittently or use a dark background behind the test chamber to observe if the cigarette continues to burn. Record the time that the cigarette stops burning on the test sheet. After recording the time, observe the substrate assembly for 1 to 2 minutes to insure that smoldering has stopped. Measurements are made to document the length of unburned cigarette for those not burning to the filter. Record this measurement to the nearest mm.

9. After a test is completed, leave the test chamber door open to allow air to circulate throughout the chamber volume. After the chamber has been cleared, preparations may begin for additional testing.

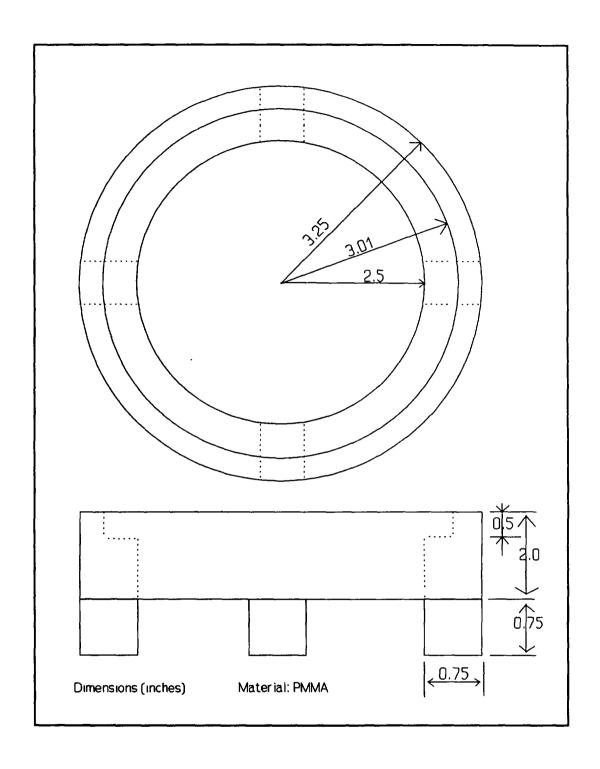


Figure E-1. Details of the Filter Paper Holder Support Structure.

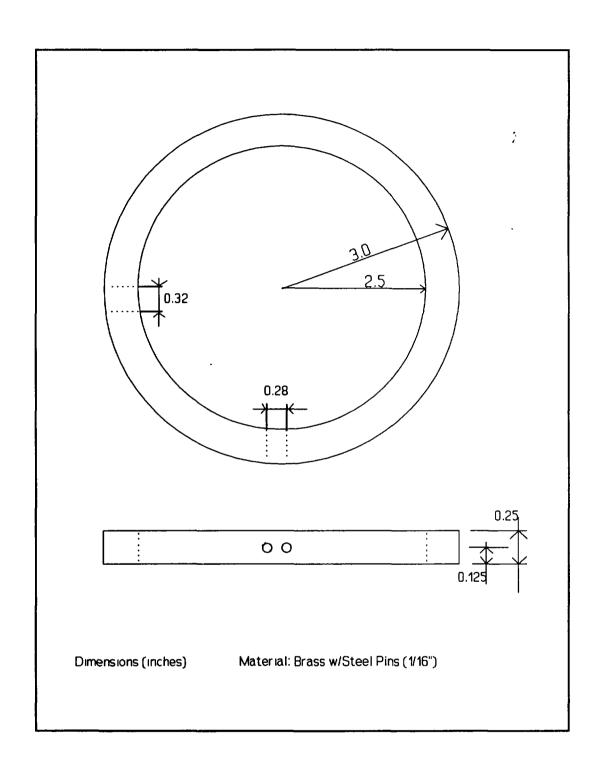


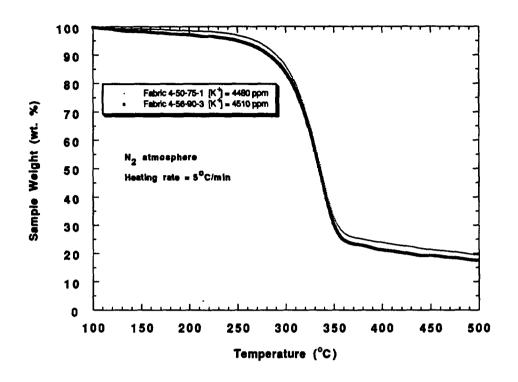
Figure E-2. Brass Holddown Ring and Cigarette Motion Restrainers.

APPENDIX F

REPRESENTATIVE THERMOGRAVIMETRIC DATA FOR TEST METHOD MATERIALS

Samples of all of the test materials were subjected to thermogravimetric analysis in a Mettler TA 2000C Thermoanalyzer. All tests were run with the samples in aluminum oxide crucibles 8.0 mm OD by 4.5 mm depth. Sample weight in all cases was 2.0 ± 0.1 mg. For the cotton ducks this required four to five pieces of yarn removed from near the location where metal ion concentrations had been measured; these yarn pieces were laid in an open, cross-wise array on the bottom of the crucible. The polyethylene film and filter paper samples were in the form of disks on the bottom of the crucible. The polyurethane foam was in the form of a few small chunks, ca. 1 mm, removed with a tweezers from well beneath any previously exposed surface of the foam block being sampled. The heating rate was 5 °C/min. and the atmosphere was 100% nitrogen (< 100 ppm of oxygen). Samples were run up to 80 °C and held there until dry; they were then heated at the noted rate to 500 °C.

The results are shown in Figures F-1 to F-6. In most cases the original data (weight vs. temperature) plus a numerically-derived rate of weight loss are both shown. Samples from two sources of each material are shown. For the polyurethane foam two buns were sampled, as well as two locations within each bun. For the cotton ducks, the potassium level in the adjacent material is reported. Note that the only material showing appreciable variability is the polyethylene film.



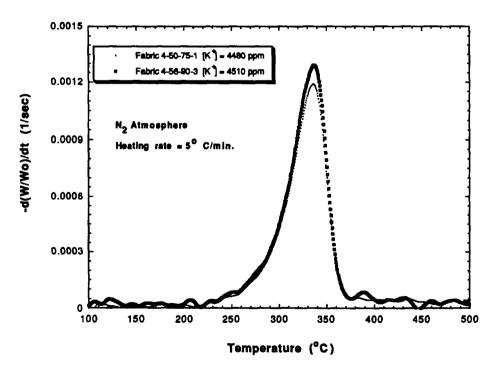
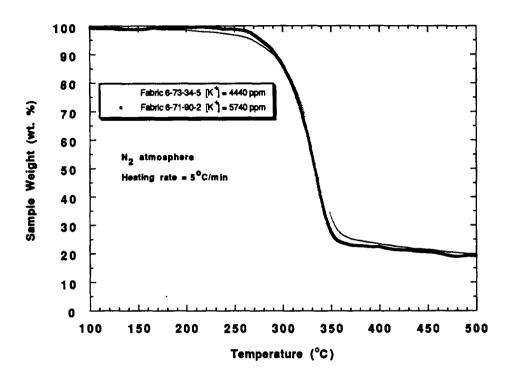


Figure F-1. TG Behavior of Two Samples of Cotton Duck #4.



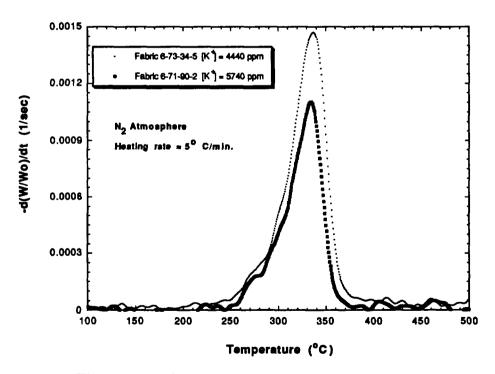
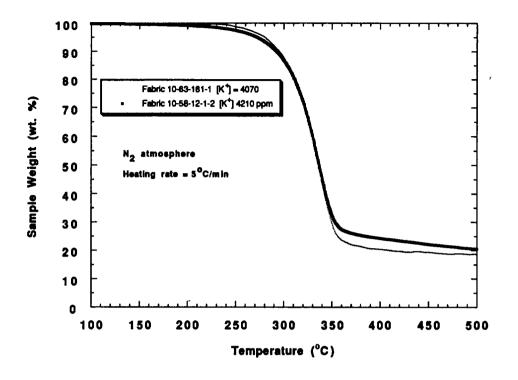


Figure F-2. TG Behavior of Two Samples of Cotton Duck #6.



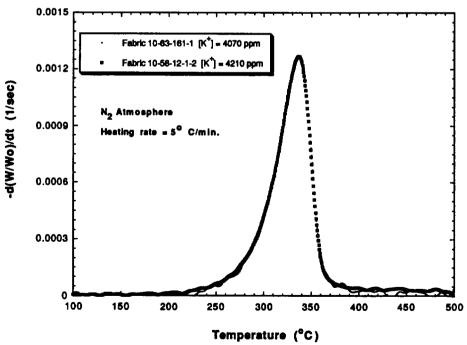
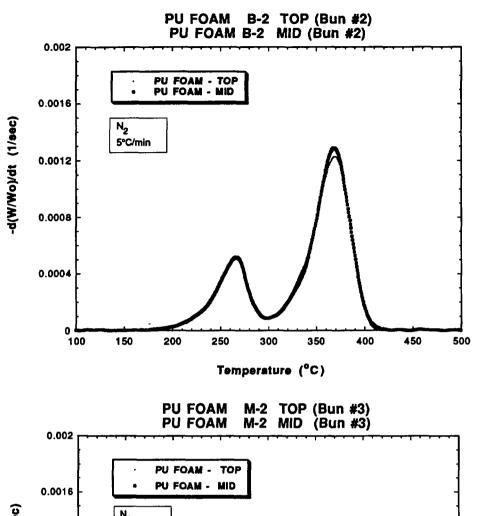


Figure F-3. TG Behavior of Two Samples of Cotton Duck #10.



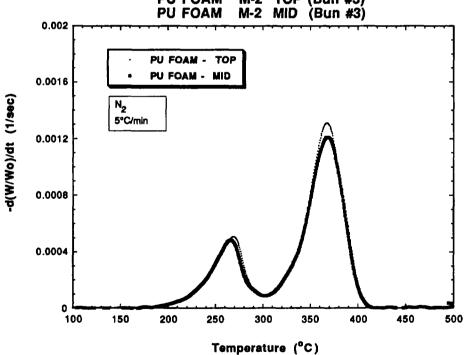
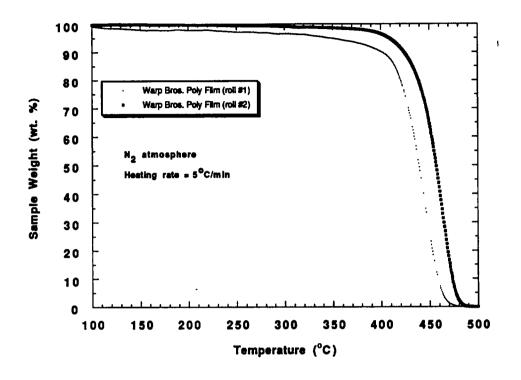


Figure F-4. Derivative TG Data: Two Polyurethane Foam Samples from Top and Bottom of Original Bun.



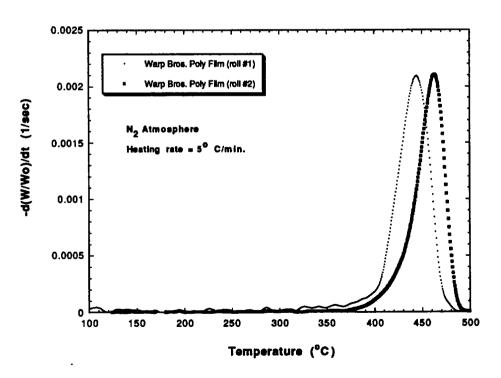
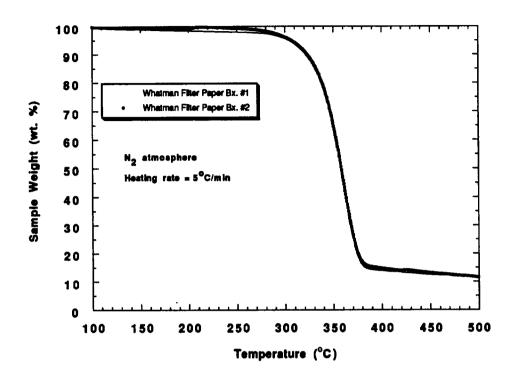


Figure F-5. TG Data for Two Samples of Polyethylene Film.



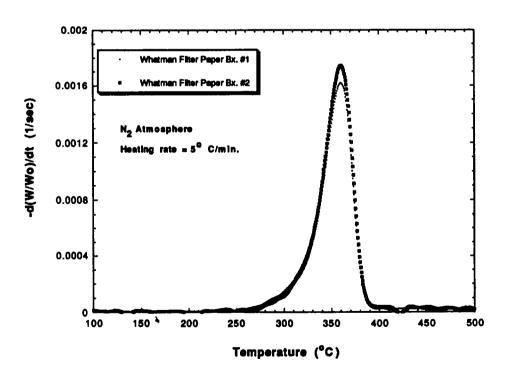


Figure F-6. TG Data for Two Samples of Whatman Filter Paper.

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