

Cigarette Type	Substrate	Laboratory	Test Results		
			Ignitions	Non-Ignitions	Self-Extinguishments
4	1	1	0	0	48
		2	0	0	48
		3	0	0	48
	2	1	0	0	48
		2	0	0	48
		3	0	0	48
	3	1	0	0	48
		2	0	0	48
		3	1	0	47
5	1	1	4	23	21
		2	0	17	31
		3	3	34	11
	2	1	45	1	2
		2	37	0	11
		3	46	0	2
	3	1	47	0	1
		2	45	0	3
		3	47	0	1

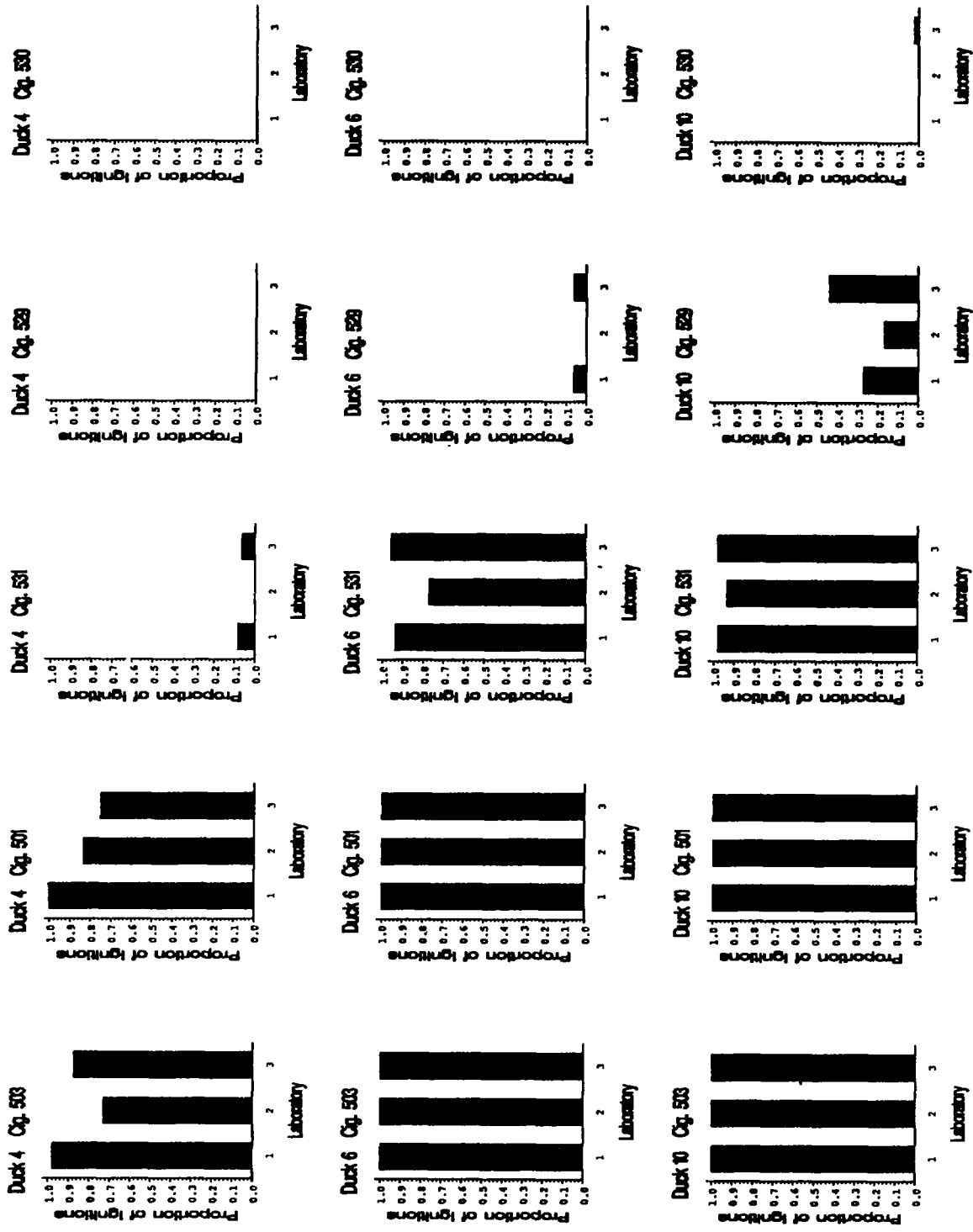


Figure 2. Comparison of Ignition Rates for the Preliminary Interlaboratory Study of the Mock-up Ignition Test Method. The 15 plots in the figure correspond to the 5 cigarette types tested, by columns, and the 3 test substrates, by rows. In each component plot, the vertical bars represent the proportions of ignitions obtained by each of the 3 participating laboratories.

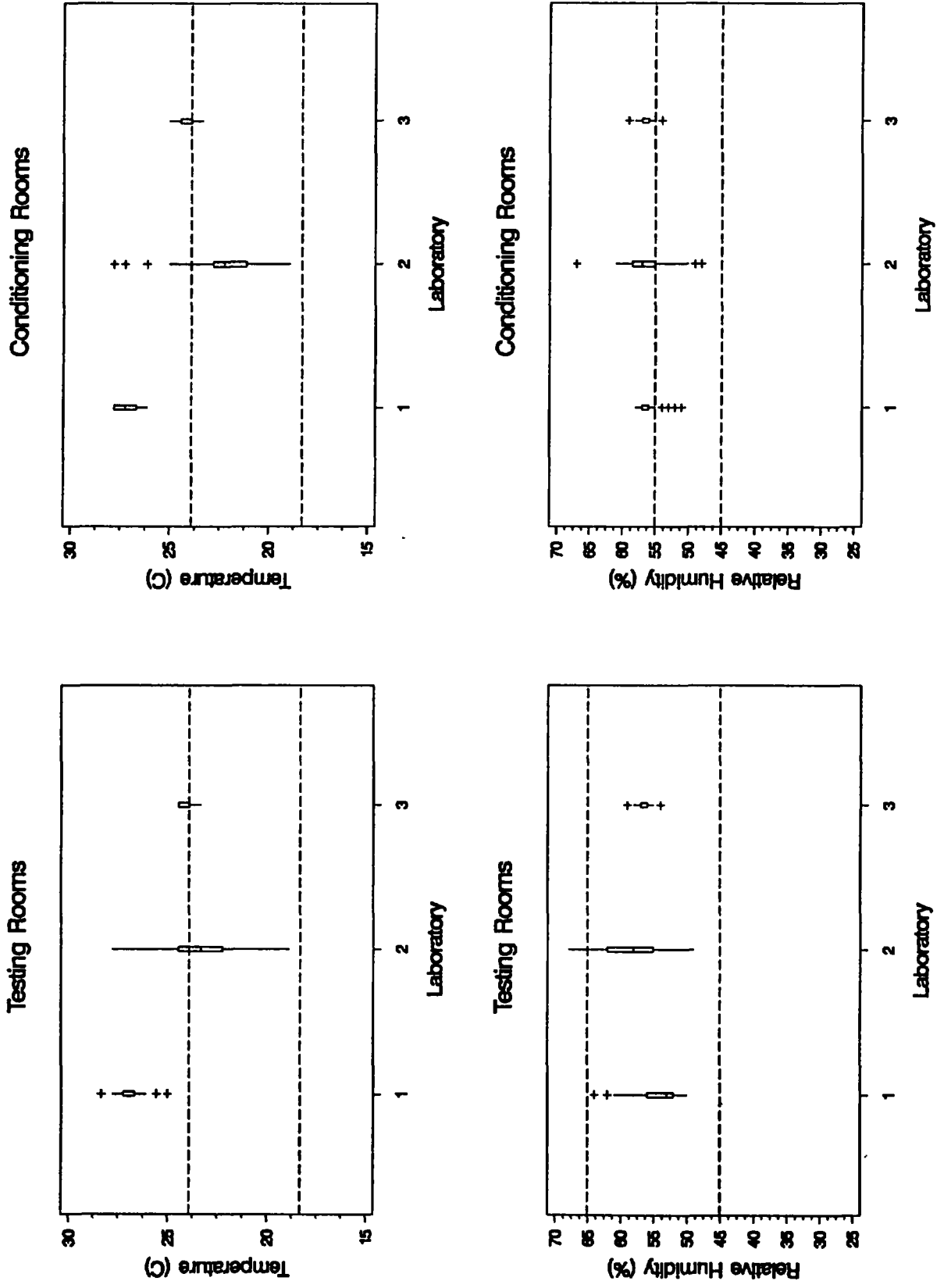


Figure 3. Environmental Conditions Reported by the Laboratories Participating in the Preliminary Interlaboratory Study of the Mock-up Ignition Test Method.

In Figure 3, the distributions of the environmental variables are represented by box plots. Box plots are constructed so that the rectangular boxes contain the range covered by the central 50% of the data, with a line drawn in the interior of the box to represent the median of the data. The "whiskers" (vertical lines) attached to the central 50% boxes extend to the upper and lower limits of all the data, except that values far away from the central portion are identified as "outliers" and are plotted separately with "+" symbols. (The statistical software used to produce these box plots identifies an outlier as any data value whose distance from the median exceeds 1.5 times the length of the box.)

Also indicated in Figure 3, by the space between the horizontal dashed lines, are the target ranges for the environmental variables stated in the instructions for conducting the testing. Comparing the locations of the box plots with the dashed lines shows how successful, or not, the labs were in maintaining the environmental conditions within the target ranges. For example, the plot in the upper left-hand corner of the figure shows clearly that temperature in the testing room of Lab 1 was always a bit higher than the target range. The same plot shows that, while the temperature varied more widely in Lab 2 than in Lab 1, the temperature in Lab 2 was inside the target temperature range approximately two-thirds of the time.

For all three labs, the humidity in the conditioning rooms tended to stay just above 55%, which was the upper limit of the target range. As mentioned in Section II.B.8.e below, this fact influenced the decision to change the target range for humidity in the main interlaboratory study.

A statistical analysis was undertaken to ascertain whether a significant portion of the variation in the ignition results could be attributed to variation in the four environmental variables: TSTTEMP, TSTRH, CNDTEMP, and CNDRH. The statistical procedure used was logistic regression analysis, implemented by the CATMOD procedure in SAS<sup>6</sup>. This analysis proceeded by first fitting a full model in which IGN was modeled as a function of the variables LAB, CIG\_TYPE and SUBSTRAT, plus the four continuous environmental variables. This model was compared with a reduced model in which the environmental variables were omitted. The overall result of this analysis was that *none* of the environmental variables showed a statistically significant effect on ignitions, based on the criterion that the significance probability, or "p-value," was greater than 0.05.

By contrast, the other three variables, LAB, CIG\_TYPE and SUBSTRAT were all statistically significant well below the 0.05 significance level. This result was not unexpected. The five cigarettes were selected for testing to represent a range of ignition behaviors. Similarly, the three substrates were designed to elicit a range of ignition responses. The finding of significant variation between labs, while not particularly desired, is nonetheless a common occurrence in interlaboratory studies of test methods of all kinds. General experience with interlaboratory studies at NIST has been that a statistically significant difference between laboratories can be detected more often than not.

The statistical analysis also investigated whether the ignition results were significantly affected by the four categorical variables: CHAMBER, TST\_BLK, OPERATOR, and AMPM. Both logistic regression and the Cochran-Mantel-Haenszel test procedure were used for this purpose, again implemented by the CATMOD procedure in SAS<sup>6</sup>. The Cochran-Mantel-Haenszel procedure was implemented in a way which tests for a significant association between ignition results (IGN) and each

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<sup>6</sup> SAS<sup>®</sup> statistical software, Version 6. SAS<sup>®</sup> is a registered trademark of SAS Institute Inc., SAS Circle, Box 8000, Cary, NC 27512-8000.

categorical variable while controlling for the effects of LAB, CIG\_TYPE and SUBSTRAT. For example, when applied to the OPERATOR variable, these procedures test whether the difference in ignition results between the experienced and inexperienced operators was greater than would be expected due to random variation. As was the case for the environmental variables, these tests were all nonsignificant, using a significance level criterion of 0.05.

Results from the preliminary ILS showed that the test procedure could be successfully replicated in more than one laboratory. The results from the three laboratories also indicated that the test procedure could be expected to be acceptably repeatable within a laboratory and reproducible between laboratories. The findings from this study pointed the way to the main ILS.

#### **e. Main Interlaboratory Study**

The main interlaboratory study was similar to the preliminary version, but a number of changes were made based on findings from the earlier study. The same factorial experimental design was used, expanded to include nine laboratories.

With this test round, as before, each new laboratory was shipped test chamber kits which they assembled. With this shipment they also received the square brass frames and cigarette holders for each chamber. An operator training program was held in September, 1992 for all participating laboratories. Again, each laboratory was asked to provide an operator experienced with cigarette fire testing and an inexperienced tester. During the training session, each operator acquired hands-on experience with the mock-up ignition method and the cigarette extinction method. Both test methods were included in the training because an ILS for the cigarette extinction method was planned to follow the ILS for the mock-up ignition method.

Based on the satisfactory performance of the test cigarettes and mock-up assemblies in the preliminary round, each was retained for the main ILS. Each of the participating laboratories was shipped approximately 200 cigarettes of each type, as used in the preliminary study. The three sets of mock-up test materials that duplicated those used in the preliminary study were also shipped to each laboratory. However, the original lot of polyethylene film had been depleted. Additional polyethylene film was ordered from the retail supplier, and they inadvertently substituted a nominally similar material, which was included in the shipments to the nine laboratories. The film difference was discussed in Section II.B.3.

An additional 125 cigarettes with a separate designation ("F") were sent to the labs with the other cigarette lots and an extra box of fabric and foam substrate materials was shipped to each laboratory. These additional cigarettes and mock-up materials were provided to give the test operators an opportunity to practice running the test before beginning the main study. The practice tests would only be run after a visit by the interlaboratory study coordinator. Test operators were instructed to use cigarette "F" and the extra box of fabric and foam materials to run twelve practice tests. After completing the practice tests with cigarette "F," each lab would report their results to NIST by FAX.

As was done with the preliminary ILS, the interlaboratory coordinator visited each laboratory to review laboratory environmental conditions and control, check test chamber functioning and provide a relative humidity calibration for the lab. Also the coordinator met with laboratory management and test operators to review the test protocol and to answer last minute questions. All laboratory visits

were completed by the middle of October, 1992. Most laboratories began testing the practice cigarette materials shortly after the coordinator visit was completed. Upon receiving the reports by FAX on the practice round, NIST reviewed the data; and laboratories were given permission to start the main round. All laboratories completed their assigned test work and submitted their data to NIST by the middle of December, 1992.

**Impact of Preliminary Round: Environmental Conditions.** In the preliminary study, the conditioning room and test room environments had different specifications. Conditioning room/chamber relative humidity was to be controlled at  $50 \pm 5$  %. The test room was to be controlled at a level of  $55 \pm 10$  % RH. This difference in environmental conditions appeared to create some confusion within the participating laboratories. Therefore, discussions with the laboratories and a review of test results brought about agreement on a change to the required conditions. When the test procedure was rewritten for the main ILS, it included a single new environmental requirement for both conditioning room and test rooms:  $55 \pm 5$  % RH. This modification of the procedure also allowed the cigarette company labs to operate under conditions normal to their needs, and it also allowed laboratories using test procedures requiring relative humidities of 50% to maintain their normal laboratory conditions.

**Impact of Preliminary Round: Selecting Cigarette Test Weights.** In the preliminary ILS, NIST sampled and weighed each test cigarette lot to establish the range of cigarette weights to be tested. In the main study, this process was transferred to each laboratory. The laboratories were shipped approximately 200 cigarettes of each type and were instructed to follow this same procedure in determining the proper weights of cigarettes to be tested from each lot.

**Impact of Preliminary Round: Cigarette Self-Extinction.** In the preliminary ILS, it was found that certain types of cigarettes had burning characteristics that resulted in self-extinction shortly after they were ignited. Such a cigarette would often go out before being laid onto the mock-up assembly; hence it was discarded and the test was re-run. Even if one of these cigarettes burned long enough to be placed onto the mock-up, it would soon self-extinguish. It became apparent that this portion of the test procedure could be modified to indicate a self-extinction at any point after a cigarette was properly ignited. During the main ILS, it was specified that if a cigarette should self-extinguish at any point after being properly ignited, even if it had not been placed onto the mock-up, the test was complete. Data from such cases were recorded as self-extinctions.

**Participants and Procedures.** Nine laboratories participated in the main interlaboratory study. This group included industry, state and federal government laboratories and an independent testing laboratory. The list of participants follows:

- American Tobacco Company, Research Laboratory
- Brown & Williamson Tobacco Company, Research Laboratory
- Bureau of Home Furnishings, State of California
- Consumer Product Safety Commission, Engineering Laboratory
- Diversified Testing Company
- Lorillard, Research Laboratory
- National Institute of Standards and Technology, Building and Fire Research Laboratory
- Philip Morris USA, Research Laboratory
- R.J. Reynolds Tobacco Company, Research Laboratory

[Note that this listing does not correspond to the identification numbers, 1 through 9, which appear later in the report. Those numbers were assigned randomly for anonymous presentation of the results.]

As stated above, several changes were made to the test procedure based on experiences gained from the preliminary ILS. The preliminary study also indicated how the recording and reporting of data could be streamlined, and these changes were made in the main study. Each tester in the program received a booklet containing copies of the test procedure and other ILS guidelines. They also received three separate workbooks for recording data, one for each week of the three-week test program. Each laboratory received three computer disks for putting the test results into computer-readable form. The following instructions were given on new procedures for recording and submitting data to NIST for analysis: Test operators were directed to record all data in their workbooks as they prepared and completed each test. At the end of each day they were to enter their data into the computer, which would then print out a daily summary for each operator. These summary sheets were to be FAXed to NIST at the end of each day. At the end of each week, the weekly workbooks and the weekly computer disks were to be mailed to NIST by two-day delivery. This method of acquiring data helped to reduce the time needed for assembling the test data and preparing it for analysis.

**Purpose and Methods of Analyzing Results.** Information provided in the daily FAX report from the laboratories was used by NIST to evaluate laboratory progress. It also provided information necessary for preparing data files for each laboratory. As the ILS progressed, not all of the laboratories consistently submitted the daily FAX reports. Some labs would accumulate several days of testing before FAXing the reports to NIST. As the test results were submitted to NIST, they were organized into appropriate data files used for review of accuracy and for analysis. These files were compared to the data recorded in the test workbooks, and corrections were made as needed. Again with this ILS, there were missing data in the workbooks and incorrect units reported. The computer files again showed typos, transposed numbers and mixed units. There were 6,480 test results returned from the main ILS. Of these data, approximately five percent contained errors of the types discussed above. One operator in each of two laboratories failed to enter all the ignition data in the lab workbooks; however, all data were entered into the computer files. Less than 0.3 percent of the ignition data were missing from the workbooks.

A few cases were noted in which laboratories ran some of the test replications on the wrong mock-up configuration, compared to what was called-for in the test plan. The result was that the number of tests actually conducted differed from the intended number (48 replications) by  $\pm 2$ , for some combinations of cigarette type and mock-up configuration. These cases are reflected in rows of Table 22 for which the total number of Test Results reported (Ignitions + Non-Ignitions + Self-Extinguishments) sums to 46 or 50, instead of 48. In all cases, the total number of tests performed by the laboratory was correct.

**Raw Data.** As noted, the combined computer file contains 6480 lines of data, corresponding to 720 ignition results per laboratory for each of 9 laboratories. The 720 results per laboratory arise from 48 tests of each of 5 cigarettes on each of 3 substrates ( $720 = 48 \times 5 \times 3$ ).

The raw data on each line of the computer file represent the same set of thirteen variables described previously in Table 20. There are two minor changes in the data for these variables compared to their descriptions in Table 20: the LAB variable has a range from 1 to 9 in the main round, rather than 1 to 3, as in the preliminary round; and the variable OPERATOR no longer differentiates one of the operators as "Experienced" and the other as "Inexperienced," but rather codes the two operators simply as number "1" and number "2," in no particular order. This change was made because it was impractical, and seemed unnecessary, to recruit truly inexperienced operators at each of the nine laboratories for the main round evaluation. Generally, both operators at each lab had some previous experience in testing ignition propensities of cigarettes or had sufficient acclimation after the first several days of testing to be regarded as experienced.

A summary of the test results, by LAB, CIG\_TYPE and SUBSTRAT, is presented in Table 22. Note that identifying numbers were assigned *independently* to the laboratories in the preliminary and in the main rounds of the interlaboratory evaluation so, for example, laboratory number 3 in the preliminary round is not the same as number 3 in the main round. In Table 22, the ignition results are recorded in three categories, as given by the variable TST\_RSLT. As was done for the preliminary round, the two types of non-ignitions were combined into a single category for the statistical analyses. Thus, as before, the test results were analyzed in terms of the derived variable IGN, which simply records the results as ignition (IGN=Y) or non-ignition (IGN=N).

A graphical display of the data in Table 22 is shown in Figure 4, where, for each cigarette (by columns) and substrate (by rows) the proportion of ignitions is represented by a vertical bar for each laboratory. The cigarette types are shown from left to right in order of decreasing ignition propensity, with cigarette 503 having the most ignitions (in the left-most column of plots) and cigarette 530 having the fewest ignitions (in the right-most column). The three mockup configurations are shown as rows in the figure, with the least ignitable substrate (duck #4) as the top row and the most ignitable (duck #10) as the bottom row of the figure. For several cigarette and substrate combinations, all laboratories showed either 100% ignitions (charts near the lower left corner of the figure) or 0% ignitions (those near the upper right corner). Cases with intermediate ignition percentages fall near the diagonal (upper left to lower right) in the figure.



**Table 22. Summary of Test Results for Main Interlaboratory Study, Mock-Up Ignition Method**

Cigarette Type	Substrate	Laboratory	Test Results		
			Ignitions	Non-Ignitions	Self-Extinguishments
501	1	1	2	46	0
		2	5	43	0
		3	1	47	0
		4	3	45	0
		5	1	47	0
		6	3	45	0
		7	6	42	0
		8	16	34	0
		9	11	37	0
	2	1	48	0	0
		2	48	0	0
		3	48	0	0
		4	48	0	0
		5	48	0	0
		6	48	0	0
		7	48	0	0
		8	46	0	0
		9	48	0	0
	3	1	48	0	0
		2	48	0	0
		3	48	0	0
		4	48	0	0
		5	48	0	0
		6	48	0	0
		7	48	0	0
		8	48	0	0
		9	48	0	0

Cigarette Type	Substrate	Laboratory	Test Results		
			Ignitions	Non-Ignitions	Self-Extinguishments
503	1	1	20	28	0
		2	26	22	0
		3	19	29	0
		4	33	15	0
		5	22	26	0
		6	19	27	2
		7	21	27	0
		8	34	14	0
		9	36	12	0
	2	1	48	0	0
		2	48	0	0
		3	46	0	0
		4	48	0	0
		5	48	0	0
		6	48	0	0
		7	48	0	0
		8	46	0	0
		9	48	0	0
	3	1	48	0	0
		2	48	0	0
		3	50	0	0
		4	48	0	0
		5	48	0	0
		6	48	0	0
		7	48	0	0
		8	50	0	0
		9	48	0	0

Cigarette Type	Substrate	Laboratory	Test Results		
			Ignitions	Non-Ignitions	Self-Extinguishments
529	1	1	0	0	48
		2	0	0	48
		3	0	0	48
		4	0	0	48
		5	0	0	48
		6	0	0	48
		7	0	0	48
		8	0	0	48
		9	0	0	48
	2	1	1	0	47
		2	3	1	44
		3	6	0	42
		4	3	0	45
		5	0	0	48
		6	3	1	44
		7	0	0	48
		8	6	0	42
		9	11	0	37
	3	1	10	0	38
		2	15	0	33
		3	18	0	30
		4	13	0	35
		5	5	0	43
		6	14	0	34
		7	12	0	36
		8	20	0	28
		9	24	0	24

Cigarette Type	Substrate	Laboratory	Test Results		
			Ignitions	Non-Ignitions	Self-Extinguishments
530	1	1	0	0	48
		2	0	0	48
		3	0	0	48
		4	0	0	48
		5	0	0	48
		6	0	0	48
		7	0	0	48
		8	0	0	48
		9	0	0	48
	2	1	0	0	48
		2	0	0	48
		3	0	0	48
		4	0	0	48
		5	0	0	48
		6	0	0	48
		7	0	0	48
		8	0	0	48
		9	1	0	47
	3	1	0	0	48
		2	0	0	48
		3	1	0	47
		4	0	0	48
		5	0	0	48
		6	0	0	48
		7	1	0	47
		8	3	0	45
		9	6	0	42

Cigarette Type	Substrate	Laboratory	Test Results		
			Ignitions	Non-Ignitions	Self-Extinguishments
531	1	1	0	20	28
		2	0	22	26
		3	0	27	21
		4	0	28	20
		5	0	25	23
		6	0	30	18
		7	0	24	24
		8	0	28	20
		9	0	31	17
	2	1	44	0	4
		2	42	0	6
		3	48	0	0
		4	47	0	1
		5	46	0	2
		6	48	0	0
		7	46	0	2
		8	44	0	4
		9	45	0	3
	3	1	48	0	0
		2	46	0	2
		3	47	0	1
		4	47	0	1
		5	45	0	3
		6	48	0	0
		7	47	0	1
		8	48	0	0
		9	47	0	1

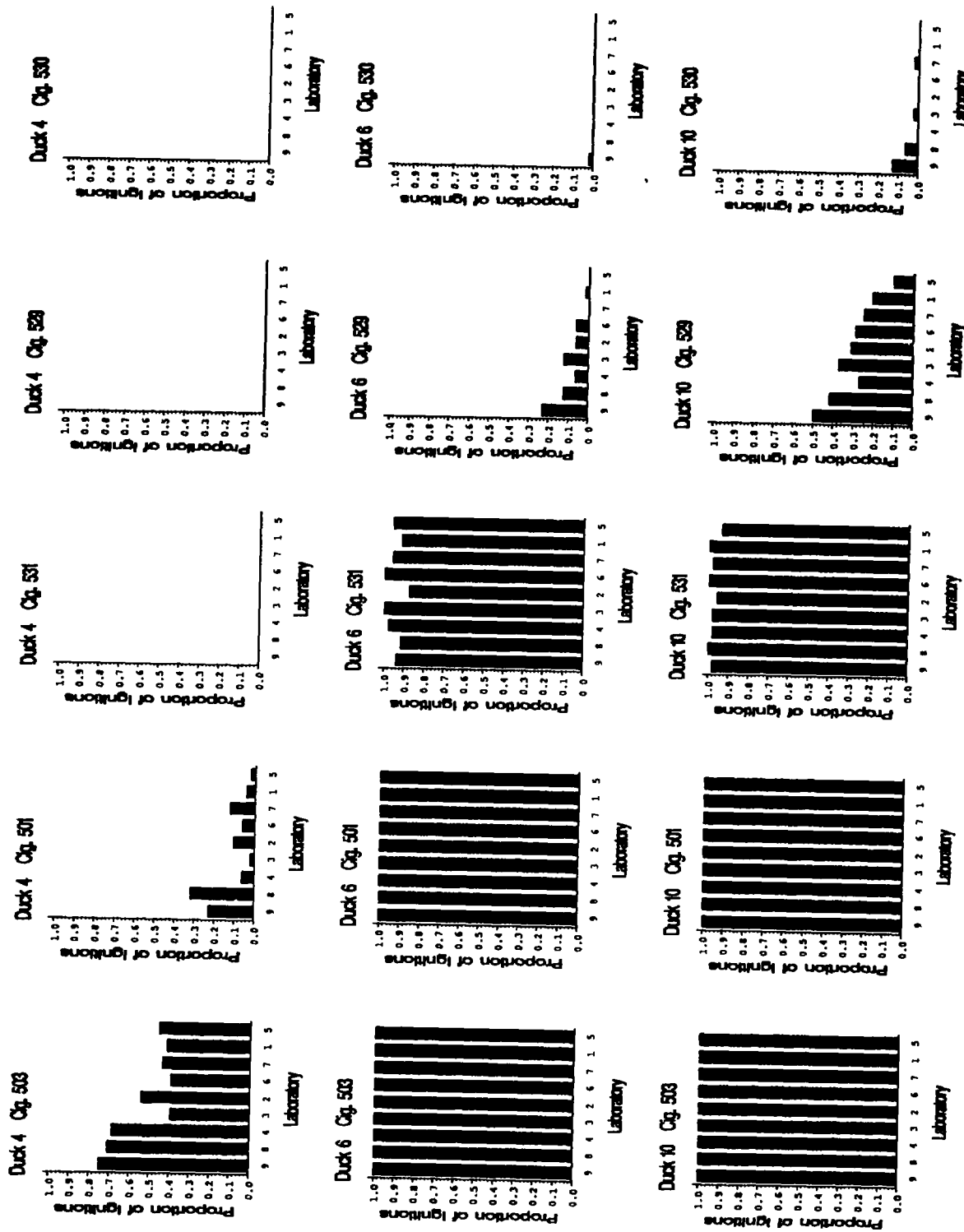


Figure 4. Comparison of Ignition Rates for the Main Interlaboratory Study of the Mock-up Ignition Test Method.

**Auxiliary Variables.** A statistical analysis was undertaken to detect possible dependence of the ignition results, through the variable IGN, on the eight auxiliary variables described in Table 20. It was not the purpose of this analysis to develop a detailed understanding of such dependencies from the interlaboratory study data — a designed experiment in a single laboratory would be better for that purpose. Rather, the purpose of the analysis of the auxiliary variables was to reveal features of the data that might point to any major problems with the execution or performance of the method in different laboratories.

The ranges of environmental conditions experienced by the laboratories are shown in Figure 5, using box plots to represent the range of data from each laboratory. Details of the interpretation of box plots are described later in this Section. The figure shows that most labs had at least a few temperature and/or humidity readings outside the target limits. Except for the temperatures in lab 8, the labs generally had most of their readings within the prescribed limits, or very nearly so.

Various statistical techniques were applied to investigate whether the ignition results were strongly influenced by the four environmental variables or by the discrete auxiliary variables, CHAMBER, TST\_BLK, OPERATOR and AMPM. The Cochran-Mantel-Haenszel test was again used for the discrete variables. Conclusions from this test procedure showed no significant relation of IGN with CHAMBER or TST\_BLK (*i.e.*, "week"), but did indicate the existence of possible effects due to both OPERATOR and AMPM.

In the case of OPERATOR, a statistically significant difference between the two operators was shown only for lab 4. In lab 4, the two operators obtained the same number of ignitions for 9 of the 15 cigarette/substrate combinations, but in each of the remaining six cases, the number of ignitions obtained by Operator 1 was always greater than the number obtained by Operator 2. The difference between the two operators was not large: Operator 1 obtained a total of just 12 more ignitions than Operator 2, out of 360 tests by each operator. It was mainly the consistency of the difference across the six cases that led to the attainment of statistical significance. In this study no attempt has been made to determine whether this observed difference can be traced to any differences in test procedure used by these two operators.

The variable AMPM, which indicates whether a test was run before or after noon, also showed a statistically significant, but small, effect. Overall, there was a slightly higher percentage of ignitions in the PM (47.4%) compared to the AM (45.7%). The Cochran-Mantel-Haenszel procedure, which identified this effect, is sensitive to both the size of the AM-PM difference and the consistency of its direction, while controlling for the LAB, CIG\_TYPE and SUBSTRAT variables. While no satisfactory physical explanation was found for this overall effect, the following illustrates the kind of minor ambiguities that remain in the data. In a sub-analysis using data from lab 6 only, the increased ignition rate in the PM was also associated with an increase (of about 1 °C) in the average temperature of the test room (TSTTEMP). It is not possible with the information at hand, however, to establish a *causal* link between the two increases.

An attempt was made to perform a global analysis of the effects of all the study variables through the use of a detailed logistic regression model. This model-fitting exercise was not completely successful because of the large number of cells in the data matrix where the percentage of ignitions was either 0% or 100%. This feature of the data leads to a requirement of "infinite" parameter values in the model, with the additional result that the significance probabilities computed by the statistical software are not reliable, and therefore may not be worth pursuing.

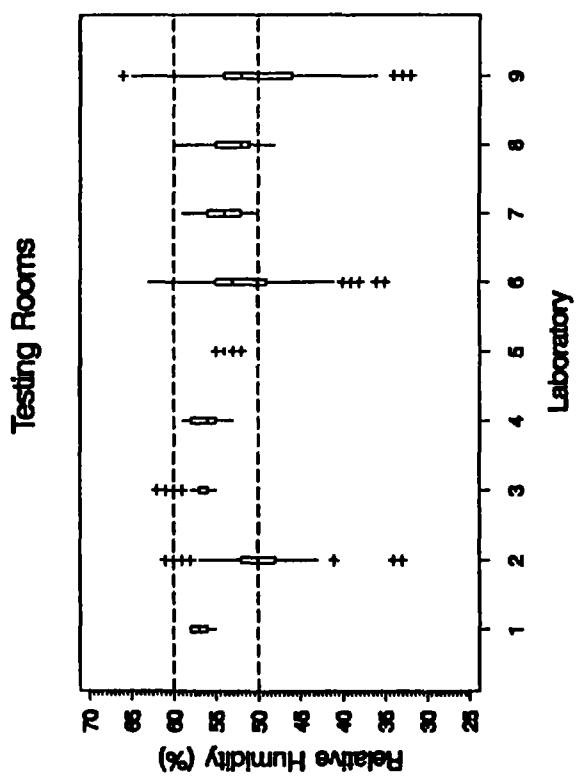
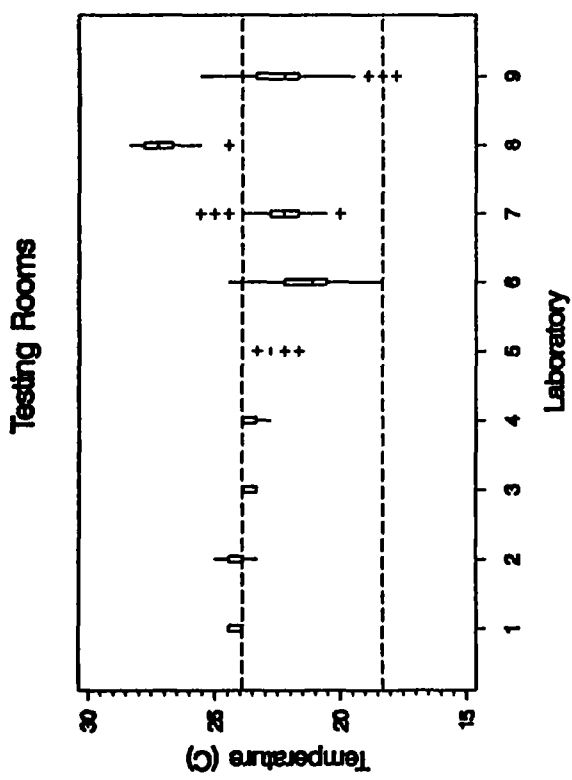
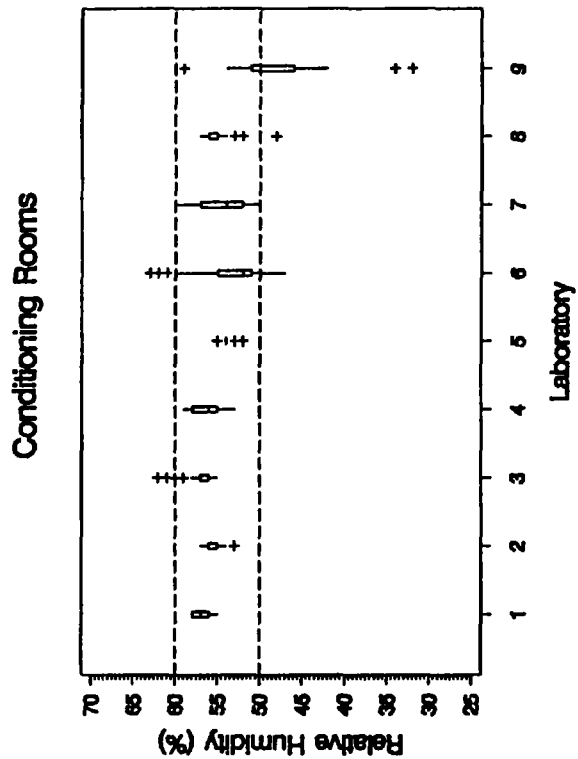
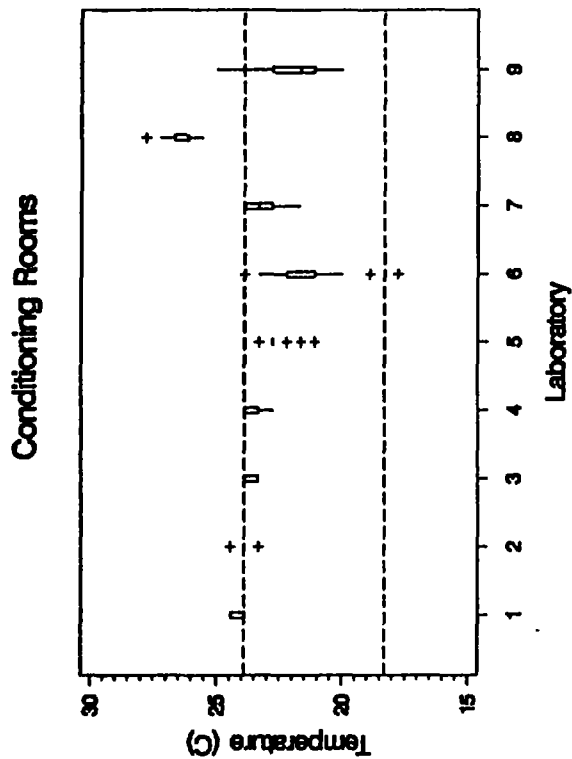


Figure 5. Environmental Conditions Reported by the Laboratories Participating in the Main Interlaboratory Study of the Mock-up Ignition Test Method.



A readily interpretable result from the global analysis pertains to the environmental variables. In the global model, the logarithm of the odds ratio in favor of ignition (IGN) was modeled as a function of all the variables (except DATE) in Table 20, together with several interactions. In this fitted model, the regression coefficients for all four environmental variables had the expected signs (though none had a statistically significant magnitude): the two temperature coefficients (for TSTTEMP and CNDTEMP) were positive, implying that increasing temperature increases the odds in favor of ignition, and the humidity coefficients (for TSTRH and CNDRH) were both negative, implying that increased humidity decreases the odds in favor of ignition.

Overall, the logistic analyses showed some indications of small, but possibly real, dependencies between the auxiliary variables and the ignition results. However, they did not reveal any major problems in the data. These indications are consistent with the general observation there will always be some means by which the test results could be improved by further refinements to the test method protocols.

Primary Variables. It is possible, and of some interest, to test whether the three primary explanatory variables in the data set, namely LAB, CIG\_TYPE and SUBSTRAT, show statistically significant effects on the ignition results. In short, the IGN variable shows statistically significant effects due to all three of the primary explanatory variables.

For the LAB variable, statistically significant differences between labs were shown for essentially all cigarette/substrate combinations where the percentage of ignitions was far enough away from both 0% and 100% to allow differences to show. Specifically, significant between-laboratory differences were identified for cigarette types 503 and 501 on the duck #4 mock-up, for cigarette 529 on the duck #6 mock-up, and for cigarettes 529 and 530 on the duck #10 mock-up. These individual tests, for each cigarette and substrate, were combined into an overall test by the Cochran-Mantel-Haenszel procedure with the result that the overall hypothesis that there is no between-laboratory variation is rejected. The magnitude of the between-lab variability is summarized below through the estimation of repeatability and reproducibility standard deviations.

The significance test for differences between cigarette types was also carried out using the Cochran-Mantel-Haenszel procedure. The results showed that all five cigarette types have ignition rates that are statistically different from each other, with p-values less than 0.001 in each case. Turning to substrates, the Cochran-Mantel-Haenszel procedure also showed significant differences between the ignition results across all three mock-ups.

Repeatability and Reproducibility. In ASTM standard E-691, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method" [25], the summary precision statement for a test method under evaluation is based on statistical calculations of repeatability and reproducibility limits for the method. Recall that repeatability refers here to the consistency of test results from a single laboratory; reproducibility refers to the consistency of results among different laboratories. The calculation formulas described in ASTM E-691 are appropriate for test methods that yield measurements on a continuous scale, rather than the categorical "yes/no" outcomes that characterize the IGN variable of this study, and cigarette ignition testing generally. However, the repeatability and reproducibility measures can be adapted to categorical data applications, as will be described.

In ASTM E 691, the repeatability standard deviation is defined as the best estimate of the within-laboratory standard deviation of single measurement results. At the present stage of development, the definition of what constitutes a "single" measurement result for the Mockup Ignition Test Method has not been specified, except that it is understood that a single measurement result is the proportion of ignitions in some number (to be specified) of replications of the operation of placing a single type of lighted cigarette on a single type of mockup. For a cigarette having ignition rate  $P$  (a number between 0 and 1) on a given substrate, the standard deviation of the observed proportion of ignitions in  $m$  replications is equal to  $[P(1-P)/m]^{1/2}$ , based on standard properties of the binomial distribution. Some mathematical simplicity will be gained in what follows by initially taking  $m$  to be equal to the total number of replications actually conducted by each laboratory;  $m = 48$  in the case of the mock-up ignition method. Further calculations using a simple statistical model will then allow comparisons of repeatability and reproducibility values that could be expected assuming different values of  $m$ .

Applying this approach to data for any substrate and cigarette type, the best (quasi-likelihood) estimate [26] of the *repeatability* standard deviation,  $S_r$ , is

$$S_r = \sqrt{\frac{\bar{p}(1-\bar{p})}{48}}$$

where  $\bar{p}$  represents the mean proportion of ignitions across the nine laboratories and  $m$  has been set equal to 48. As defined,  $S_r$  is the best estimate of the pooled within-laboratory standard deviation, in that it combines information across all laboratories.

Using the convention that a single measurement result is defined as the proportion of ignitions in  $m = 48$  replications, the number of single measurement results per laboratory is 1 in this study, and so the *reproducibility* standard deviation,  $S_R$ , as defined in ASTM E 691, is simply the between-laboratory standard deviation,

$$S_R = \sqrt{\frac{\sum_{i=1}^9 (p_i - \bar{p})^2}{8}}$$

where  $p_i$  represents the proportion of ignitions for laboratory  $i$  and  $i = 1, 2, \dots, 9$ .

The repeatability and reproducibility standard deviations for the main round interlaboratory evaluation are summarized in Table 23.

**Table 23. Observed Repeatability and Reproducibility Standard Deviations for Mock-Up Ignition Method**  
**Main Interlaboratory Study;  $m=48$  Replications per Laboratory**

Cigarette I.D.	Substrate	Average Proportion of Ignitions	Repeatability S.D. $S_r$	Reproducibility S.D. $S_R$
501	1	0.110	0.045	0.102
	2	1.000	0	0
	3	1.000	0	0
503	1	0.532	0.072	0.145
	2	1.000	0	0
	3	1.000	0	0
529	1	0.000	0	0
	2	0.076	0.038	0.074
	3	0.303	0.066	0.117
530	1	0.000	0	0
	2	0.002	0.007	0.007
	3	0.025	0.023	0.043
531	1	0.000	0	0
	2	0.949	0.032	0.042
	3	0.979	0.021	0.021

As previously mentioned, the repeatability standard deviation is a function of the average proportion of ignitions,  $\bar{p}$ , for each cigarette type. In Table 23, it is apparent that the reproducibility standard deviation also depends on  $\bar{p}$  to some extent. In order to provide a succinct summary of the data, it of interest to combine the estimates of repeatability and reproducibility across the substrates and cigarette types in this study.

ASTM Standard E 691 [25], in section 21.3, *Variation of Precision Statistics with Property Level*, gives the following general guidance in formulating precision statements for test methods where the repeatability and reproducibility depend on the level of the property being measured.

"Quite often the values of  $S_r$  and  $S_R$  will be found to vary with the values of the property level,  $\bar{x}$  [corresponding to our  $\bar{p}$ ].... The manner in which the statistics vary with the property level should be shown in presenting the precision information in the

precision statement of the test method. The statistician should recommend the most appropriate relationship to present, using Practice E 177 as a guide."

ASTM E 177 [24], in section 28.5.4, recommends summarizing the relationship of the method precision to the property level "by a simple formula, or by a plot." For repeatability, the definition above specifies  $S_r$  as a simple function of the property level,  $\bar{p}$ . This relationship can be extended to accommodate the reproducibility standard deviation by use of a simple statistical model for what is variously called "extra binomial variation" [27] or "over dispersion" [26] or simply "heterogeneity" [28].

In this model, the between-laboratory variance, or reproducibility variance  $S_R^2$ , is related to the within-laboratory variance, or repeatability variance  $S_r^2$ , as follows:

$$\begin{aligned} S_R^2 &= \frac{p(1-p)}{m} [1 + \varphi(m-1)] \\ &= S_r^2 [1 + \varphi(m-1)] \end{aligned}$$

The quantity  $[1 + \varphi(m-1)]$  in the above expression is called the "heterogeneity factor" in Finney's classic text on probit analysis [28]. The parameter  $\varphi$  in the heterogeneity factor plays the role of a correlation coefficient among replications performed within the same laboratory.

This model suggests that  $S_R^2$  should vary roughly as a constant multiple of  $S_r^2$ . Figure 6 shows this approximate relationship for the data in Table 23, along with a least squares straight line through the origin given by

$$S_R^2 = (3.72)S_r^2.$$

Figure 6 shows that the simple statistical model gives a satisfactory summary of the relationship of  $S_R^2$  to  $S_r^2$  and, therefore, to  $\bar{p}$ . Identifying the slope of the least squares line in the Figure, 3.72, with the heterogeneity factor,  $[1 + \varphi(48-1)]$ , yields the estimated value  $\varphi = 0.058$ .

In terms of this model, then, the repeatability and reproducibility standard deviations can be approximated for any case of interest by using the formulas

$$S_r = \sqrt{\frac{p(1-p)}{m}} \quad (1)$$

$$S_R = \sqrt{\frac{p(1-p)}{m}[1 + \varphi(m-1)]} ,$$

where  $p$  is the assumed ignition rate,  $m$  is the assumed number of replications per test result, and  $\varphi$  is the parameter for the heterogeneity factor, as estimated from relevant data ( $\varphi = 0.058$  for the Mock-Up Ignition Test Method ILS.)

In Table 24, these formulas are used to calculate "repeatability limits," defined as  $2.8 \times S_r$ , and "reproducibility limits," defined as  $2.8 \times S_R$ , for the mock-up ignition method. The factor "2.8" in the definition of the repeatability and reproducibility limits is recommended in ASTM E 691 as a means to generate approximate 95% probability limits for the possible difference between two measurement results (*i.e.*, proportions based on  $m$  replications) obtained within the same laboratory (repeatability limit =  $2.8 \times S_r$ ) or in different laboratories (reproducibility limit =  $2.8 \times S_R$ ). For example, from Table 24 the reproducibility limit calculated as 0.39 for  $m = 48$  runs means that, if the proportion of ignitions is obtained for  $m = 48$  runs on the same cigarette/mock-up combination in each of two laboratories, then one might expect that the difference between the two proportions will be less than about 0.39 if the average cigarette ignition rate is near  $p = 0.5$ .

In interpreting the values in Table 24, the reader should bear in mind the statements in ASTM E 691 [25] that repeatability and reproducibility limits "should be considered as useful general guides," but "not exact mathematical quantities which are applicable to all circumstances and uses."

Table 24 allows one to compare the repeatability and reproducibility limits corresponding to several values of  $m$ , the assumed number of replications per "single measurement result." It is clear from the table, and from the formulas, that  $S_r$  is more strongly affected by increasing the number of replications than is  $S_R$ . This fact is highlighted in Table 24 by inclusion of the case where  $m = 9600$  runs is assumed. In general, the repeatability decreases as the square root of  $m$  whereas the reproducibility approaches a non-zero limit for large  $m$ , which reflects the between-lab component of variability. This behavior shows the limitation to how much the reproducibility precision can be improved by increasing the number of replications within each laboratory.

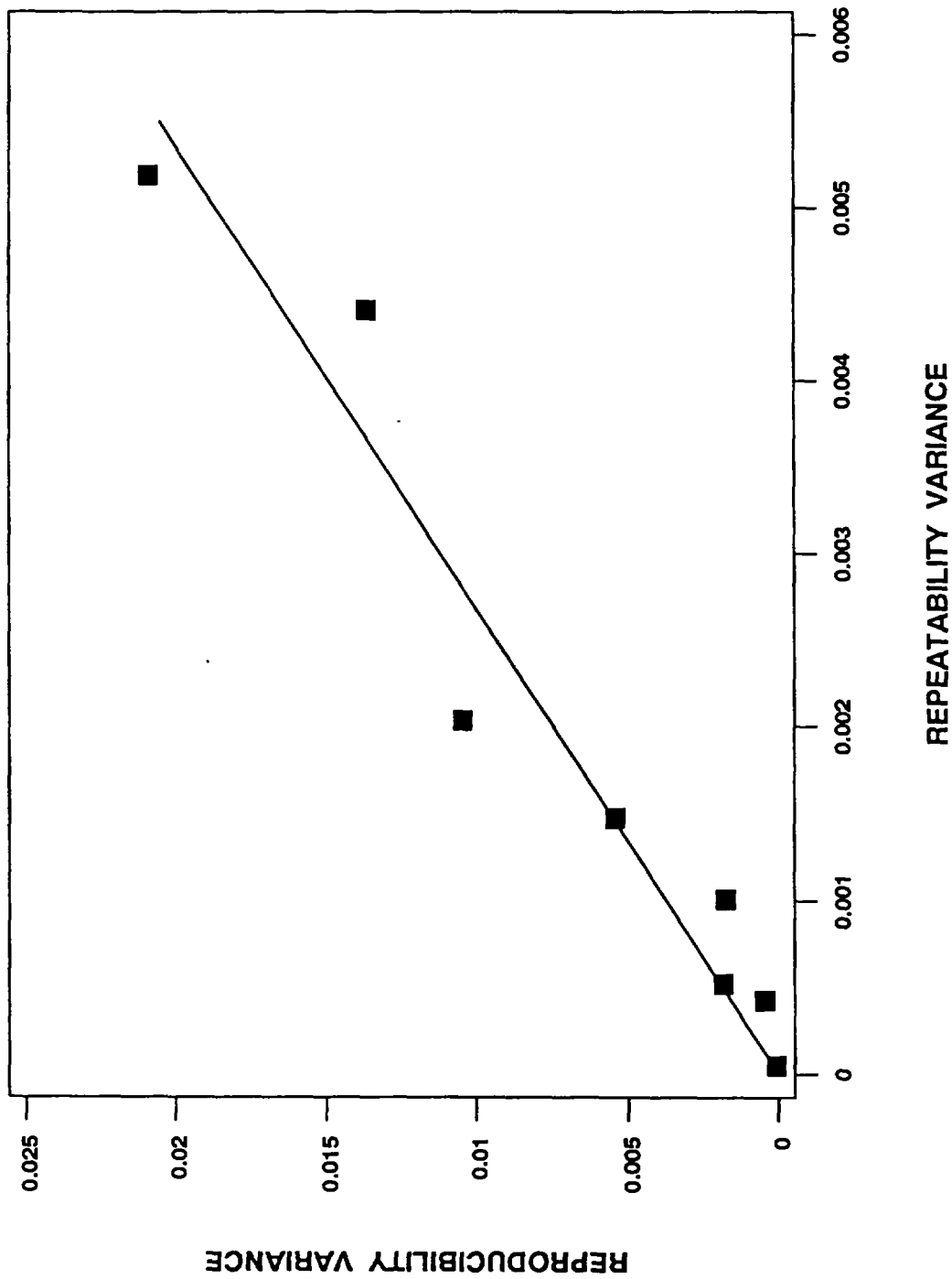


Figure 6. Empirical Relation of Reproducibility Variance,  $S_R^2$ , to Repeatability Variance,  $S_r^2$ , Based on Data in Table 23 from the Main Interlaboratory Study of the Mock-up Ignition Test Method. The equation of the least squares line shown is  $S_R^2 = (3.72)S_r^2$ .

**Table 24. Mock-Up Ignition Method:  
Calculated Repeatability (*r*) and Reproducibility (*R*) Limits  
for Various Assumed Numbers of Replications (*m*) and Ignition Propensities (*p*)**

<b>p</b>	<b>m = 16</b>		<b>m = 32</b>		<b>m = 48</b>		<b>m = 96</b>		<b>m = 9600</b>	
	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>
0.05 or 0.95	0.15	0.21	0.11	0.18	0.09	0.17	0.06	0.16	0.006	0.15
0.10 or 0.90	0.21	0.29	0.15	0.25	0.12	0.23	0.09	0.22	0.009	0.20
0.20 or 0.80	0.28	0.38	0.20	0.33	0.16	0.31	0.11	0.29	0.011	0.27
0.30 or 0.70	0.32	0.44	0.23	0.38	0.19	0.36	0.13	0.33	0.013	0.31
0.40 or 0.60	0.34	0.47	0.24	0.41	0.20	0.38	0.14	0.36	0.014	0.33
0.50	0.35	0.48	0.25	0.41	0.20	0.39	0.14	0.36	0.014	0.34

**m:** Assumed number of replications per laboratory  
**p:** Assumed long-run proportion of ignitions for cigarette and substrate combination under test  
**r:** Repeatability limit =  $2.8S_r$ , where  $S_r$  is calculated from Equation (1)  
**R:** Reproducibility limit =  $2.8S_R$ , where  $S_R$  is calculated as from Equation (1), with  $\varphi = 0.058$ .

Results from the main ILS show that the mock-up ignition test method can effectively differentiate the ignition propensities of various cigarettes, albeit at a limited degree of resolution. The most important limiting factor affecting the resolution of the test method is measured by the reproducibility limits. In Table 24, the repeatability limits (*r*) summarize the precision of the test method under the most favorable conditions for obtaining low variation (same lab, same equipment, short time period, same operators, etc.), whereas the reproducibility limits (*R*) measure the long-term stability of the test method.

The repeatability limits calculated in Table 24 represent the theoretical minimum amount of statistical variability that is inherent in data recorded as binary outcomes (ignitions vs non-ignitions). The amount by which the reproducibility exceeds the repeatability of this, or any, test method measures the degree to which unknown or uncontrolled influence factors affect the test results in the long

term. Data from the nine laboratories in the ILS show that the ratio of repeatability to reproducibility limits is  $R/r = S_R/S_r = \sqrt{3.72} = 1.9$  for the mock-up ignition method. See Figure 6.

This ratio is comparable to the  $R/r$  ratio for other fire test methods currently being used to regulate materials which may be involved in unwanted fires. For example, ASTM E648 (Standard Method for Critical Radiant Flux of Floor Covering Systems) has an  $R/r$  ratio of 1.1 to 1.6 [29]; ASTM E662 (Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials) has  $R/r$  ranging from 1.2 to 4.0 [30]; and ASTM E1354 (Standard Test Method for Heat and Visible Smoke Release for Materials and Products Using an Oxygen Consumption Calorimeter) has  $R/r = 1.8$  for ignition delay time [31].

### C. Cigarette Extinction Test Method

As previously mentioned, an ignition propensity test method need not directly simulate the upholstery material ignition process. Many flammability tests are imperfect representations of the hazard under consideration. This is because full simulation of the fire of concern is often not possible at bench scale, is too costly, or is otherwise impractical. Thus, a cigarette ignition propensity test method could measure, *e.g.*, heat release rate, were it shown to correlate with real-world ignition performance. Such a method can be useful in practice, at least over the range of cigarette designs for which it has been calibrated; and it may also be more convenient to apply.

As can be inferred from the discussion in Section II.B, the substrate requirements for a cigarette ignition propensity test method may be more readily met on a long-term basis if upholstery materials are avoided. This prompted the pursuit of alternative methods in this study. This section of the report describes the work performed in developing such a test method for the measure of cigarette ignition propensity.

#### 1. Prior Alternative Methods

The search for a method for the evaluation of cigarette ignition propensity that is free of upholstery materials has been ongoing intermittently for over ten years. In 1981, Krasny *et al.* [32] reported a series of experiments that ultimately led to the development of a test method that employed alpha cellulose paper as a surrogate substrate. As possible indicators of cigarette ignition potential, they compared four measures of cigarette behavior to mock-up test results obtained for the same cigarettes on a variety of upholstered furniture substrates. These measures were:

- static burning rate of the cigarettes,
- surface temperature of the cigarette burn cone,
- burning behavior of the cigarettes in contact with heat sinks, and
- burning behavior of the cigarettes on alpha cellulose paper.

They concluded that weight loss rate from the cigarette/paper system was a good measure of cigarette ignition propensity, while there were shortcomings with the other three measures. Thirty commercially available cigarettes were evaluated by this test method. Reasonable agreement was found between cigarette propensity to ignite upholstered furniture substrates and weight loss rate of the cigarette/paper system. Subsequent work that was part of the TSG study [3, 3] with low ignition



propensity cigarettes showed that the alpha cellulose paper would not smolder, and the cigarettes would all self-extinguish. This resulted in no recorded weight loss and, therefore, no discrimination between cigarettes.

By 1985, Norman [33] had investigated several methods for assessing cigarette ignition propensity. He used four experimental cigarettes and measured:

- the heat transfer rate to a receiver below the cigarette coal and the total heat release of a cigarette smoldering in air,
- the weight loss rate of various cigarette/substrate systems, and
- the imprint of a cigarette smoldering on a block of polyurethane foam.

While Norman could not correlate free-burn heat transfer data for cigarettes burning in air to ignition propensity and the weight loss rate was dependent on specific characteristics of the substrate, the foam imprint method appeared to hold some promise. Gann *et al.* [3] further pursued this latter method. Rather than measure the volume of the imprint, they measured the weight loss of the foam block after removal of the charred remains of the cigarette. They also recorded weight loss vs. time of the cigarette/foam system during the cigarette smoldering process. They found only a weak correlation between weight loss and cigarette ignition propensity as measured by the number of ignitions on a selected group of fabric/foam substrates.

Gann *et al.* [3] also investigated the possibility of using a heated glass plate to characterize cigarette ignition propensity. By adjusting the temperature of the glass plate, they found that cigarettes could be made to smolder their entire length. Commercially available cigarettes smoldered their entire length at ambient conditions. Low and moderate ignition propensity cigarettes would smolder their entire length only when the temperature of the glass plate was raised to between 86 and 97 °C. As with the case of the alpha cellulose paper, they noted that "No difference between the low and moderate cigarettes was evident."

## 2. Approaches Examined in This Study

It has been previously shown [3] that cigarettes with a wide range of ignition propensities are possible. Thus, a useful test method needs to be able to discriminate over this wide potential. Mock-up-based test methods accomplish this by using a range of substrate assemblies. The alternative methods investigated in this study accomplish this by either changing surrogate substrate characteristics or by measuring a critical cigarette property. The search for an acceptable indirect test method involved the use of various non-reactive and reactive substrates.

The Series 100 cigarettes were used in these experiments. Recall that they were effectively calibrated as to ignition propensity on both mock-ups and full-scale chairs in the TSG study. Thus they can serve here to calibrate candidate alternative test methods. The properties of these cigarettes and their ignition performance seen in the TSG study are listed in Section II.A.1.

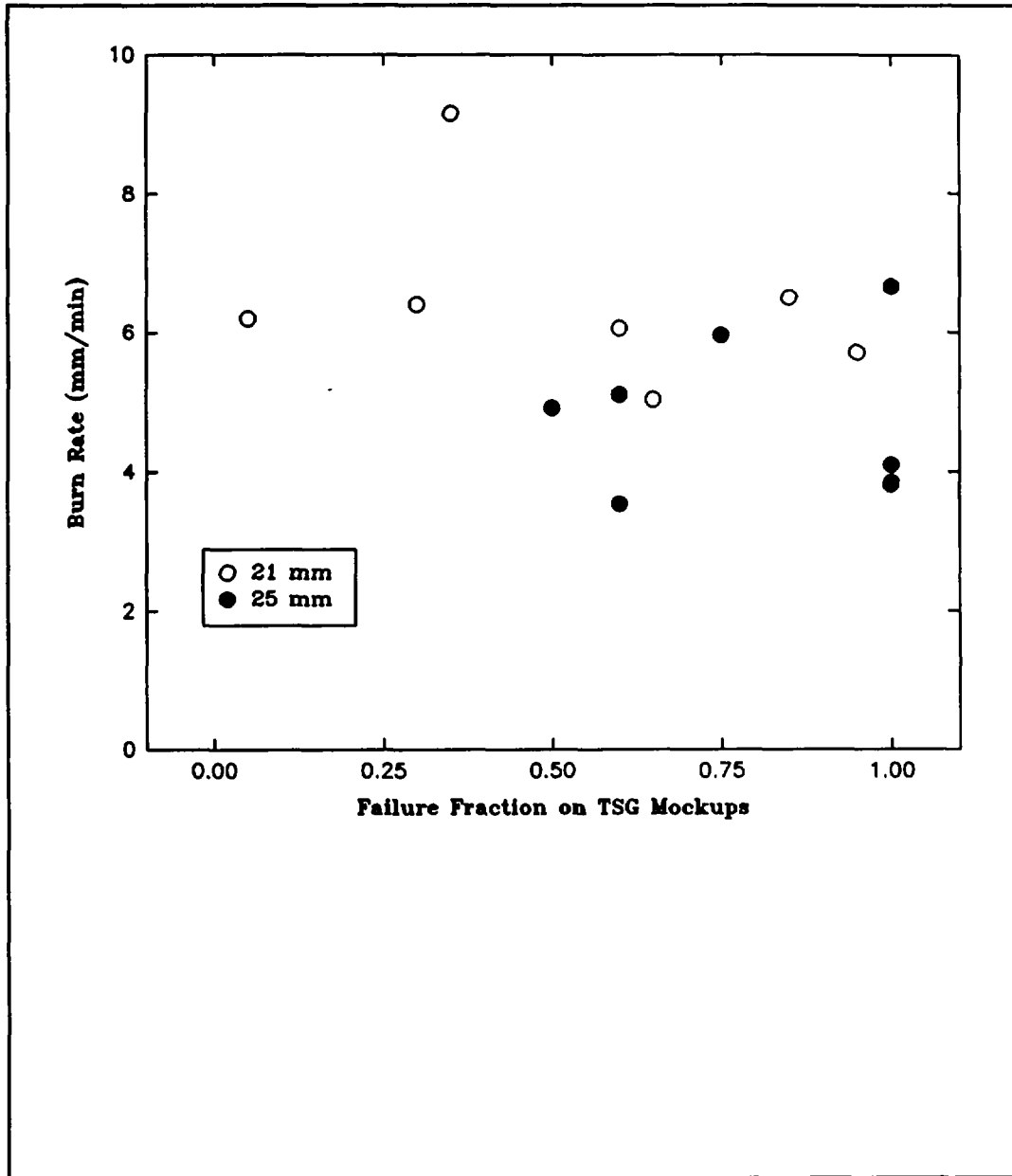


Figure 7. Free Burning Rate of Various Cigarettes Suspended in Quiescent Air as a Function of the Fraction of the TSG Mock-Up Failures.

**Cigarette free-burn rate.** In order to characterize baseline cigarette performance and revisit the possible use of a no-substrate test method, the burning rates of certain of the experimental cigarettes were determined under what is called "free-burn" conditions. Cigarettes were allowed to smolder while suspended horizontally in a quiescent atmosphere. All cigarettes tested in this configuration smoldered their entire length. In Figure 7, the burning rates of 16 of the TSG experimental cigarettes are plotted as a function of the ignition fractions of these cigarettes on the TSG mock-up substrates. The average burn rate for all cigarettes was  $5.9 \pm 1.9$  mm/min. The scatter clearly exceeds the slope of a least squares line through the data. Therefore the free burn rate of a cigarette cannot be used by itself as a predictor of ignition propensity.

**Non-Reactive Substrates.** Non-reactive substrates are those that do not generate or absorb heat chemically when heated by a cigarette. An example of such a substrate from previous work is a glass plate. Aside from the heated glass plate previously reported, research efforts were directed at investigating three types of non-reactive substrates. These were: a bed of glass beads, a set of glass rods, and a non-woven glass fiber paper system.

**Glass Beads.** Figure 8 shows a schematic representation of a test setup that was used for the evaluation of glass beads as a suitable substrate for a secondary test method. The test setup consisted of a Pyrex funnel with a 125 mm diameter opening. A wire screen was suspended in the funnel such that a space of 25 mm existed between the screen and the lip of the funnel. This space was filled with glass beads, either 6 mm or 2 mm in diameter. Initial tests were conducted in a quiescent atmosphere. Subsequent tests were conducted with an imposed air flow through the glass beads, which served to disperse the flow evenly. This flow was perpendicular to the direction of cigarette smoldering. An attempt was made to find an air flow necessary to force a cigarette to smolder its entire length.

Preliminary tests were conducted on a selected subset of the TSG cigarettes at three air flows, as listed in Table 25. The air speeds through the bed of glass beads (calculated from the measured volumetric flow and the bed cross-section) were very small: 0, 0.44, 0.89 cm/min. Table 25 shows that for cigarettes 106 and 129, the number that burned their entire length increased somewhat in going from 0 cm/min to 0.44 cm/min. However, the same two cigarettes, when tested at 0.89 cm/min, showed a decrease in the number of cigarettes burning their entire length. Cigarettes 106, 129, and 130 were retested at the intermediate airflow rate of 0.44 cm/min. The retests showed somewhat erratic results, as can be seen in Table 26.

It became clear as more replicate tests were performed that cigarette performance depended a great deal on the contact characteristics of the cigarette/glass bead interface and that a reliable and reproducible contact profile could not be assured with the glass bead system. Thus this approach was not pursued further.

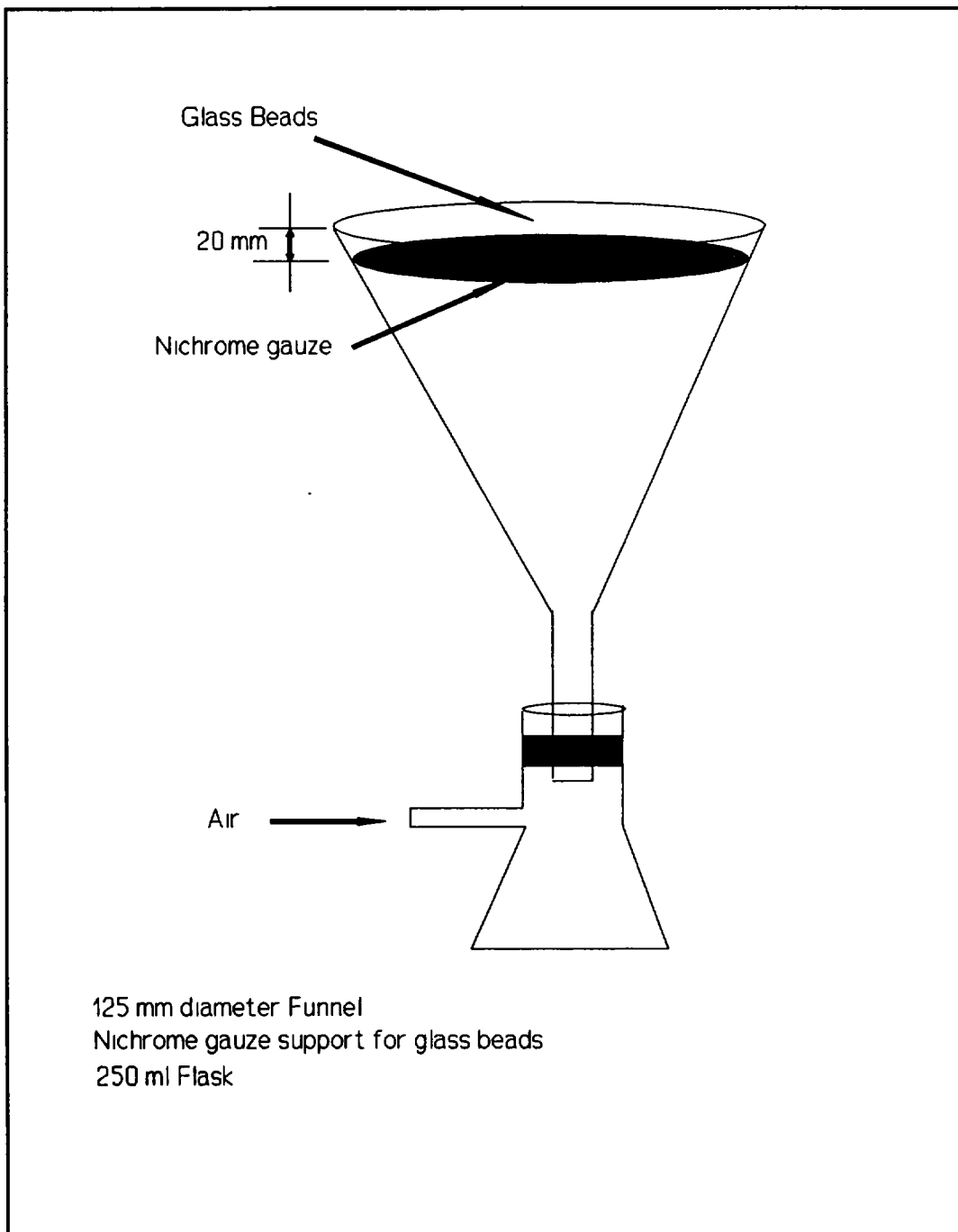


Figure 8. Schematic Representation of the Test Assembly for the Glass Bead/Rod Substrate Tests.

**Table 25. Large Glass Bead Non-Reactive Substrate Test Results for Selected Cigarettes and Air Speeds**

Cigarette Designation	0 cm/min		0.44 cm/min		0.89 cm/min	
	# Burn/ # Tested	Burn Time (s)	# Burn/ # Tested	Burn Time (s)	# Burn/ # Tested	Burn Time (s)
101	5/5	800 ± 60	5/5	730 ± 40		
103	5/5	590 ± 40	5/5	600 ± 20		
106	0/5	340 ± 130	5/5	665 ± 110	3/5	480 ± 200
108	5/5	570 ± 70	5/5	520 ± 95		
120	5/5	725 ± 30	5/5	690 ± 45		
129	1/5	520 ± 405	3/5	880 ± 100	1/5	390 ± 345
130	0/5	100 ± 60	0/5	635 ± 225		
131	5/5	640 ± 53	5/5	720 ± 85		
Total	26/40		33/40		4/10	

**Table 26. Re-test of Selected Cigarettes on Large Glass Bead Substrate; Air Speed = 0.44 cm/min**

Cigarette No.	# Burn/ # Tested	Burn Time (s)
106	1/5	540 ± 100
129	3/5	740 ± 25
130	0/5	145 ± 130

Glass Rods. The bed of glass beads was replaced by a pair of parallel glass rods, which were positioned on the supporting screen described above. The cigarette was placed on the rods parallel to their length, and the rods were spaced to ensure minimal contact between the cigarette and the glass rods. Air flowed upward past the smoldering cigarette as in the previous experiments. All the test cigarettes self-extinguished. This avenue of research was not pursued any further, although several test variables could have been adjusted that might have improved the discrimination capabilities of the test setup. These include: varying the temperature of the imposed airflow, replacing the glass rods with other materials, and altering the temperature of the supporting rods.

**Non-Woven Glass Fiber Paper.** In all cases, cigarettes burned their entire length when placed on a single sheet of non-woven glass fiber filter paper (with the paper suspended horizontally in air). The effective thermal inertia of this glass fiber filter paper was sufficiently low that it extracted heat from the burning cigarette less effectively than did the glass beads. While it was expected that multiple layers of this filter paper would reduce the likelihood of a cigarette burning its entire length, no such effect was observed. Instead, cigarettes such as 106 would continue burning even when supported on 10 layers of glass fiber filter paper.

This result suggested that it might be possible to use this type of filter paper to measure the heat transfer from a smoldering cigarette to a substrate material. An apparatus (Figure 9) was designed and constructed that consisted of a PMMA (polymethylmethacrylate) box with outside dimensions of 90 mm by 125 mm by 25 mm. A sheet of the glass fiber filter paper served as a cover. Three thermocouples were placed along a long diagonal of the box in an air gap approximately 12.7 mm deep between the box cover paper and the interior base of the box. The thermocouples were wired in parallel to monitor the average temperature of the three sensors.

Figure 10 shows a typical plot of the results from a single test. The graph shows the temperature-time history resulting from a complete cigarette burn. Several attempts were made to summarize and interpret the results of a single test. The best correlation to cigarette ignition propensity was an estimated heat transfer to the air gap. This involved computing a number proportional to an approximate measure of the heat content of the air in the gap, as follows:

$$Q' = T_{pk} \times \tau_{pk} \quad (^\circ\text{C} \cdot \text{min})$$

where

$T_{pk}$  = Peak temperature ( $^\circ\text{C}$ )

$\tau_{pk}$  = Time of peak temperature (min)

Figure 11 summarizes the results for 16 different cigarettes, plotting the estimated heat content as a function of percentage mock-up ignitions from the TSG study [3]. While the data suggest that a correlation exists, the method requires further investigation and refinement. It was found that results could be dramatically affected by the specific location of the cigarette on the glass fiber filter relative to the thermocouples. Also, in order to yield repeatable results, the holder assembly needed to be cooled to ambient conditions between tests (this could take 20 to 30 minutes). Additional work along these lines was not pursued because of the success of the reactive substrate method described below.

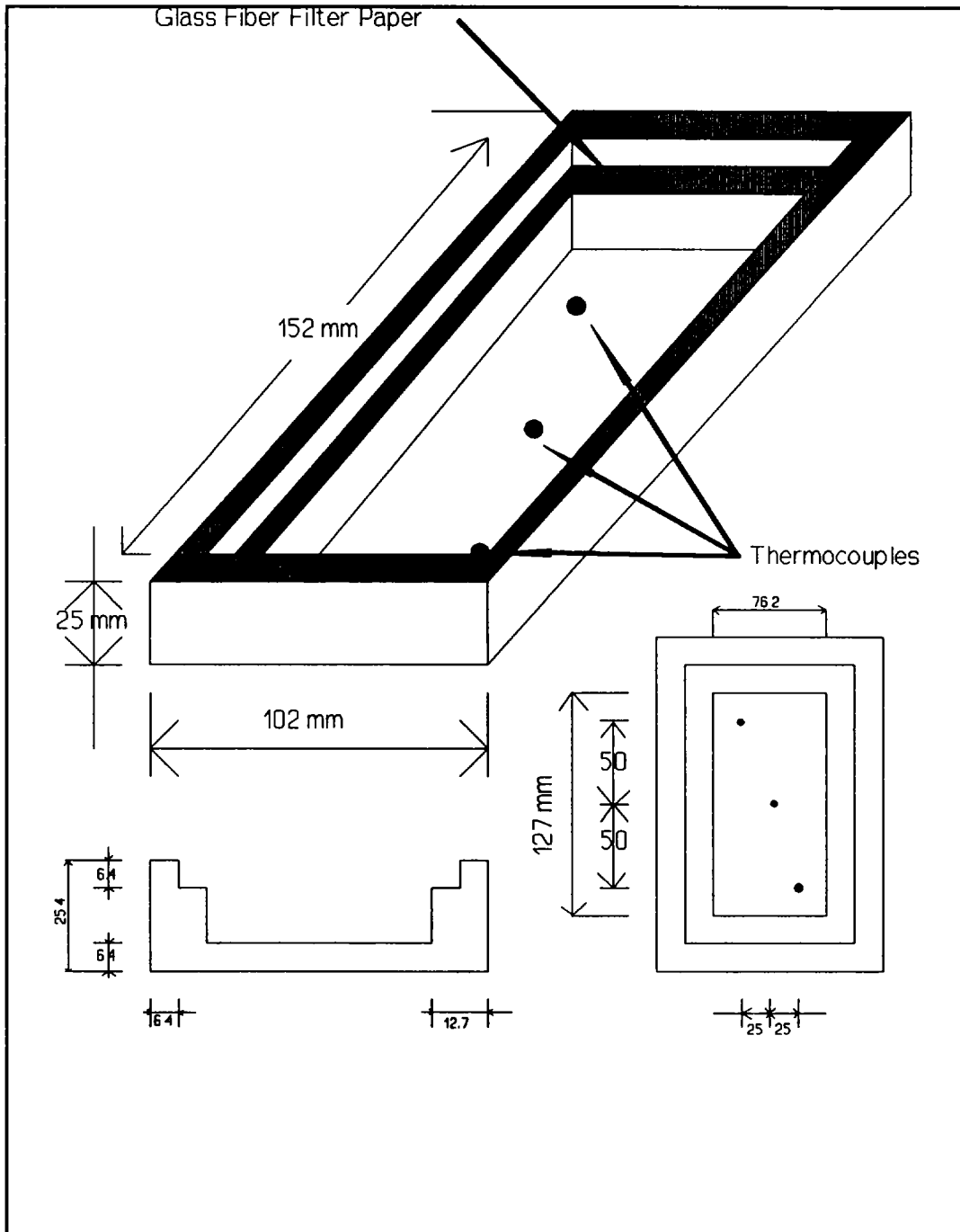


Figure 9. Drawing of the Cigarette Thermal Transfer Test Assembly.

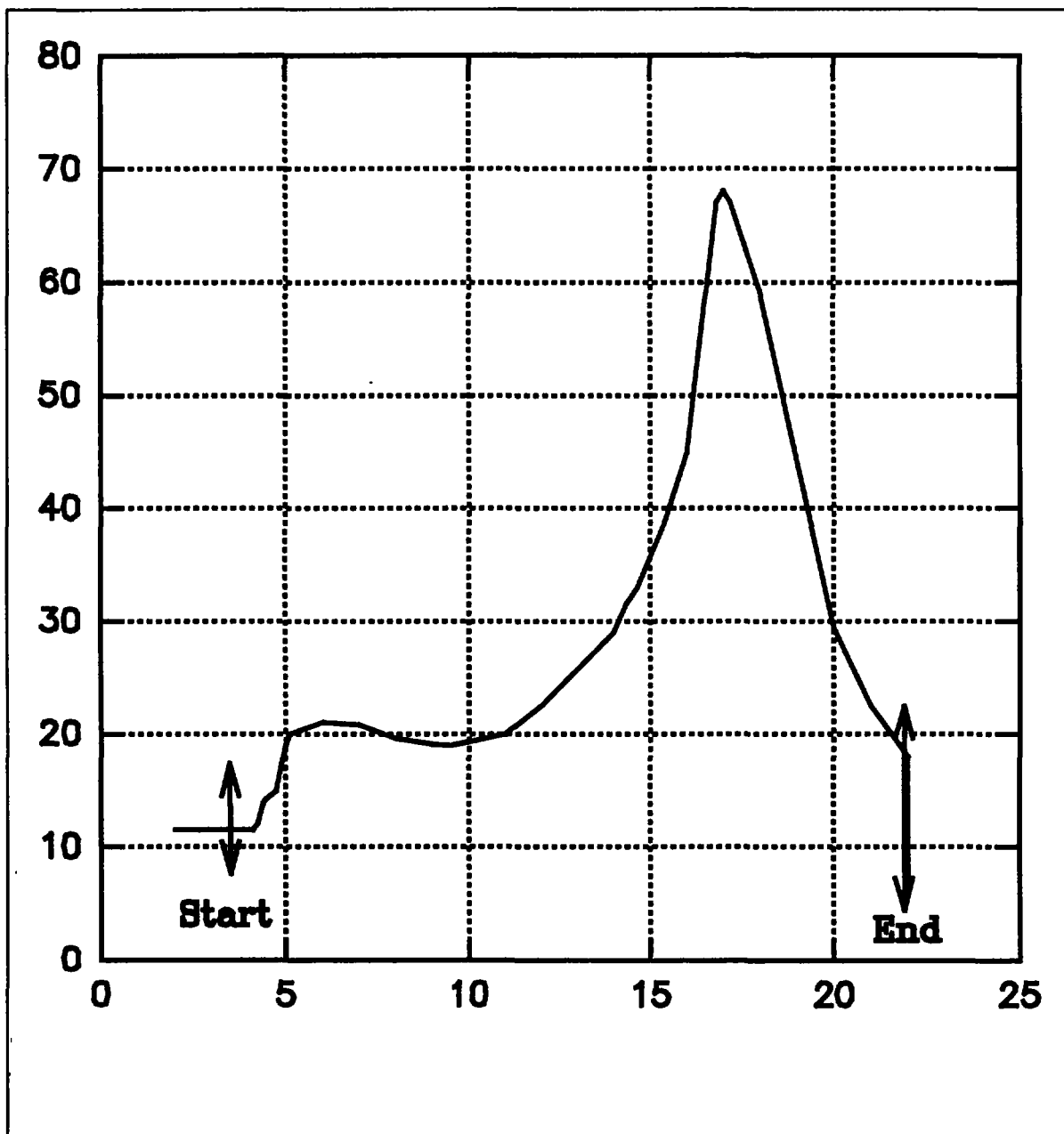


Figure 10. Typical Average Temperature-Time Trace for a Cigarette Burning on a Glass Fiber Filter Paper in the Cigarette Thermal Transfer Test Assembly.



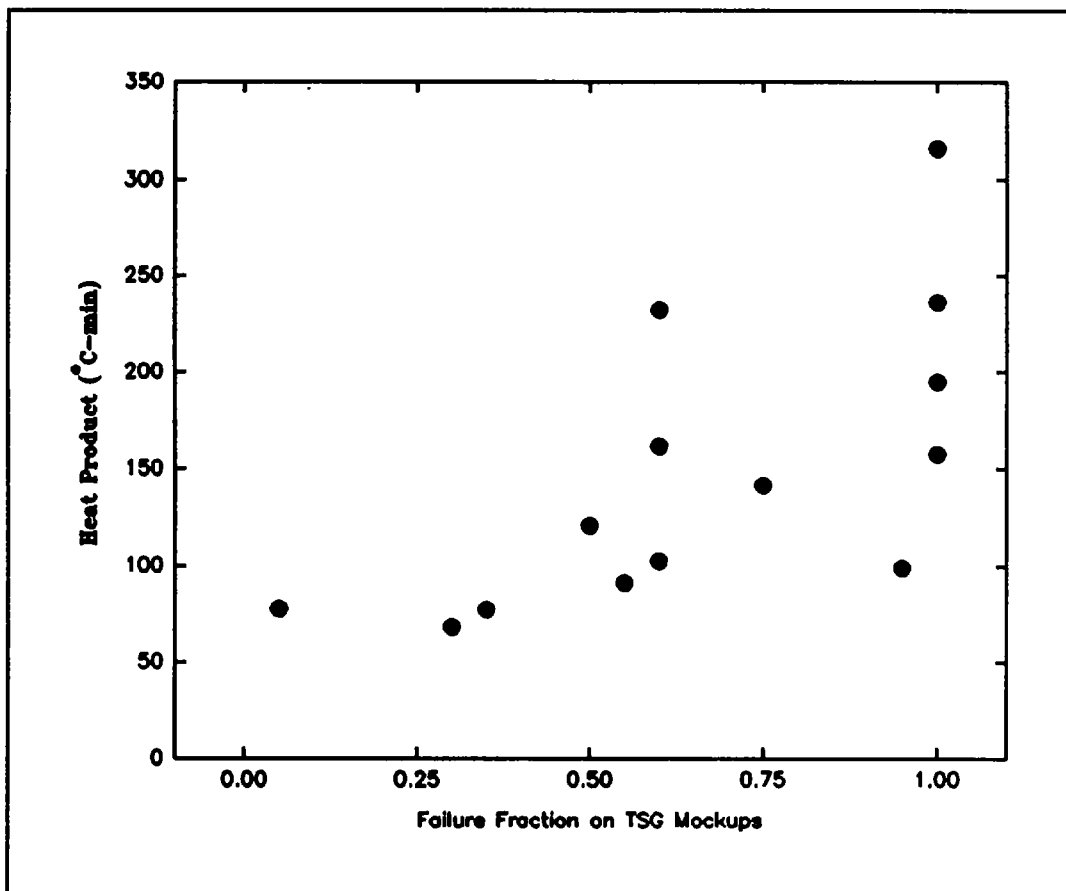


Figure 11. Estimated Energy Transferred to a Substrate from a Smoldering Cigarette Burning in the Thermal Transfer Apparatus as a Function of the Fraction of TSG Mock-Up Failures.

**Reactive Substrates.** Reactive substrates undergo significant chemical change when heated by a cigarette coal. In the present study, the need was to identify reactive substrates that had advantages over the foam/fabric assemblies. Thus, these materials had to be:

- easily obtained now and in the future;
- well-characterized;
- highly uniform, both within a sample and batch-to-batch;
- smooth-surfaced; and
- available in large quantities.

After screening tests were conducted on several substrate materials (e.g., lens paper, different grades of filter paper, bond paper, etc.), Whatman #2 filter paper emerged as the choice. The idea for using an alpha cellulose substrate had originated with Krasny in 1981 [32]. He used multiple layers of alpha-cellulose to support the cigarette. Since the smoldering promotor ion concentration is very small in these papers, any charring of the substrate is limited to the cigarette/substrate contact area. When the cigarette extinguishes, the substrate will not continue to smolder.

Here, it was initially assumed that the smoldering rate of a cigarette in contact with the substrate could be used as an indicator of ignition propensity. That is, the heat loss to the pyrolyzing paper would slow the burning rate of a cigarette; and at some magnitude of heat loss, the cigarette would self-extinguish. Since a single sheet of the paper is thermally-thin (small temperature gradient through its depth), more layers would extract more heat. Preliminary experiments were aimed at developing a relationship between cigarette burn rate and substrate thickness as defined by the number of filter layers in the substrate assembly. It was expected that as the number of filter paper layers increased, the burning rate would decrease. As was true for all the work performed in this program, tests were conducted in a enclosure system comparable to that used in the mock-up testing program described in the TSG report [3].

Figure 12 shows the smoldering rates of three TSG cigarettes as a function of the number of filter papers in the substrate assembly. There is a general downward trend in the data for a given cigarette. The changes, however, are not sufficient to discriminate even among cigarettes of distinctly differing ignition propensities, such as cigarette 106 with a TSG rating of 5% ignitions and cigarette 112 with a TSG rating of 100% ignitions.

However, it was noted that, as the number of filter paper layers was increased, specific cigarettes would not burn their entire length. Therefore, further work was directed at whether there was a relationship between the maximum number of filter papers in a substrate assembly that just allows a cigarette to burn its entire length and its ignition propensity as defined by the TSG ignition probabilities. A simple apparatus consisting of layers of filter paper on a metal ring was used to test the hypothesis. The preliminary data (3-5 replicates) in Figure 13 show that such a relationship does exist. The correlation covers a wide dynamic range: TSG ignition probabilities from 5% to 100% and 1 to 20 layers of filter paper.<sup>7</sup>

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<sup>7</sup> Note that there is nothing to be gained by going to an indefinitely greater number of sheets. The heat from the cigarette can penetrate only so far in the time available. It is estimated that 25-30 sheets constitute a thermally thick medium.

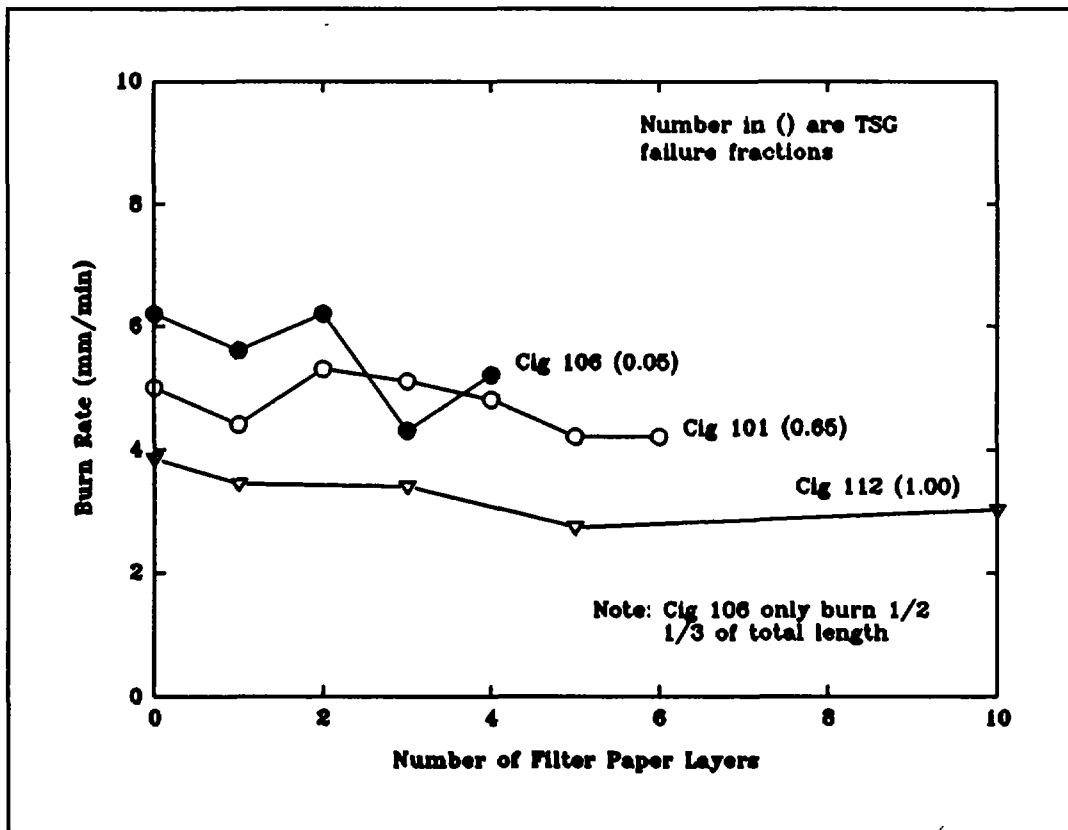


Figure 12. Smoldering Rates of Three Experimental Cigarettes as a Function of the Number of Filter Papers Making up the Substrate Assembly.

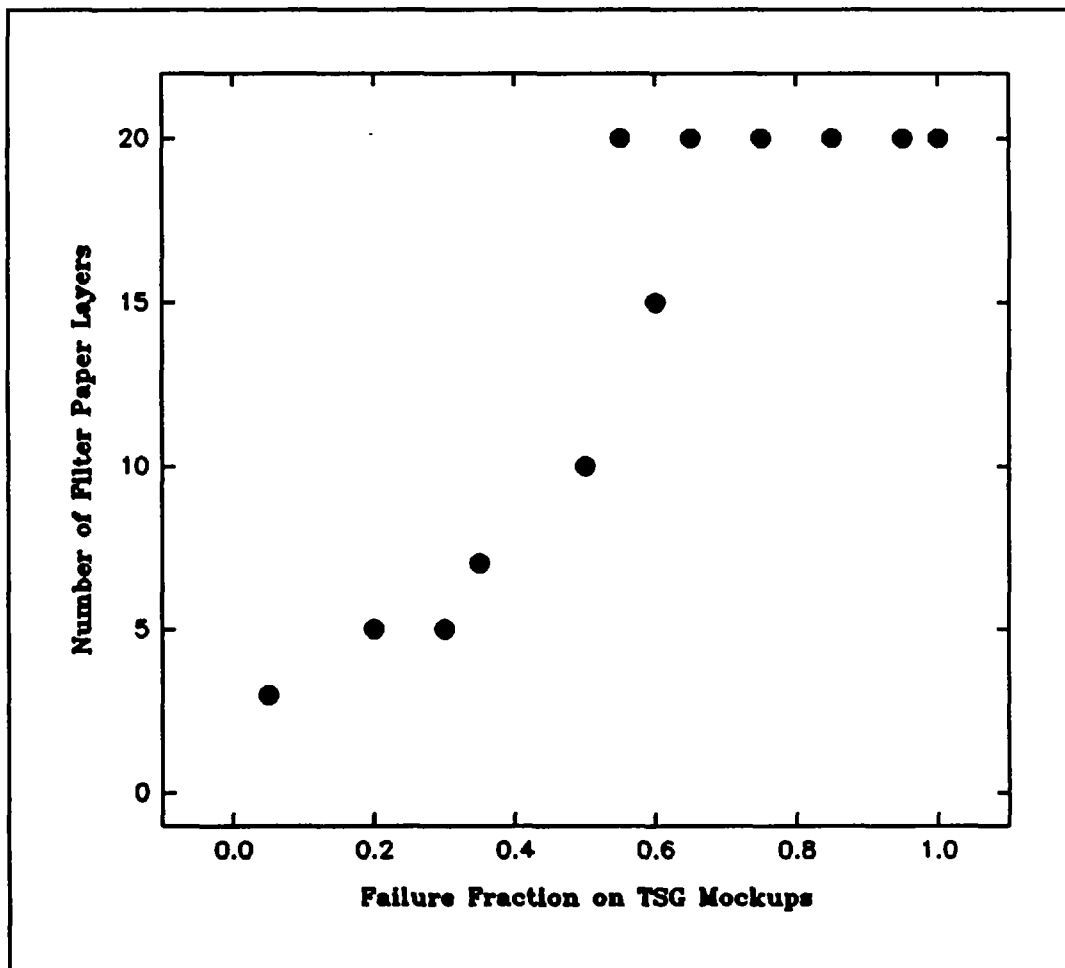


Figure 13. Number of Filter Papers Causing Extinguishment of the Cigarette as a Function of the TSG Failure Fraction.

Further testing showed that some cigarettes had a tendency to roll across the surface of the filter paper during a test. This altered the estimate of cigarette ignition propensity. A modified substrate holder assembly was developed. The holder assembly held folded filter paper such that each side of the filter paper stack was set at an angle of 20° from the horizontal. This helped ensure that cigarettes would not roll across the paper surface.

Because of the costs involved in manufacturing the 20° holder assembly and the tendency for filter paper separation to occur at the crevice joint, restraints were developed instead for the flat holder assembly. The final system is shown in Figure 14. It is composed of a brass hold-down ring with two sets of small metal rods to prevent a cigarette from rolling (yet without applying excessive pressure on the cigarette) and a plastic filter paper support structure. Each set of metal rods are spaced for a small range of cigarette diameters. As additional cigarette diameters are encountered, appropriately spaced metal rods can be added.

### 3. Standard Materials

**Paper Substrate.** The cigarette extinction test method uses multiple layers of Whatman #2 filter paper as the substrate material. It is a well-characterized material, having a well-defined porosity and filtration speed and a smooth surface finish. A single sheet has an areal density of  $9.8 \times 10^{-2} \text{ kg/m}^2$ , with low variability (Table 27). It is made from a single material (alpha-cellulose) and should be obtainable indefinitely into the future. It is also readily available in a variety of shapes and sizes. Because of the lengths of currently manufactured cigarettes, it was felt that the standard 150 mm diameter size would be sufficient for cigarette ignition propensity measurements. The precut material reduces handling damage that might occur if each technician had to cut the paper to size.

The data in Table 27 were obtained by averaging six samples taken at random from six different boxes of Whatman #2 filter paper.

**Table 27. Variability of Filter Paper Areal Density and Thickness**

Box	Areal Density ( $\text{kg/m}^2$ )	Thickness (mm)
A	$(10.02 \pm .18) \times 10^{-2}$	$0.195 \pm .004$
B	$(9.74 \pm .10) \times 10^{-2}$	$0.182 \pm .003$
C	$(9.80 \pm .13) \times 10^{-2}$	$0.181 \pm .005$
D	$(9.58 \pm .13) \times 10^{-2}$	$0.184 \pm .005$
E	$(9.71 \pm .18) \times 10^{-2}$	$0.183 \pm .004$
F	$(9.68 \pm .32) \times 10^{-2}$	$0.187 \pm .005$
Overall:	$(9.76 \pm .15) \times 10^{-2}$	$0.185 \pm .005$

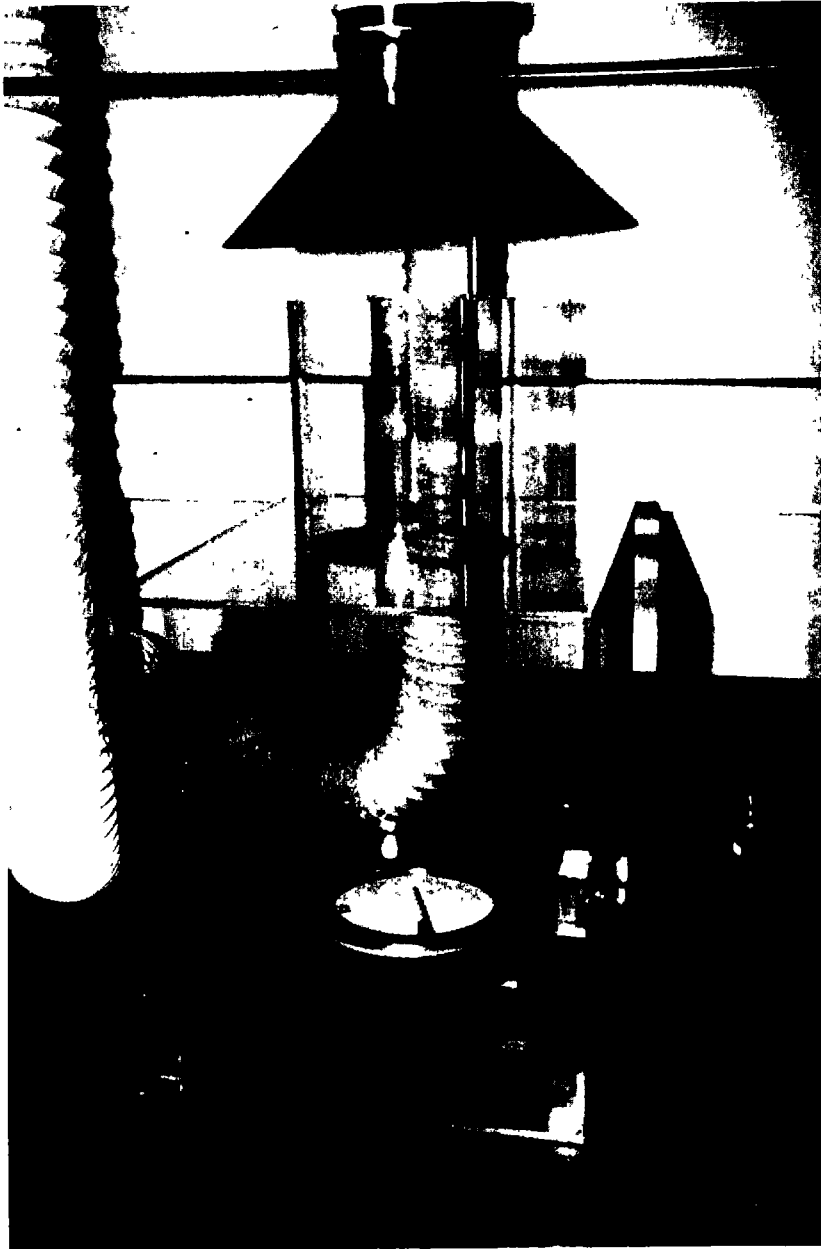


Figure 14. Photograph of a Test Chamber Containing a Mock-Up Assembly and a Cigarette.

**Substrate Description.** The test method concept originally involved determination of the actual number of filter paper layers necessary to just allow the cigarette to burn its complete length. To reduce the testing burden on the participating laboratories as well as to reduce the amount of filter paper used in each cigarette evaluation, the interlaboratory evaluation was performed with three specific numbers of layers. This also enabled using a statistical design comparable to the one used for the mock-up ignition test method. The substrates comprised 3, 10, and 15 layers of Whatman #2 filter paper. In practice, the original concept may have application as well.

#### **4. Enclosure Design**

This test method simply replaces the fabric/foam substrate with an alternative substrate assembly. Since it has been demonstrated that the enclosure used in the mock-up ignition test method adequately protects the cigarette-substrate system from laboratory induced air flows, that enclosure was also employed in the cigarette extinction test method.

#### **5. General Description of the Test Method**

Appendix E gives a detailed description of the cigarette extinction test method. In brief, the test method measures whether a type of cigarette continues smoldering after being placed on substrate assemblies that have different thermal absorptivities. The appropriate number of layers of Whatman #2 filter paper are mounted on the support structure described above and placed in the enclosure. The cigarettes and substrate assemblies are conditioned at a relative humidity of  $55 \pm 5\%$  and a temperature of  $23 \pm 3$  °C. Cigarettes are ignited and pre-burned to a 15 mm mark as described for the Mock-Up Ignition Test Method. The principal determination is whether the cigarette burns its full length or not.

#### **6. Interlaboratory Study of the Test Method**

##### **a. Participants and Procedures**

The nine laboratories participating in this phase of the interlaboratory study were the same as previously listed for the main ILS. See the list of participants in Section II.B.8.e. The general test protocol for this phase of the ignition propensity study followed that outlined for the ILS of the mock-up extinction method. The only major differences were that (a) a different method was being studied and (b) fewer replicates were performed. The latter was proposed since the substrate variability, thought to be a potential factor in the precision in the mock-up method, was minimal here. The reduced test plan used only that portion of the plan specified for each laboratory in the first week of testing during the main ILS. The following outlines key parameters pertaining to each laboratory in this study:

- 5 cigarettes
- 3 filter paper substrates (3, 10 and 15 sheets thick)
- 16 replicates per cigarette per substrate
- 2 operators
- 4 test chambers

- 1 week test period

Each laboratory received 25 boxes of filter paper, four plastic filter paper supports and brass hold-down rings, 100 coded cigarettes, plus instructional manuals and lab workbooks for each operator. Each laboratory already had in their possession test chamber enclosures, cigarette lighters, cigarette holders, etc. from the mock-up ignition interlaboratory test program. Similar procedures were followed to ensure timely arrival of test data to NIST via the FAXing of daily summary sheets. At the end of the one-week test program, workbooks and data disks were returned to NIST. These were reviewed for consistency between the workbooks, disk data files, and daily summary sheets. Discrepancies were noted and resolved to ensure an accurate set of data files from each laboratory.

#### **b. Analysis of Results**

**Raw Data.** As was done for the previous interlaboratory studies, the data for the cigarette extinction test method were organized into a single computer file for analysis. For this study, the resulting computer file contained 2160 lines of data, corresponding to 240 ignition results per laboratory for each of 9 laboratories. The 240 results per laboratory arise from 16 tests of each of 5 cigarettes on each of 3 substrates.

As was the case for the main ILS of the Mock-Up Ignition Method, there were a few cases in the ILS of the Cigarette Extinction Method where laboratories ran some test replications on the wrong mock-up configurations. This resulted in some cases where the number of results reported for a given cigarette type and mock-up configuration differ, again by  $\pm 2$ , from the desired number of 16 replications.

The raw data on each line of the computer file represent essentially the same set of thirteen variables described in Table 20, with the familiar changes that (a) the LAB variable ranges from 1 to 9, and (b) the OPERATOR variable codes the two operators as number "1" and number "2." These differences were discussed in more detail in Section II.B.8.c. Another difference from Table 20 is that the TST\_BLK variable was not used because this study was done in a single week of testing.

A summary of the test results, by LAB, CIG\_TYPE and SUBSTRAT, is presented in Table 28. The identifying numbers for the laboratories are the same as those used in Table 22. The test results for the extinction method were reported in two categories, full-length burns and self-extinguishments.



**Table 28. Summary of Test Results for Interlaboratory Study of Cigarette Extinction Method**

Cigarette Type	Substrate (No. of Layers)	Laboratory	Test Results	
			Full-Length Burns	Self-Extinguishments
501	3	1	16	0
		2	16	0
		3	16	0
		4	16	0
		5	16	0
		6	16	0
		7	16	0
		8	16	0
		9	16	0
	10	1	16	0
		2	16	0
		3	16	0
		4	16	0
		5	16	0
		6	16	0
		7	16	0
		8	16	0
		9	16	0
	15	1	16	0
		2	16	0
		3	16	0
		4	16	0
		5	16	0
		6	16	0
		7	16	0
		8	16	0
		9	16	0

Cigarette Type	Substrate (No. of Layers)	Laboratory	Test Results	
			Full-Length Burns	Self-Extinguishments
503	3	1	16	0
		2	16	0
		3	16	0
		4	16	0
		5	16	0
		6	16	0
		7	16	0
		8	16	0
		9	16	0
	10	1	16	0
		2	16	0
		3	16	0
		4	18	0
		5	16	0
		6	16	0
		7	16	0
		8	16	0
		9	16	0
	15	1	16	0
		2	16	0
		3	16	0
		4	14	0
		5	16	0
		6	16	0
		7	16	0
		8	16	0
		9	16	0

Cigarette Type	Substrate (No. of Layers)	Laboratory	Test Results	
			Full-Length Burns	Self-Extinguishments
529	3	1	7	9
		2	8	8
		3	11	5
		4	9	7
		5	8	8
		6	10	6
		7	8	8
		8	8	8
		9	13	3
	10	1	0	16
		2	1	15
		3	0	16
		4	0	16
		5	0	16
		6	1	15
		7	0	16
		8	1	15
		9	5	11
	15	1	0	16
		2	0	16
		3	0	16
		4	0	16
		5	0	16
		6	0	16
		7	0	16
		8	1	15
		9	2	14

Cigarette Type	Substrate (No. of Layers)	Laboratory	Test Results	
			Full-Length Burns	Self-Extinguishments
530	3	1	2	14
		2	0	16
		3	0	16
		4	0	16
		5	0	16
		6	2	14
		7	1	15
		8	1	15
		9	2	14
	10	1	0	16
		2	0	16
		3	0	16
		4	0	16
		5	0	16
		6	0	16
		7	0	16
		8	0	16
		9	0	16
	15	1	0	16
		2	0	16
		3	0	16
		4	0	16
		5	0	16
		6	0	16
		7	0	16
		8	0	16
		9	0	16

Cigarette Type	Substrate (No. of Layers)	Laboratory	Test Results	
			Full-Length Burns	Self-Extinguishments
531	3	1	16	0
		2	16	0
		3	16	0
		4	16	0
		5	16	0
		6	16	0
		7	15	1
		8	16	0
		9	16	0
	10	1	14	2
		2	16	0
		3	15	1
		4	13	3
		5	16	0
		6	16	0
		7	15	1
		8	16	0
		9	15	1
	15	1	10	6
		2	13	3
		3	14	2
		4	15	1
		5	15	1
		6	14	2
		7	16	0
		8	15	1
		9	15	1

A graphical display of the data in Table 28 is shown in Figure 15, where, for each cigarette (by columns) and substrate (by rows) the proportion of full-length burns is represented by a vertical bar for each laboratory. The cigarette types are shown from left to right in order of decreasing ignition propensity, as determined by the results of the main interlaboratory study of the Mock-Up Ignition Test method (see Figure 4). A comparison of Figure 15 with Figure 4 shows that, except for cigarettes 501 and 503 which are tied in Figure 15, the relative positioning of the cigarettes was the same in both the studies. The three mock-up configurations are shown as rows in the figure, with the greatest heat-sink substrate (15 layers of filter paper) as the top row and the least heat-sink substrate (3 layers of filter paper) as the bottom row of the figure. The stronger the heat sink ability of the substrate, the more difficult it is for a cigarette to burn its full length on that substrate.

**Auxiliary Variables.** A few statistical procedures were run to check for any interesting or large effects on the test results associated with the available auxiliary variables. No statistically significant effects were found associated with the temperature and humidity variables over the ranges occurring in these tests. For comparison with the previous interlaboratory studies, box plots showing the ranges of the temperatures and humidities during testing are given in Figure 16.

The Cochran-Mantel-Haenszel test was used to check for significant effects on ignition results due to the discrete variables. No significant effect was found due to OPERATOR; however, the tests on AMPM and CHAMBER did achieve statistical significance. For the AMPM variable, lab 7 showed a significant effect. Detailed study of the data for lab 7 revealed that the significant difference that was picked-up by the overall Cochran-Mantel-Haenszel test procedure was entirely due to the results for cigarette 529 on the 3-layer substrate. In that case, there were 7 ignitions and 1 self-extinguishment in the AM and just the opposite, 1 ignition and 7 self-extinguishments, in the PM. Using Fisher's Exact Test for the resulting 2x2 contingency table yields a significance probability of 0.01. No other aspect of the data for this case looks unusual, so the decision was made to accept the data as-is. It is relevant to note that, since a large number of significance tests were conducted on this data set, one would expect a few cases to show statistical significance simply due to the expected amount of random variation in the data.

For CHAMBER, the statistically significant effect flagged by the Cochran-Mantel-Haenszel test was for lab 3 only. Within the data for lab 3, the significance was caused by the results for the 16 tests of cigarette 529 on the 15-layer substrate. The significance probability for this case was 0.01 by Fisher's Exact Test. Again, nothing else unusual was found regarding the data in question, and the existing data were used in the repeatability and reproducibility summary without modification.

**Primary Variables.** Statistical tests were carried out to examine whether these interlaboratory study data reveal differences between the labs, cigarette types and substrates. For labs, there is a statistically significant difference only for cigarette 529 on the 10-layer substrate. The fact that only one case showed a difference for the Cigarette Extinction Method is at least partly due to the fact that only 16 replications were done per laboratory, rather than the 48 in the main ILS of the Mock-Up Ignition Method. With fewer data, fewer significant differences are likely to be found, even if the long-run differences are about the same.

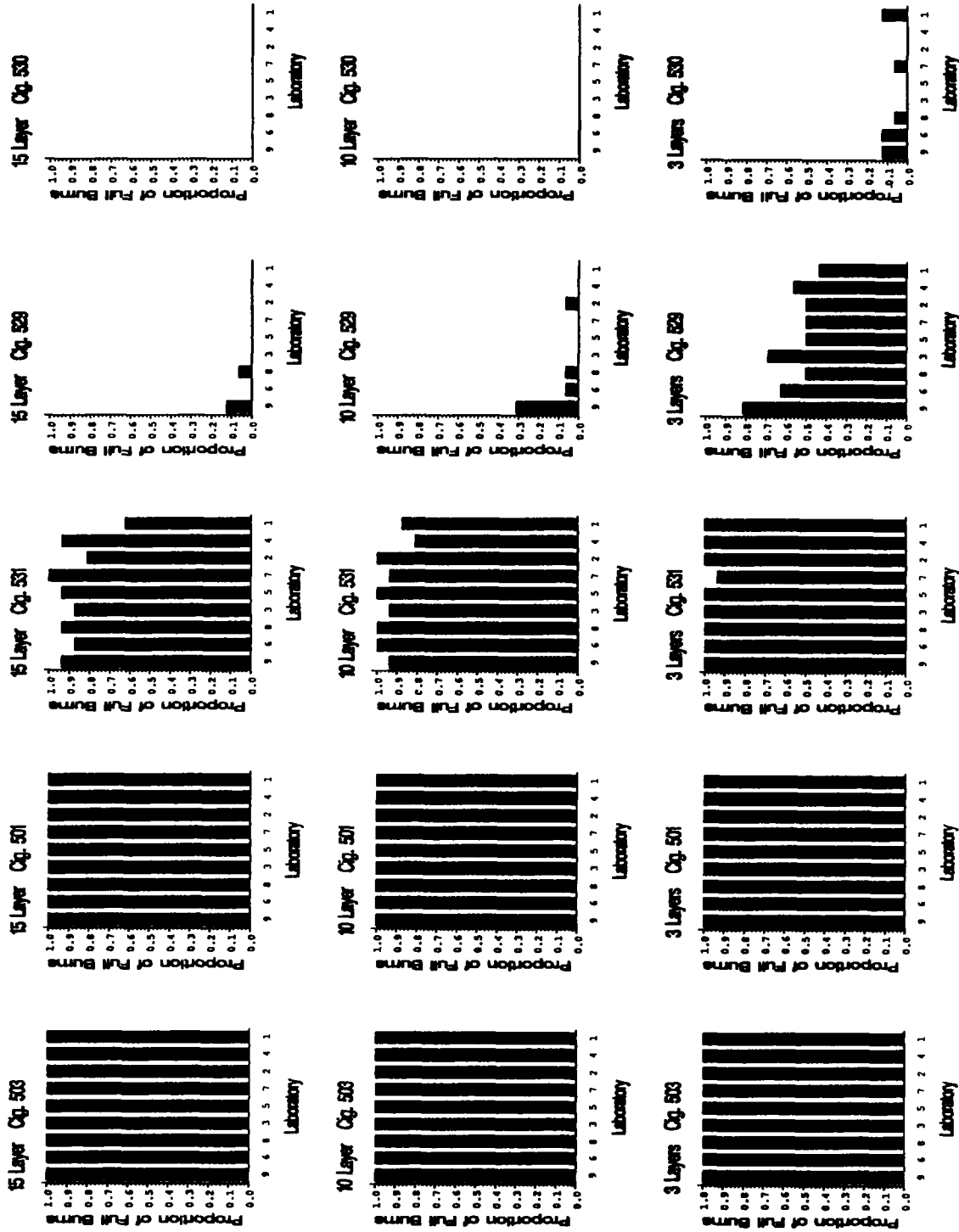


Figure 15. Comparison of Full Burn Rates for the Interlaboratory Study of the Cigarette Exinction Test Method. The 15 plots in the figure correspond to the 5 cigarette types tested, by columns, and the 3 test substrates (15, 10, and 3 filter layers), by rows. In each component plot, the vertical bars represent the proportions of full burns obtained by each of the 9 participating laboratories.

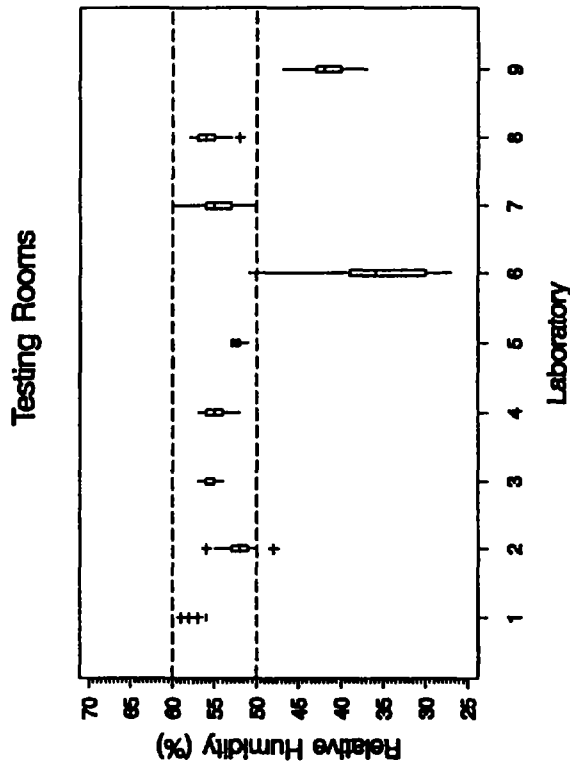
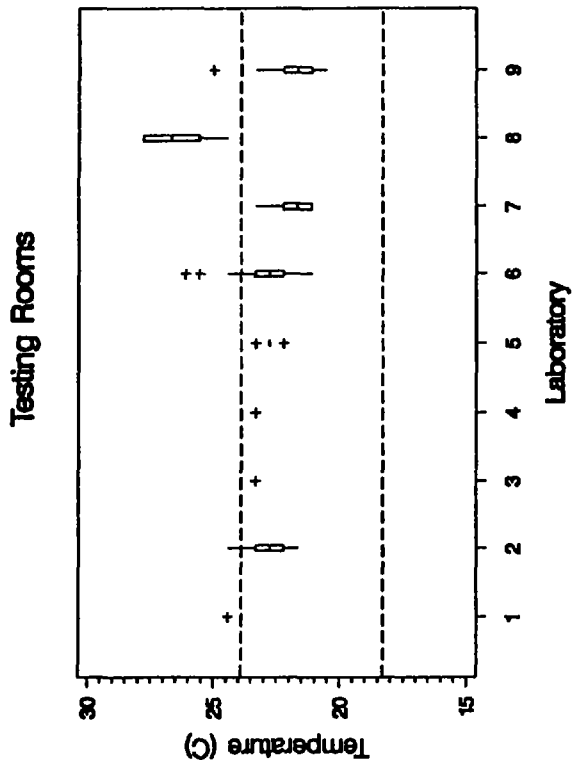
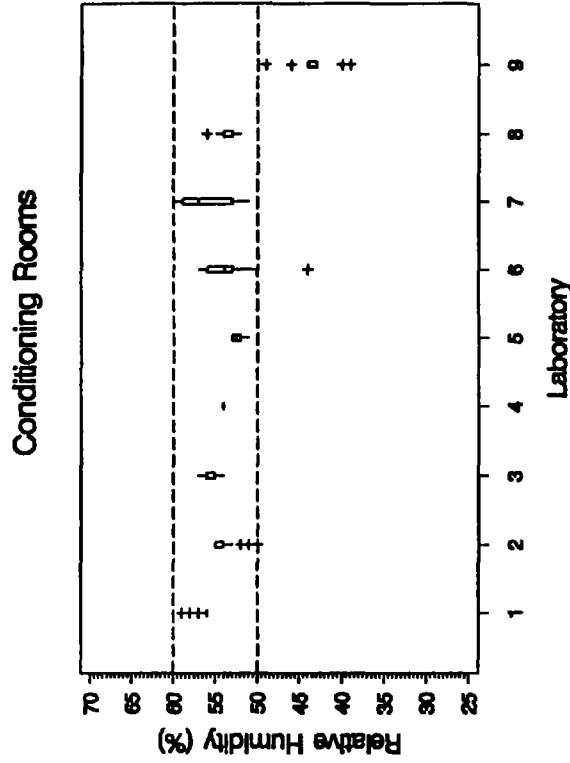
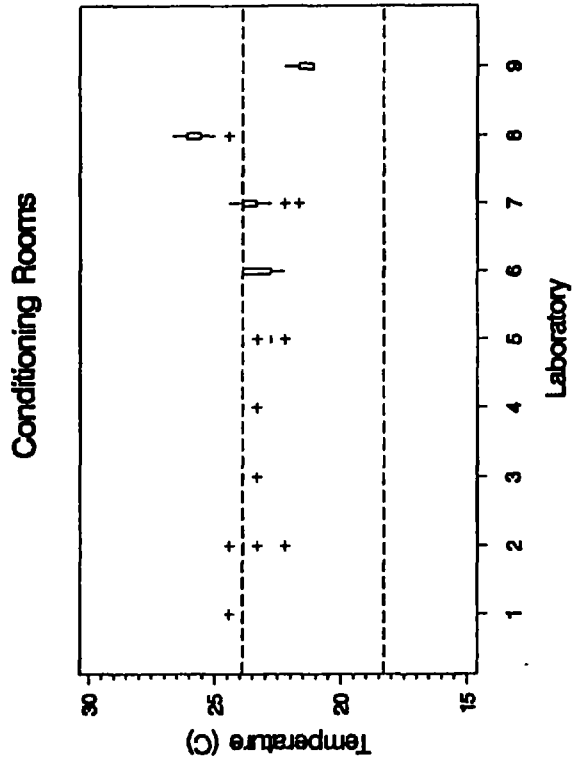


Figure 16. Environmental Conditions Reported by the Laboratories Participating in the Interlaboratory Study of the Cigarette Extinction Test Method.



Except for the fact that cigarettes 503 and 501 gave identical results (100% full-length burns for all labs and all substrates) the Cochran-Mantel-Haenszel procedure showed that the cigarettes differ from each other: 501 and 503 having higher full-length burn proportions than 531, which is higher than 529, which is higher than 530. Similarly, the observed differences in full-length burn proportions between the three substrates are all statistically significant according to the Cochran-Mantel-Haenszel procedure.

**Repeatability and Reproducibility.** The summary shown in Tables 29 and 30 follows the same methodology described previously for Tables 23 and 24. The only difference in detail is that, in the interlaboratory study for the Cigarette Extinction Method, the number of replications per lab was  $m=16$ , compared to  $m=48$  in the Mock-Up Ignition Method. Therefore, in Table 29, the repeatability standard deviation is calculated as  $S_r = [\bar{p}(1-\bar{p})/16]^{1/2}$ .

The model for extra-binomial variation used previously for the Mock-Up Ignition Method ILS was also applied to these data. The observed relation between the reproducibility variance,  $S_R^2$ , and the repeatability variance,  $S_r^2$ , for the Cigarette Extinction Method ILS is shown graphically in Figure 17. The slope of the least squares line in the Figure is 1.146. Setting this value equal to the heterogeneity factor,  $[1 + \varphi(16-1)]$ , and solving for  $\varphi$ , yields the estimate  $\varphi=0.0097$ . The resulting summary of repeatability and reproducibility limits is shown in Table 30.

Because the estimate of the correlation parameter,  $\varphi$ , was somewhat smaller for the Cigarette Extinction Method, where  $\varphi=0.0097$ , compared to the Mock-Up Ignition Method, where  $\varphi=0.058$ , the values of the reproducibility limits ( $R$ ) are somewhat smaller for the Cigarette Extinction Method, Table 30, compared to the Mock-Up Ignition Method, Table 24. In contrast, the repeatability limits ( $r$ ) are exactly the same in Tables 24 and 30 because both use the same formula for  $S_r$ , as given by Equation (1) of Section II.B.8.e.

**Table 29. Observed Repeatability and Reproducibility Standard Deviations  
for Cigarette Extinction Method Interlaboratory Study  
 $m=16$  Replications per Laboratory**

<b>Cigarette I.D.</b>	<b>Substrate (No. of Layers)</b>	<b>Average Proportion of Full Length Burns</b>	<b>Repeat-ability S.D. <math>S_r</math></b>	<b>Reproduc-ibility S.D. <math>S_R</math></b>
501	3	1.000	0	0
	10	1.000	0	0
	15	1.000	0	0
503	3	1.000	0	0
	10	1.000	0	0
	15	1.000	0	0
529	3	0.569	0.124	0.119
	10	0.056	0.057	0.101
	15	0.021	0.036	0.044
530	3	0.056	0.057	0.058
	10	0.000	0	0
	15	0.000	0	0
531	3	0.993	0.021	0.021
	10	0.944	0.057	0.066
	15	0.882	0.081	0.110

**Table 30. Cigarette Extinction Method:  
Calculated Repeatability (*r*) and Reproducibility (*R*) Limits  
for Various Assumed Numbers of Replications (*m*) and Full-Length Burn Proportions (*p*)**

<b>p</b>	<b>m = 16</b>		<b>m = 32</b>		<b>m = 48</b>		<b>m = 96</b>		<b>m = 9600</b>	
	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>	<b>r</b>	<b>R</b>
0.05 or 0.95	0.15	0.16	0.11	0.12	0.09	0.11	0.06	0.09	0.006	0.06
0.10 or 0.90	0.21	0.22	0.15	0.17	0.12	0.15	0.09	0.12	0.009	0.08
0.20 or 0.80	0.28	0.30	0.20	0.23	0.16	0.20	0.11	0.16	0.011	0.11
0.30 or 0.70	0.32	0.34	0.23	0.26	0.19	0.22	0.13	0.18	0.013	0.13
0.40 or 0.60	0.34	0.37	0.24	0.28	0.20	0.24	0.14	0.19	0.014	0.14
0.50	0.35	0.37	0.25	0.28	0.20	0.24	0.14	0.20	0.014	0.14

**m:** Assumed number of replications per laboratory  
**p:** Assumed long-run proportion of full-length burns for cigarette and substrate combination under test  
**r:** Repeatability limit =  $2.8S_r$ , where  $S_r$  is calculated as in Table 24.  
**R:** Reproducibility limit =  $2.8S_R$ , where  $S_R$  is calculated as in Table 24, with  $\phi = 0.0097$

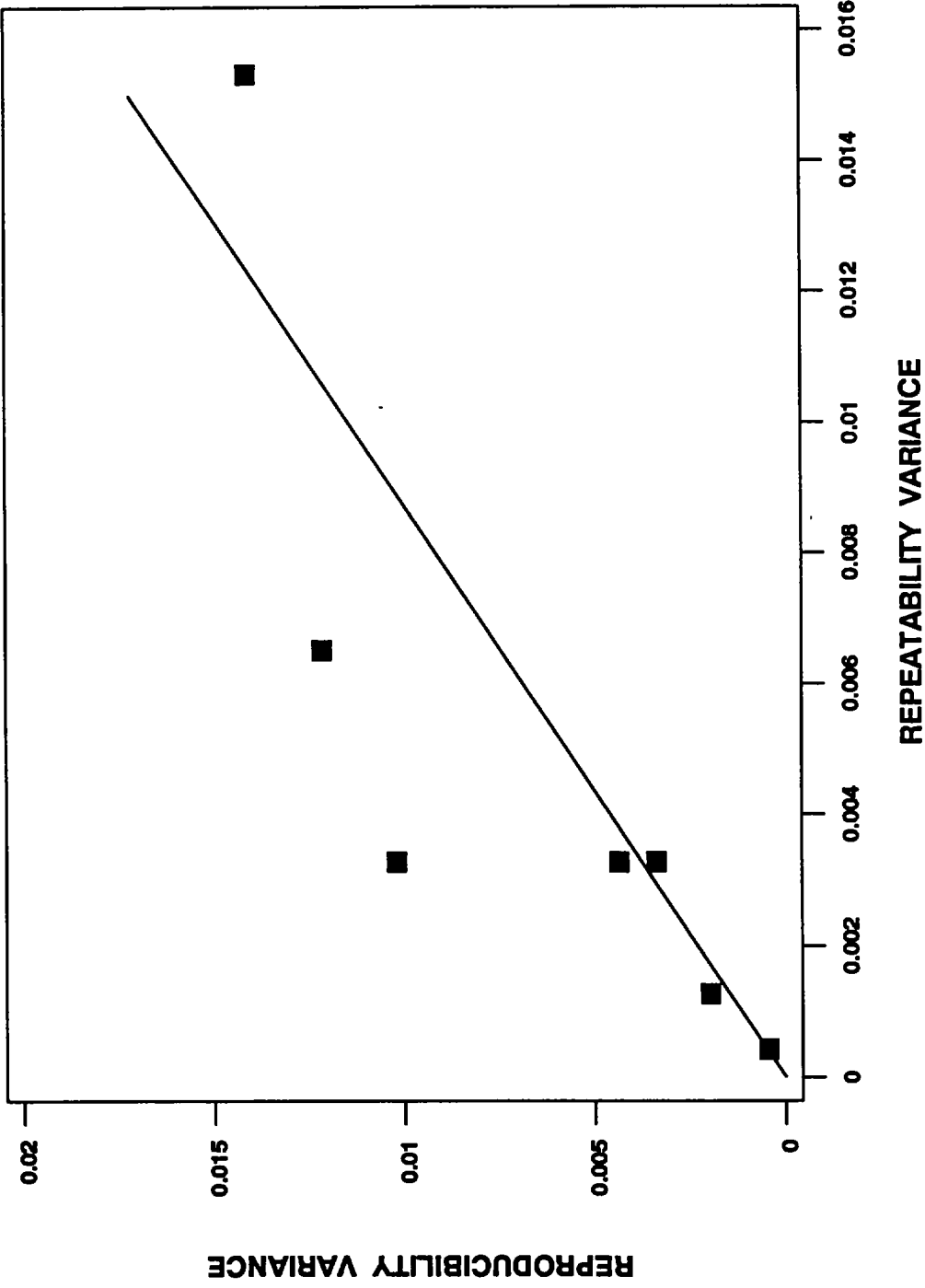


Figure 17. Empirical Relation of Reproducibility Variance,  $S_R^2$ , to Repeatability Variance,  $S_r^2$ , Based on Data in Table 29 from the Interlaboratory Study of the Cigarette Extinction Test Method. The equation of the least squares line shown is  $S_R^2 = (1.146)S_r^2$ .

### III. CONSIDERATIONS REGARDING THE USE OF THE TWO TEST METHODS

#### A. Mock-Up Ignition Test Method

The mock-up method developed here broadly meets the criteria described in Section I for an acceptable test method. It is a performance-based method that employs a cigarette/substrate combination bearing a strong (although not perfect) similarity to the real-world fire safety hazard. The relation between the test results and real upholstered chair ignition behavior is traceable through the use of cigarettes calibrated in the TSG study. The test output is quantitative and provides differentiation among cigarettes of varied ignition propensity. Through choice and control of materials it should provide a stable standard of performance for the foreseeable future.

As is generally the case with fire tests, this method has potential limitations that are a consequence of incomplete knowledge of the real-world scenarios. First, in the apparatus, the ambient atmosphere is perturbed only by the cigarette plume. This case is believed to be a highly relevant analog for real-world accidental ignitions occurring in a chair crevice. As noted previously, if further information on real-world ignitions indicates a significant fraction occurring in external air flow conditions *at the ignition location*, it may be appropriate to supplement the results of the current method with those obtained in the presence of a comparable flow. This would require a method development process comparable to that described in this report. A second limitation is the small number of upholstery substrates used to relate mock-up behavior to real-world chairs [3]. It is presumed that this correlation is representative of the aggregate furniture market. The existence of this correlation virtually assures that there will be some real-world benefit in moving toward cigarettes which perform well in this test method. Should sufficient evidence emerge in the future that a large fraction of the furniture at risk does not follow the correlation that was demonstrated in the TSG study, it may be appropriate to replace one or more of the Mock-Up Ignition Test Method substrates.

The interlaboratory study demonstrated the level of lab-to-lab reproducibility one can expect of this method. Table 24 above shows that this level cannot be made substantially greater with a very large number of replicates; conversely, this level does get significantly less desirable if the number of replicates is reduced substantially below 48. The achievable lab-to-lab reproducibility is an appropriate measure of how finely a test method can differentiate among test subjects for regulatory purposes. It is apparent, then, that the mock-up method cannot make fine distinctions in ignition propensity among cigarette designs. With 48 replicates on a given mock-up, the proportion of ignitions obtained by two separate laboratories can be expected to differ by up to about 0.4. *This places a limit on the degree of resolution possible for regulatory use of the method.* Finer distinctions than this could be made only within a single laboratory, presumably for product development purposes. In that case, a number of replicates greater than 48 would appreciably improve the differentiation; see Table 24.

Three mock-ups were included in the interlaboratory study and all were found to differ significantly in ease of ignition. The results in Figure 4 above show the response of this set of mock-ups to a broad spectrum of experimental cigarette designs, though all are of a conventional construction. Given the limitation above on the resolution of the test in differentiating ignition propensity, it is apparent that cigarettes of high ignition propensity gave effectively the same response on two out of three of the mock-ups. A cigarette of low ignition propensity also gave effectively the same response

on two (different) mock-ups. This does not mean that one mock-up can be omitted from the entire test set, because the duplication of response occurs on differing mock-up pairs with differing cigarettes. It does, however, suggest the possibility of the need for fewer replicates on at least one mock-up, and possibly on two, for some cigarettes.

Figure 4 shows that the duck #4 mock-up is, for the set of cigarettes examined, consistently harder to ignite than the other two mock-ups; and, in turn, the duck #6 mock-up is harder to ignite than the duck #10 mock-up. If a cigarette were tested first on the duck #4 mock-up and gave all ignitions in 48 replicates, it should be possible to do fewer replicates on the other two mock-ups to verify that there were no unexpected reversals in ignition behavior. This could provide significant labor savings on what is otherwise a rather labor-intensive test protocol.

Performing fewer than 48 tests will result in some loss of information, and a corresponding increase in statistical uncertainty regarding the long-run ignition rate that would be observed for those mock-ups. The (within-lab) statistical uncertainty, for any number of replications, can be quantified by use of confidence bounds on the ignition probabilities for a given cigarette on a given mock-up, as follows.

If a cigarette is tested on, *e.g.*, the duck #4 mock-up, and all 48 replications result in ignitions, then a 95% lower confidence bound on the long-run ignition probability is  $(1-0.95)^{1/48} = 0.94$ . Speaking loosely, one can be 95% confident that the ignition probability (in the same laboratory) is greater than about 0.94. With that result, it may be sufficient for some purposes to know that the ignition probability on the duck #6 and duck #10 mock-ups would be about 0.6 or higher, since the degree of interlaboratory resolution is about 0.4, as noted above. If so, then only  $n=6$  runs would be required, because  $(1-0.95)^{1/6} = 0.61$ . That is, if 6 runs were conducted resulting in 6 ignitions, then the 95% lower confidence bound for the ignition probability would be 0.61. There can be, of course, no guarantee that 6 runs on a more ignitable mock-up would *necessarily* result in 6 ignitions. If one or more non-ignitions did occur, it would be appropriate to run a full set of 48 replications for each of the three mock-ups.

Table 31 shows the relationship between the number of runs and the corresponding lower confidence bounds in the case of 100% ignitions. The table is useful for comparison and to help decide whether the increased uncertainty due to running fewer than 48 replications is acceptable for a particular purpose.

These same arguments can be made for any cigarette and mock-up combination where a 100% response, either ignitions or non-ignitions, is obtained. It is the choice of the regulator whether this trade-off is implemented in any adopted test method.

**Table 31. 95% Lower Confidence Bounds  
for the Long-Run Ignition Probability  
Assuming that  $n$  Tests Result in  $n$  Ignitions**

$n$ = Number of Runs	Lower Confidence Bound
4	0.47
8	0.69
12	0.78
24	0.88
36	0.92
48	0.94

Informal reports of in-progress cigarette industry studies imply that some upholstery fabrics will respond to contact with a lit cigarette in a substantially different manner from that seen with the cotton ducks used in this method. Even if this is verified, the possible results of employing the Mock-Up Ignition Test Method developed here are as follows:

- Some cigarette designs will produce fewer ignitions (than the current market cigarettes) both in the test and when in contact with furniture containing fabrics which behave like the cotton ducks used here. The test method in this case is a true indicator of less fire-prone cigarettes.
- Some cigarette designs will produce fewer ignitions in the test, but will not produce a reduced number of ignitions when in contact with furniture containing fabrics which is dissimilar in response to cotton ducks. It seems implausible that such designs would show greater real-world ignition propensity than do current commercial cigarettes.
- Still other cigarette designs will produce a number of ignitions, both in the test method and when in contact with furniture containing fabrics which behave like the cotton ducks used here, comparable to current commercial cigarettes. For these, the test method is again a true indicator of expected fire performance.
- Some designs will produce a number of ignitions in this test that are comparable to current commercial cigarettes, but will produce a reduced number of ignitions when in contact with furniture containing fabrics which is dissimilar in response to cotton ducks. These designs would not likely be pursued; but, if they were, they would unobtrusively reduce fire losses.

The result of the second and fourth occurrences is an uncertainty in the *degree* to which real-world fire losses are reduced.

At present there are insufficient data available to estimate what fraction of real-world furniture might contain fabrics differing substantially (*i.e.*, beyond the reproducibility of the test method) from cotton

ducks in their ignition behavior. If further data become available indicating that such fabrics are a significant fraction of the real-world population, it would be an option to supplement the results of cigarette testing using this method with results based on other carefully-chosen fabrics.

## **B. Cigarette Extinction Test Method**

An analog to most of the discussion in the preceding section applies to this method as well. The potential limitation on the imposition of an external air flow manifests itself in both methods. Since the filter paper is a surrogate material, the pertinent consideration is the degree of differentiation of cigarette ignition propensity. This is reflected in the numbers of layers selected for the test substrates. For instance, were there an interest in better discrimination among cigarettes of high ignition propensity than is shown in Figure 15, one might be inclined to select 20 or 25 layers to replace the 15 in the first substrate. However, limited data indicate that this increase has no effect on the burning behavior of cigarettes in this test series. Thus, this method is less appropriate than the mock-up method for distinguishing initial progress from current market cigarettes toward those of lower ignition propensity.

The limit on the degree of resolution for this method is similar to that using mock-ups. Table 30 shows that the level of lab-to-lab reproducibility is about 0.4 for 16 replicate tests. Only modest improvement is achievable for a reasonably larger number of tests. It is apparent that the Cigarette Extinction Test also cannot make fine distinctions in ignition propensity among cigarette designs. *Again, this places a limit on the degree of resolution possible for regulatory use of the method.* As above, finer distinctions could be made within a single laboratory by using a number of replicates greater than 16.

It is also possible to calculate how one might use fewer tests on a substrate, having measured 100% ignitions on a substrate of higher thermal capacitance. For example, after 16 full-length burns in 16 tests on the 15-layer substrate, a 95% lower confidence bound on the long-run full-length burn probability is  $(1-0.95)^{1/16} = 0.83$ . In other words, one can be 95% confident that the full-length burn probability within the same laboratory is greater than about 0.83. With that result, it may be sufficient for some purposes to know that the full-length burn probability on the 10-layer and 3-layer substrates would be about 0.6 or higher, since the degree of interlaboratory resolution is about 0.4, as noted above. If so, then only about  $n=6$  runs would be required, because  $(1-0.95)^{1/6} = 0.61$ . That is, if 6 runs were conducted resulting in 6 full-length burns, then the 95% lower confidence bound for the full-length burn probability would be 0.61. It is, of course, possible that in the 6 tests, one or more self-extinguishments might occur. It would then be appropriate to run a full set of 16 replications for each of the three substrates. Note that the savings in resources with this method is somewhat smaller than with the mock-up method.

## **C. Allowable Material Variability**

The lab-to-lab reproducibility seen in each ILS for the two test methods (Figures 4 and 14) is a result of the variability of the test operators, the laboratory environment, the substrate materials, and the products being tested. Measuring variations in the cigarettes is part of the purpose of testing. Therefore, in order to assure that the test reproducibility is maintained at the observed level, the substrate material variability limits existent in the present study must be applied to all future



materials. This may be a more stringent requirement than necessary but, without further study, there is no justification for looser controls on the materials.

For the Mock-Up Ignition Test Method, the most critical material is the fabric. Acceptable fabrics must be 100% raw cotton ducks which meet the physical requirements of reference 12.<sup>8</sup> Open-end spinning should also be specified to ensure similarity to the fabrics used in the interlaboratory studies. Since reference 12 does not explicitly specify such details as yarn count and yarn plies and the influence of these structural parameters on cigarette ignitability has not been extensively explored, the values listed in Table 6 should be adhered to. Where the limits on other properties are narrower for the cotton ducks actually used in this study, those narrower limits must apply. Thus the acceptable areal density and air permeability ranges are those given in Tables 11 and 12. Reference 12 contains no specification on metal ion content of the fabrics. The data in Table 7 for duck #6 suggest that potassium ion levels in the range from 4400 to 6000 ppm are acceptable. Comparison of the potassium levels in the various ducks in Tables 7 and 8 suggests that all of the ducks can generally be held in this range for an extended production period, though conclusions about multi-year variability obviously cannot be made on the basis of the present study. Sodium is potentially as catalytic to smoldering ignition as is potassium; thus it should (and probably naturally will) be held to the negligible levels seen in Table 7. Calcium and magnesium cations are weak smolder promoters; Table 7 suggests an acceptable range of both is 500 to 750 ppm. Cotton ducks should be stored in the dark at room temperature or below and at a humidity low enough to preclude any microbial action. Under these circumstances, shelf life should be at least one year.

As noted previously, the Mock-Up Method using duck #4 in combination with a polymer film as an added heat sink is sensitive to the properties of that film. Table 15 lists the properties of the Warp Brothers Poly-Film used in the main ILS. In reviewing the results shown in Figures 2 and 4, a film like this is preferred. The areal density is believed to be the most critical property here (along with the heat capacity of the film, which has not been measured but should be fixed by the composition). An areal density change from 0.015 to 0.012 g/cm<sup>2</sup> (Poly-America film) caused a substantial change in the ignition proportions of cigarettes 501 and 503. Cigarette 501, in particular, decreased from an ignition proportion range of 0.7-1.0 to 0-0.3 when the higher areal density film was used. This sensitivity suggests that the areal density should be held at 0.015 g/cm<sup>2</sup> ± 5 percent.

The polyurethane foam in the Mock-Up Method probably serves more of a physical role (part insulator, part heat sink, part oxygen inflow inhibitor) than a chemical role (source or sink of chemical energy). The foam does not smolder during the fabric ignition process or soon thereafter. In this study, substitution of another foam with a 25% lower density and a 17% greater air permeability had no great effect on the ignition proportions seen. Thus there should be no difficulties introduced by allowing the typical ± 5% within-batch variations in foam density and the level of air permeability variations seen in Table 14. Reference 9 indicates that there is a weak correlation between the number of urea bonds in the foam formulation and ignitability. The number of urea bonds is proportional to the water level in the foam formulation which is unknown for the foams in this study. The study reported in reference 9 also indicated that ignitability is an order of magnitude more sensitive to foam air permeability than to urea bond levels. Since these two foam properties are somewhat related (water is added as a foam blowing agent), the preceding restriction

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<sup>8</sup> Reference 12 accepts recycled cotton as raw material. That is undesirable here because of the possibility of chemical contamination which could affect ignitability by cigarettes.

on foam density variations should suffice, provided the foam is a polyether/TDI formulation typical of current technology. The foam also should contain no inorganic fillers. Polyurethane foams show substantial color changes when exposed to typical room lighting. The possibility of significant alterations via this mechanism or via slow aging should be precluded by storage under an opaque covering at room temperature for no more than six months before use.

The filter paper is the only critical material used in the Cigarette Extinction Test Method. The paper used here is Whatman No. 2 filter paper, a staple for qualitative analysis. The nominal ash content specification is 0.06%. Here again the critical property is the areal density, since the principal function of the paper is to serve as a heat sink. It is likely that the thermal conductivity also plays a role here, especially in the substrates with the greatest number of paper sheets. This should be proportional to the density of the paper. The data in Section II.C.3 are for the filter papers used in the present study. The areal density and thickness determine the paper density as well. The variability of these properties shown there is acceptable.

Appendix F includes thermogravimetric data on all of the materials used in both test methods. These data are included as general guidance in assessing the suitability of candidate new batches of material. Any new batch of a given material should behave in substantially the same manner as the example in Appendix F for that type.

It is possible that a performance specification could be developed for mock-up materials based on a standardized, non-cigarette ignition source, such as a small black body or a carbon dioxide laser. This might obviate the need for all of the prescriptive limitations on materials given above. Other work in this program has shown that the smoldering ignition process is sensitive to such variables as the size of the spot on a mock-up surface which is heated. Thus any such performance test would require careful study of the sensitivity of the output (e.g., ignition delay time for a range of peak incident heat fluxes) to test variables. Since the materials specifications above appeared to be sufficient, this alternative approach has not been pursued to completion.

#### **D. Standardization of Test Methods**

It is common practice, upon development of a fire test method for professional use, to proceed with its adoption as a voluntary consensus standard in either ASTM or the National Fire Protection Association (NFPA). Because these processes generally take several years, this is not possible under the Fire Safe Cigarette Act of 1990, which expires September 10, 1993. This report contains sufficient documentation of the two test methods and interlaboratory evaluations of each so that the methods could be submitted to ASTM or NFPA for formal approval. The relevance to fire safety is contained here and in reference 3. Thus, all necessary materials for initiating the standardization process are now available.

#### **E. Effectiveness of the Methods**

It will be the role of the regulator to determine which (if any) future cigarettes are tested, by whom, how frequently, and to what requirements. The last of these is likely to be based, in part, on the additional degree of life safety desired. The findings of the TSG demonstrated that measurements

using mock-ups are reasonable indicators of full-scale performance [2]. The work to date provides modest guidance in relating performance under these new methods to real-world performance.

There are data to "calibrate" the methods at the high end of the ignition propensity scale. The current commercial cigarettes are associated with the fire losses of today. The commercial cigarette data in Section IV of this report and the data on older commercial cigarettes in reference 3 establish typical performance for these cigarettes. In the two new test methods, this is seen as a large number of ignitions on the #4 cotton duck or full-length burning on the 15-layer paper substrate. This establishes the test results for the high ignition propensity end of the scale.

Both the current work and cigarette industry studies [20] demonstrate the performance of cigarettes that never or rarely ignited a variety of substrates. The correlation of mock-up results with chair tests in reference 3 indicates that such results can be expected to be indicative of real-world performance of such substrates. In the two new test methods, this behavior is observed as few ignitions on the #10 cotton duck or few full-length burns on 3 layers of filter paper. This indicator of test results for the low ignition propensity end of the scale is less quantitative than the high end indicator mentioned earlier.

In between these extremes, one would like to be able to predict a reduced number of fires as fewer ignitions are measured in the laboratory. The full-scale tests in reference 3 support this. At least for coarse changes in test performance, real-world savings seem highly likely. When considering smaller increments in test performance, however, one must keep in mind the accuracy limits of the methods as discussed above.

## **IV. TESTING OF COMMERCIAL CIGARETTES**

### **A. Introduction**

Having completed the development of standardized testing procedures, NIST has evaluated a sample of current commercial cigarette types, its second obligation under the Fire Safe Cigarette Act of 1990. The results of this performance testing:

- demonstrate the utility of the method for routine testing of production cigarettes,
- provide baseline data for comparison with commercial cigarettes of the future, and
- present examples of the recommended reporting format for the ignition propensity data.

### **B. Rationale for Commercial Cigarette Choices**

The cigarettes were chosen with two objectives in mind: (a) to incorporate packings which comprise a significant portion of the consumer market, and (b) to include several packings judged likely to yield a lower ignition propensity compared to the best sellers. After reviewing available physical characteristic data, fourteen packings in the former category were tested and six in the latter.

Consumer market data were obtained from the February 10, 1992 Maxwell Consumer Report. This includes complete sales data only through 1990, and it is the 1990 data which were used. The Maxwell Report data indicate that the fourteen packings chosen comprised 38% of the market in 1990.

These best selling brands do not vary widely in physical parameters such as packing density, paper permeability or tobacco rod circumference. Thus a second, smaller group of cigarettes was identified which do show more substantial deviations in their physical parameters. The particular emphasis was on cigarettes having two physical parameters which deviate in a direction which the TSG study [3] would suggest as likely to lower ignition propensity, *e.g.*, lower paper porosity, circumference, tobacco density. [Since the data on which these decisions were based were identified as confidential and proprietary by the cigarette industry, they are not tabulated in this report.] The six selected packings comprised less than one percent of the market in 1990.

Samples of these cigarettes were obtained courtesy of the Tobacco Institute Testing Laboratory (TITL), Rockville, Maryland. This laboratory is responsible for the official tar and nicotine ratings of all commercial cigarettes. It contracts to have packs of cigarettes purchased from around the country once each year, in the first quarter of the calendar year. The cigarettes tested in this study were from surplus packs purchased in the first quarter of 1992; typically the quantity obtained was 80-100 packs of each type. The cigarettes were in opened packs which had been sealed in plastic bags after TITL's sampling and stored at room temperature. They were also stored at NIST in this manner until removed for conditioning.