

Laboratory 8

Critical Trials Dust Covers

Start Date _____ End Date _____

Conditioning Room Conditions:

Temperature 68°F
Humidity 53%

Test Room Conditions:

Temperature 66°F
Humidity 45%
Barometric Pressure _____

Flame Ht 35mm Gas Pressure 0.4psig Gas Flowrate 177

Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
151	A	1	3	---	---	---	---	10*
152	A	2	5	---	---	---	---	10
153	A	3	7	---	---	---	---	11
154	A	4	8	---	---	---	---	12
155	A	5	9	---	---	---	---	9
156	A	6	10	---	---	---	---	11
157	A	7	6	---	---	---	---	14
158	A	8	2	---	---	---	---	11
159	A	9	1	---	---	---	---	11
160	A	10	4	---	---	---	---	9
161	B	1	3	0	0	0	0	
162	B	2	5	0	0	0	0	
163	B	3	7	0	0	0	0	
164	B	4	8	0	0	0	0	
165	B	5	9	0	0	0	0	
166	B	6	10	0	0	0	0	
167	B	7	6	0	0	0	0	
168	B	8	2	0	0	0	0	
169	B	9	1	0	0	0	0	
170	B	10	4	0	0	0	0	

* Flame reached edge before 20 second flame application done. Time recorded is time it took flame to reach edge from time of application

Upholstery Flammability Test Interlaboratory Study Data Sheet

Laboratory ID: #9 _____

Apparatus ID SEAT FIXTURE #5 _____

Start Date _____ End Date _____

Conditioning Room Conditions:

Temperature _____

Humidity _____

Test Room Conditions:

Temperature _____

Humidity _____

Barometric Pressure _____

Flame Ht _____ Gas Pressure _____

Gas Flowrate _____

Practice Trials

Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
P1	C							
	C							
	C							
P2	C							
	C							
	C							
P3	A							
	A							
	A							
P4	G							
	G							
	G							
P5	H							
	H							
	H							

Laboratory 9

Critical Trials

Start Date 2/11/00 End Date 2/11/00

Conditioning Room Conditions:

Temperature 70°F
Humidity 50%

Test Room Conditions:

Temperature 72°F
Humidity 50%
Barometric Pressure _____

Flame Ht 35 mm Gas Pressure 0.4 psig Gas Flowrate 45

Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
1	D	1	2	4	21	0	25	
2	D	1	2	0	0	0	0	
3	D	1	2	4	6	0	10	
4	D	2	9	0	0	0	0	
5	D	2	9	0	0	0	0	
6	D	2	9	0	0	0	0	
7	D	3	5	0	2	0	2	
8	D	3	5	>120	---	---	>120	
9	D	3	5	0	2	0	2	
10	D	4	6	3	0	0	3	
11	D	4	6	4	0	0	4	
12	D	4	6	3	0	0	3	
13	D	5	4	>120	---	---	>120	
14	D	5	4	0	0	0	0	
15	D	5	4	0	0	0	0	
16	D	6	8	30	0	>90	>120	
17	D	6	8	3	0	0	3	
18	D	6	8	3	0	0	3	
19	D	7	3	2	0	0	2	
20	D	7	3	3	0	2	5	
21	D	7	3	3	2	0	5	
22	D	8	7	4	0	0	4	
23	D	8	7	5	0	0	5	
24	D	8	7	0	0	0	0	
25	D	9	10	0	0	0	0	
26	D	9	10	0	0	0	0	
27	D	9	10	0	0	0	0	
28	D	10	1	3	0	0	3	
29	D	10	1	4	0	0	4	
30	D	10	1	0	0	0	0	

Laboratory 9

Critical Trials

Start Date 2/15/00 End Date 2/15/00

Conditioning Room Conditions:

Temperature 70°F
Humidity 50%

Test Room Conditions:

Temperature 70°F
Humidity 50%
Barometric Pressure _____

Flame Ht 35mm Gas Pressure 0.4psig Gas Flowrate 45

Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
31	I	1	2	10	15	0	25	
32	I	1	2	45	0	>75	>120	
33	I	1	2	35	0	>85	>120	
34	I	2	9	0	0	0	0	
35	I	2	9	0	0	0	0	
36	I	2	9	20	0	>100	>120	
37	I	3	5	0	0	0	0	
38	I	3	5	0	0	0	0	
39	I	3	5	0	0	0	0	
40	I	4	6	20	0	5	25	
41	I	4	6	0	31	0	31	
42	I	4	6	29	0	>91	>120	
43	I	5	4	0	10	0	10	
44	I	5	4	37	0	>83	>120	
45	I	5	4	30	0	>90	>120	
46	I	6	8	0	10	0	10	
47	I	6	8	30	0	>90	>120	
48	I	6	8	22	0	>98	>120	
49	I	7	3	0	0	0	0	
50	I	7	3	32	0	>88	>120	
51	I	7	3	27	0	>93	>120	
52	I	8	7	0	0	8	8	
53	I	8	7	30	0	0	30	
54	I	8	7	37	0	>83	>120	
55	I	9	10	0	15	0	15	
56	I	9	10	35	0	>85	>120	
57	I	9	10	20	0	0	20	
58	I	10	1	35	0	>85	>120	
59	I	10	1	27	0	>93	>120	
60	I	10	1	27	0	0	27	

Laboratory 9

Critical Trials

Start Date 2/11/00 End Date 2/11/00

Conditioning Room Conditions:

Temperature 70°F
Humidity 50%

Test Room Conditions:

Temperature 75°F
Humidity 55%
Barometric Pressure _____

Flame Ht 35mm Gas Pressure 0.4psig Gas Flowrate 45

Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
61	B	1	2	>120	---	---	>120	
62	B	1	2	3	0	0	3	
63	B	1	2	3	0	0	3	
64	B	2	9	0	0	3	3	
65	B	2	9	>120	---	---	>120	
66	B	2	9	0	0	3	3	
67	B	3	5	0	0	3	3	
68	B	3	5	0	0	3	3	
69	B	3	5	0	0	3	3	
70	B	4	6	0	0	3	3	
71	B	4	6	0	0	3	3	
72	B	4	6	0	0	3	3	
73	B	5	4	0	0	4	4	
74	B	5	4	0	0	4	4	
75	B	5	4	0	0	3	3	
76	B	6	8	0	0	3	3	
77	B	6	8	0	0	3	3	
78	B	6	8	0	0	2	2	
79	B	7	3	0	0	2	2	
80	B	7	3	0	0	2	2	
81	B	7	3	0	0	2	2	
82	B	8	7	0	0	2	2	
83	B	8	7	0	0	2	2	
84	B	8	7	0	0	2	2	
85	B	9	10	0	0	2	2	
86	B	9	10	0	0	0	0	
87	B	9	10	0	0	0	0	
88	B	10	1	0	0	0	0	
89	B	10	1	0	0	0	0	
90	B	10	1	0	0	0	0	

Laboratory 9

Critical Trials

Start Date 2/14/00 End Date 2/15/00

Conditioning Room Conditions:

Temperature 70°F
Humidity 50%

Test Room Conditions:

Temperature 72°F
Humidity 50%
Barometric Pressure _____

Flame Ht 35mm Gas Pressure 0.4 psig Gas Flowrate 45

Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
91	F	1	2	>20	---	---	>20	*
92	F	1	2	>20	---	---	>20	*
93	F	1	2	>20	---	---	>20	*
94	F	2	9	>20	---	---	>20	*
95	F	2	9	>20	---	---	>20	*
96	F	2	9	>20	---	---	>20	*
97	F	3	5	>20	---	---	>20	*
98	F	3	5	>20	---	---	>20	*
99	F	3	5	>20	---	---	>20	*
100	F	4	6	>20	---	---	>20	*
101	F	4	6	>20	---	---	>20	*
102	F	4	6	>20	---	---	>20	*
103	F	5	4	>20	---	---	>20	*
104	F	5	4	>20	---	---	>20	*
105	F	5	4	>20	---	---	>20	*
106	F	6	8	>20	---	---	>20	*
107	F	6	8	>20	---	---	>20	*
108	F	6	8	>20	---	---	>20	*
109	F	7	3	>20	---	---	>20	*
110	F	7	3	>20	---	---	>20	*
111	F	7	3	>20	---	---	>20	*
112	F	8	7	>20	---	---	>20	*
113	F	8	7	>20	---	---	>20	*
114	F	8	7	>20	---	---	>20	*
115	F	9	10	>20	---	---	>20	*
116	F	9	10	>20	---	---	>20	*
117	F	9	10	>20	---	---	>20	*
118	F	10	1	>20	---	---	>20	*
119	F	10	1	>20	---	---	>20	*
120	F	10	1	>20	---	---	>20	*

* reached top of mockup

Laboratory 9

Critical Trials

Start Date 2/14/00 End Date 2/14/00

Conditioning Room Conditions:
 Temperature 70°F
 Humidity 50%

Test Room Conditions:
 Temperature 73°F
 Humidity 50%
 Barometric Pressure _____

Flame Ht 35mm Gas Pressure 0.4psig Gas Flowrate 45

Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
121	E	1	2	0	0	10	10	
122	E	1	2	7	0	10	17	
123	E	1	2	8	0	12	20	
124	E	2	9	3	0	15	18	
125	E	2	9	0	0	15	15	
126	E	2	9	5	0	15	20	
127	E	3	5	4	0	5	9	
128	E	3	5	6	0	14	20	
129	E	3	5	5	0	20	25	
130	E	4	6	6	>114	---	>120	
131	E	4	6	0	0	10	10	
132	E	4	6	6	0	20	26	
133	E	5	4	0	0	15	15	
134	E	5	4	0	0	10	10	
135	E	5	4	6	0	18	24	
136	E	6	8	0	0	14	14	
137	E	6	8	2	0	10	12	
138	E	6	8	6	0	11	17	
139	E	7	3	0	0	8	8	
140	E	7	3	0	0	10	10	
141	E	7	3	0	0	10	10	
142	E	8	7	0	0	10	10	
143	E	8	7	8	0	12	20	
144	E	8	7	4	0	10	14	
145	E	9	10	5	0	12	17	
146	E	9	10	4	0	11	15	
147	E	9	10	8	0	12	20	
148	E	10	1	0	0	10	10	
149	E	10	1	4	0	10	14	
150	E	10	1	3	0	7	10	

Laboratory 9

Critical Trials Dust Covers

Start Date 2/16/00 End Date 2/6/00

Conditioning Room Conditions:
 Temperature 70°F
 Humidity 50%

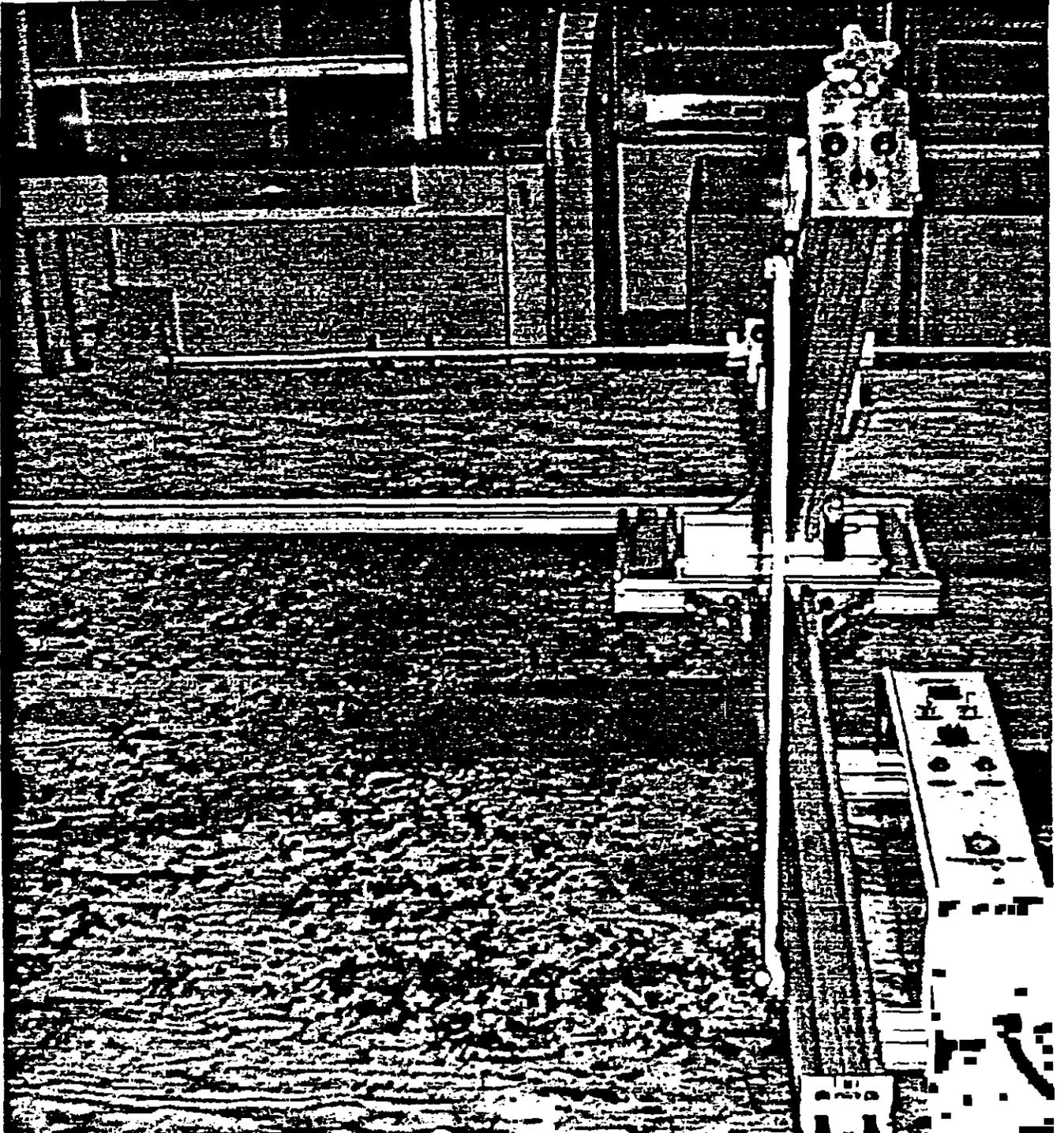
Test Room Conditions:
 Temperature 70°F
 Humidity 50%
 Barometric Pressure _____

Flame Ht 35mm Gas Pressure 0.4psig Gas Flowrate 45

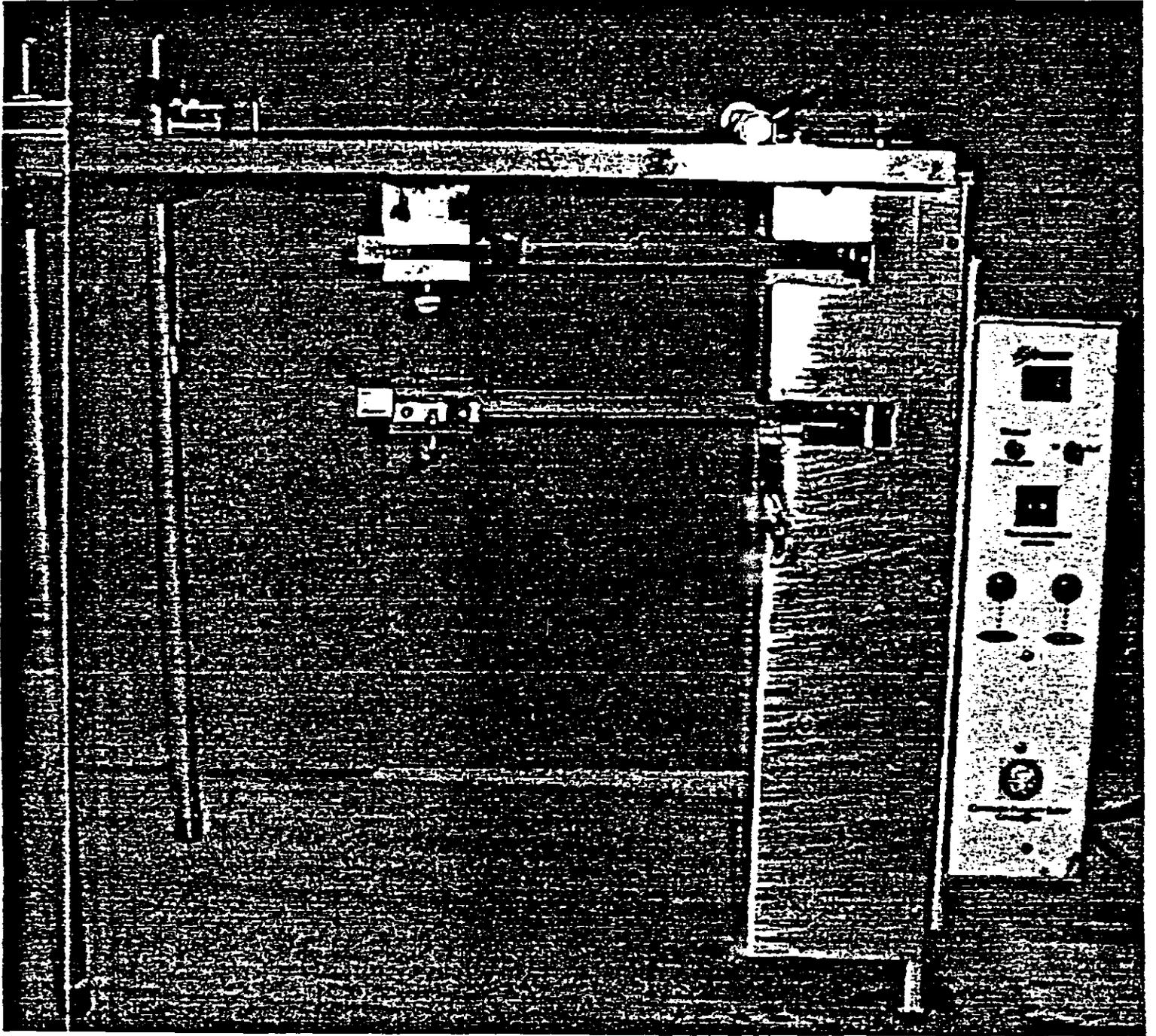
Test Order	Fabric Type	Block No.	Specimen No.	After Flame Time (sec)	After Glow Time (sec)	Smolder Time (sec)	Combustion Time (sec)	Special Notes
151	A	1	2	>120	---	---	>120	*
152	A	2	9	>120	---	---	>120	*
153	A	3	5	>120	---	---	>120	*
154	A	4	6	>120	---	---	>120	*
155	A	5	4	>120	---	---	>120	*
156	A	6	8	>120	---	---	>120	*
157	A	7	3	>120	---	---	>120	*
158	A	8	7	>120	---	---	>120	*
159	A	9	10	>120	---	---	>120	*
160	A	10	1	>120	---	---	>120	*
161	B	1	2	0	0	0	0	
162	B	2	9	0	0	0	0	
163	B	3	5	0	0	0	0	
164	B	4	6	0	0	0	0	
165	B	5	4	0	0	0	0	
166	B	6	8	0	0	0	0	
167	B	7	3	0	0	0	0	
168	B	8	7	0	0	0	0	
169	B	9	10	0	0	0	0	
170	B	10	1	0	0	0	0	

* Failure, completely burned

Attachment D



Seat Cover Fixture



Dust Cover Fixture



United States
CONSUMER PRODUCT SAFETY COMMISSION
Washington, D.C. 20207

MEMORANDUM

DATE: 28 SEP 2000

TO: Dale Ray, ECPA, Project Manager

THRU: Susan Ahmed, Ph.D., Associate Executive Director
Directorate for Epidemiology *sa*

Russell Roegner, Ph.D., Director *RR*
Division of Hazard Analysis

FROM: C. Craig Morris, Ph.D., Mathematical Statistician *ZCM*
Division of Hazard Analysis

SUBJECT: Interlaboratory Study of CPSC Draft Upholstered Furniture Small
Open Flame Test Method: Statement of Precision

Please find attached an analysis of the results of the recently completed interlaboratory study assessing the precision of the CPSC staff's draft upholstered furniture small open flame test method.

425

CPSA 6 (b)(1) Cleared
[Signature]
No Mills/Privilebs or
Products Identified

Excepted by _____

Firms Notified,
Comments Processed.



[Redacted]

Interlaboratory Study of CPSC Draft Upholstered Furniture Small Open Flame Test Method: Statement of Precision

[Redacted]

October 2000

C. Craig Morris, Ph.D.
U.S. Consumer Product Safety Commission
Directorate for Epidemiology
Division of Hazard Analysis
4330 East West Highway
Bethesda, MD 20814

426

CPSA 6 (b)(1) Cleared
9/29/00
No Mills/Priv/birs or
Products Identified
Excepted by _____
Firms Notified.

Executive Summary

CPSC staff developed a draft flammability standard and test method for upholstered furniture, designed a flammability test apparatus as part of the draft standard, and coordinated an interlaboratory study to assess the precision of the draft flammability test method. The draft test method involves application of a 20-second flame to specimens of fabric and observation of fabric combustion time following removal of the flame, with combustion times less than 120 seconds considered passing and times of 120 seconds or more considered failing.

Summary of Statistical Findings

- The 9 participating laboratories exhibited *consistent* fabric combustion time distributions (including means and variances) across the 5 fabric types tested in the study, satisfying this essential characteristic of the ASTM E691-92 guidelines for an acceptable test standard.
- The combustion times for 4 of the 5 fabrics exhibited considerable *within-laboratory variability and skew*. Within-laboratory dispersion and skew is undesirable in a test method, but appears to be an unavoidable property of the fabric combustion process.
- For both continuous and pass-fail combustion time data, reproducibility standard deviations were only slightly larger than repeatability standard deviations, indicating little between-laboratory variation. For fabrics with a very small (or very large) expected proportion of failures, both repeatability and reproducibility standard deviations are very small, satisfying this essential characteristic of the ASTM E691-92 guidelines for acceptable *precision* in a test method.

Conclusion

Results of the interlaboratory study indicated both adequate *consistency* across laboratories and *precision* within- and between-laboratories, thus satisfying these essential characteristics of the ASTM E691-92 guidelines for an acceptable test method.

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Interlaboratory Study of CPSC Draft Upholstered Furniture Small Open Flame Test Method: Statement of Precision

CPSC staff have developed a draft flammability standard and test method for upholstered furniture and designed a flammability test apparatus as part of the draft standard. Prototypes of the apparatus have been constructed and tested by CPSC staff and industry groups. CPSC staff have also coordinated an interlaboratory study to assess the precision of the draft flammability test method. The present document presents an analysis of the results of the study that is consistent with ASTM E691-92 (*Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*) and ASTM E177-90a (*Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods*).

General Procedure

The CPSC staff's draft standard requires testing the flammability of upholstery specimens as follows. First, a specimen of the seating area is assembled with a standard polyurethane foam filling material into a *mockup*. Next, a butane-gas flame of specified characteristics is applied for 20 seconds and then removed, whereupon a clock is started. The time elapsing from removal of the butane flame to the cessation of all forms of combustion (including flaming, glowing, and smoldering) is recorded. This elapsed time is referred to as *total combustion time*. If the total combustion time exceeds 2 minutes on a test trial, or if flames must be extinguished before 2 minutes have elapsed, then the combustion time for that test trial is recorded as 2 minutes.

Due to uncontrollable sources of variability inherent in test procedures, fabrics, and physical factors in the combustion process, multiple tests on randomly selected specimens of fabric are required to draw statistically valid conclusions about the average combustion time and/or failure rate for a given lot of fabric.

To assess the precision of the draft test method per ASTM E691-92 guidelines, CPSC staff circulated 4 test apparatuses among the 9 participating test laboratories (including CPSC's laboratory) and supplied each of the laboratories with 5 fabric test units (1 unit per fabric) and written directions for conducting the test. Each laboratory conducted 3 consecutive tests on each of 10 independent fabric specimens (mockups) for each of the 5 fabric types. Thus, each laboratory ran a total of $3 \times 10 \times 5 = 150$ critical test trials. Before starting the 150 critical trials, each laboratory conducted 3 consecutive tests on each of 2 mockups of another fabric type as "warm-up" practice trials. For logistical reasons, each laboratory completed all tests on a given fabric before testing the next fabric. To ensure consistency across the 9 laboratories, the 5 different fabric types (i.e., fabric test units) were tested in the same order by the 9 laboratories.

Randomized Blocks Assignment of Fabric Specimens to Test Units

ASTM E456-96 guidelines define a test unit as "the total quantity of material (containing one or more test specimens) needed to obtain a test result as specified in the test method." For each of the 5 fabric types in the present study, 9 test units were required (1 for each of 9 laboratories), and 1 additional unit was needed for supplementary tests at the CPSC laboratory. Thus, 10 test units of each fabric type were required. Each test unit for a given fabric type consisted of 10 specimens.

It was desirable that test units be as uniform as possible across laboratories. Therefore, for each fabric type, the following randomized blocks method was used to form 10 homogeneous test units. First, starting at the beginning of the first available roll of a specified fabric type, a "block" of the fabric consisting of enough fabric for exactly 10 specimens was defined. The first block was identified as block number 1. Second, the block was uniformly sectioned into 10 specimens of appropriate dimensions as specified in the standard, and the 10 specimens were identified by systematically numbering them from 1 to 10 (e.g., left to right, starting at top if 2 rows). Third, each of the 10 specimens in the block was randomly assigned to a different one of the 10 test units. After the first block was completed, the second block (block number 2) and subsequent blocks were defined similarly. The 10 specimens in each block were independently and randomly assigned to a different one of the 10 test units. This procedure ensured, for each fabric type, that each of the 10 test units received exactly 1 randomly selected specimen from each of 10 contiguous "blocks" of the fabric.

Results

Data Preparation

Since each of 9 laboratories ran 3 consecutive test trials on each of 10 specimens from each of 5 different fabrics, the data consisted of 1,350 individual *observations*. Observations for the 3 consecutive test trials on each specimen of a given fabric type by a given laboratory were averaged to obtain 1 *test determination* per specimen. Averaging the 3 consecutive observations per specimen yielded 450 test determinations. For 2 of the 450 test determinations, only 2 observations were averaged due to failure of the apparatus to retract the butane flame after the 20-sec flame application time; the number of analyzable observations was thus actually 1,348. Precision statistics were computed for each of the 5 types of fabric using the 90 test determinations on each fabric.

Combustion Time Observation and Test Determination Distributions

Figure 1 gives the observation (left panel) and test determination (right panel) frequency distributions for each of the 5 fabrics. Each observation distribution is based on 270 observations, and each test determination distribution is based on 90 observations. None of the test determination distributions exhibited the "bell-curve" *normal distribution* assumed in the ASTM 691-92 standard practice, so probability statements (*p* values and confidence intervals) associated with the precision statistics are not exact. As shown in the left panels of Figure 1, with the exception of fabric F, which failed every one of the 270 test trials, the combustion time observation distributions tended to be either bimodal or positively skewed, with observations clustered in the leftmost and rightmost portions of the range from 0 to 120 seconds. As shown in the test determination distributions in the right panels of Figure 1, averaging observations attenuated the skewing, but did not eliminate it.

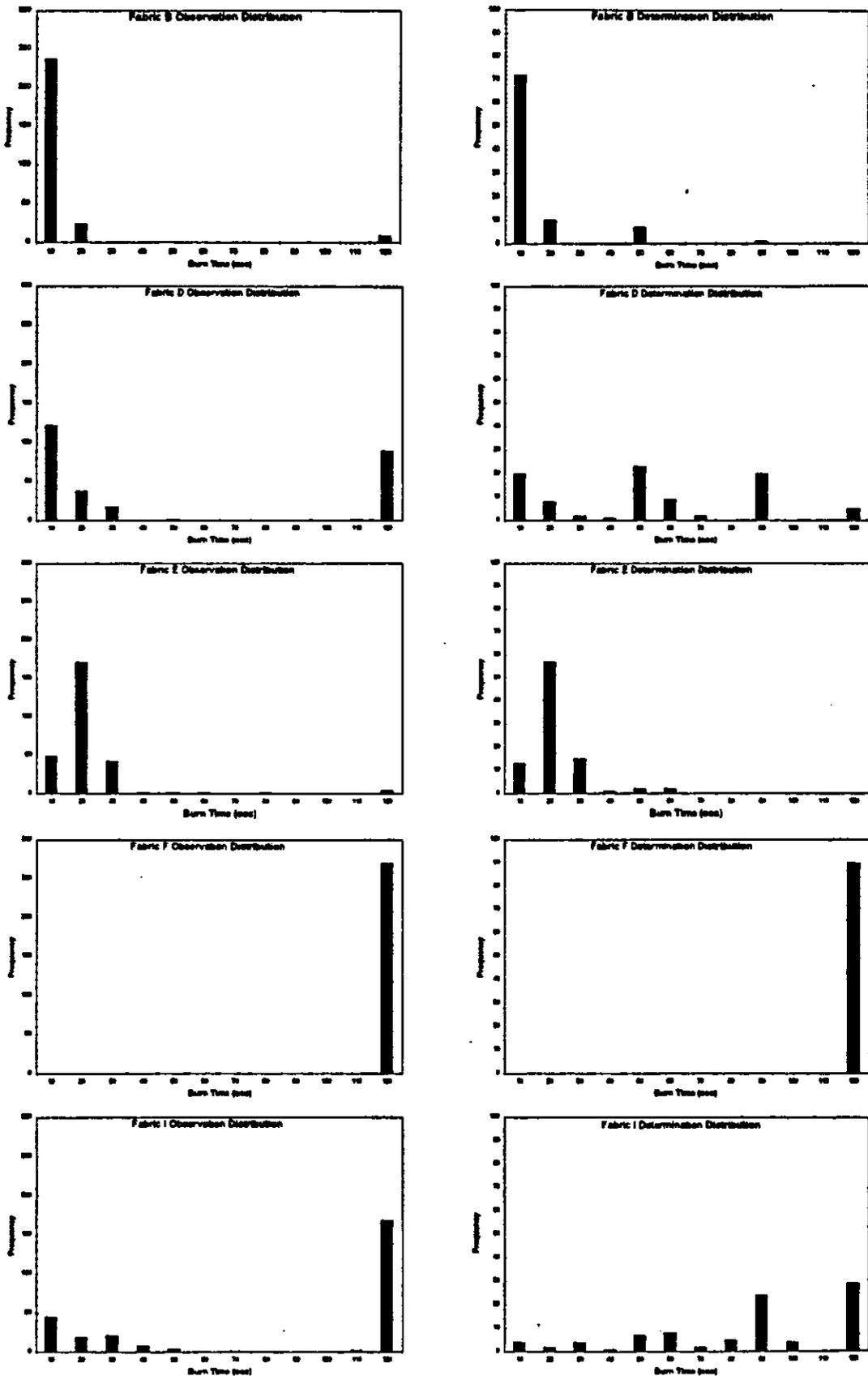


Figure 1. Observation (left) and test determination (right) frequency distributions by fabric type.

Figure 2 gives box-and-whisker ("box") plots for each fabric-laboratory combination. Each box plot depicts the 0th, 25th, 50th, 75th, and 100th percentile in the distribution. The p^{th} percentile is the score which has $p\%$ of scores in the entire distribution less than or equal to that value. The 50th percentile is the score with 50% of the observations below it; the 50th percentile is also called the *median*. The solid circle in each box depicts the median of the 10 test determination scores in that fabric-laboratory combination. The 25th and 75th percentiles are the scores with 25% and 75%, respectively, of the scores below them. The bottom and top of each box in Figure 2 depict the 25th and 75th percentile, respectively, of the 10 test determination scores in that fabric-laboratory combination. The 0th and 100th percentiles are the scores with none or all, respectively, of the scores below them. The vertical lines extending from each box are "whiskers," the ends of which represent the minimum (0th percentile) and maximum (100th percentile) test determination scores in that fabric-laboratory combination. A box plot concisely describes the *central tendency* (location on the real number line), *dispersion* (variability), and *skew* (asymmetry) of a set of test results.

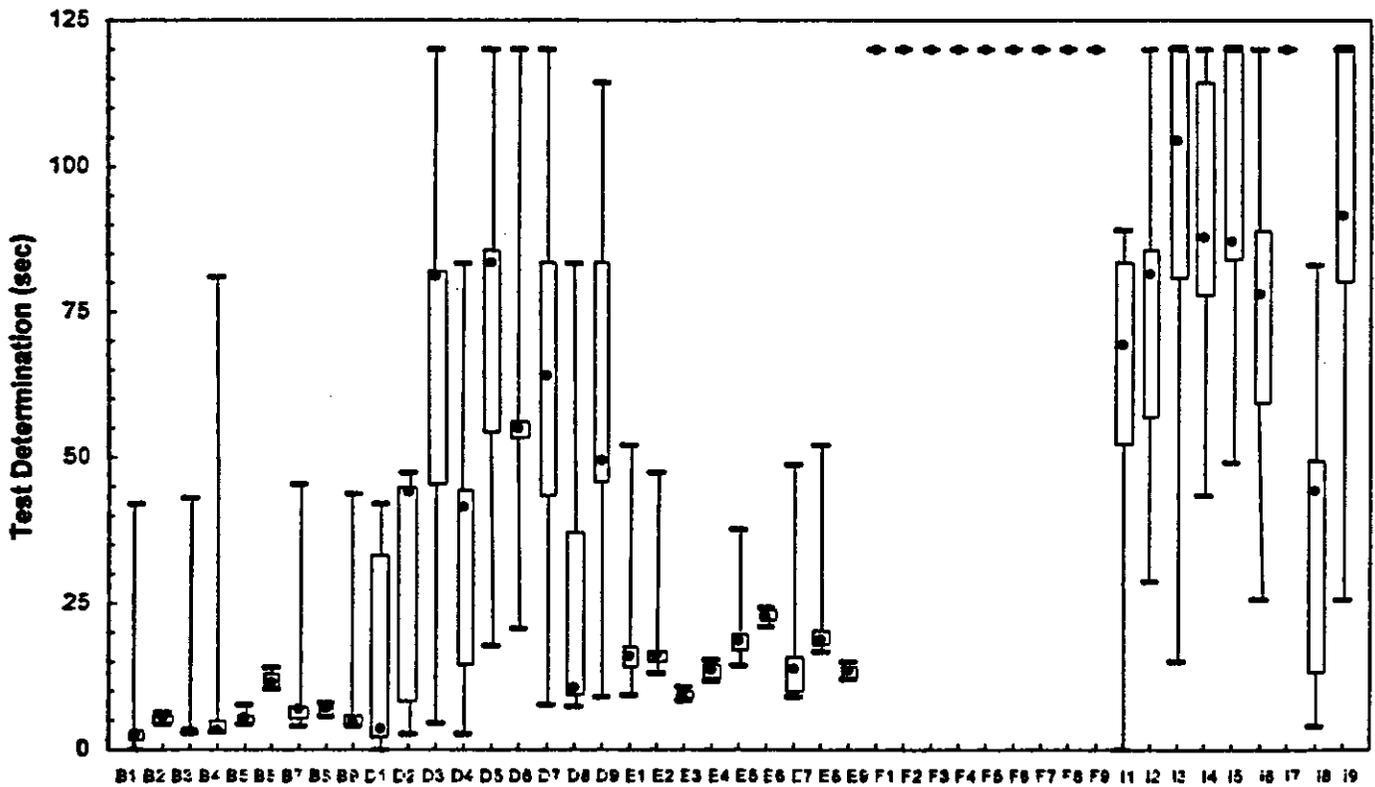


Figure 2. Box-and-whisker plots by fabric (B,D,E,F,I) and laboratory (1-9).

Figure 2 shows that laboratories tended to perform consistently on fabrics B, E, and F, although perfect consistency on fabric F is merely due to that fabric's ignition and failure on every test trial in all laboratories. For fabrics B and E, medians and other percentiles were close across the 9 laboratories. For fabrics D and I, on the other hand, medians and other percentiles tended to differ from one laboratory to the next. The dispersion of test results as measured by either the *range* (difference between the 100th and 0th percentiles) or *interquartile range* (difference between the 75th and 25th percentiles) was considerably less for fabrics B and E than for fabrics D and I.

Consistency Statistics

ASTM E691-92 prescribes statistical analyses of the consistency of results from the various laboratories participating in the interlaboratory study. Separate statistics and graphical analyses are prescribed to assess the consistency of within-laboratory variances and between-laboratory means. Within-laboratory consistency of test determination scores is assessed by computation and graphical display of so-called k statistics for each combination of fabric and laboratory, where

$k_j = S_j/S_r$, the within-laboratory consistency statistic for laboratory j ,

$S_j = (\sum(X-M_j)^2/9)^{1/2}$, the standard deviation of the 10 test determinations for laboratory j ,

$M_j = \sum X/10$, the mean of the $n=10$ test determination scores for laboratory j , and

$S_r = (\sum S_j^2/9)^{1/2}$, the *repeatability* standard deviation for the $p=9$ laboratories.

For each laboratory j , k_j measures the ratio of that laboratory's standard deviation to the repeatability standard deviation, which is a pooled estimate of the standard deviation for all laboratories. Figure 3 below gives k statistics for each combination of 9 laboratories and 5 fabrics in this study. As prescribed by ASTM 691-92, the critical value of $k = 1.56$ for the 0.5% significance level is depicted by a horizontal line in Figure 3. Data with k statistics exceeding this criterion must be scrutinized.

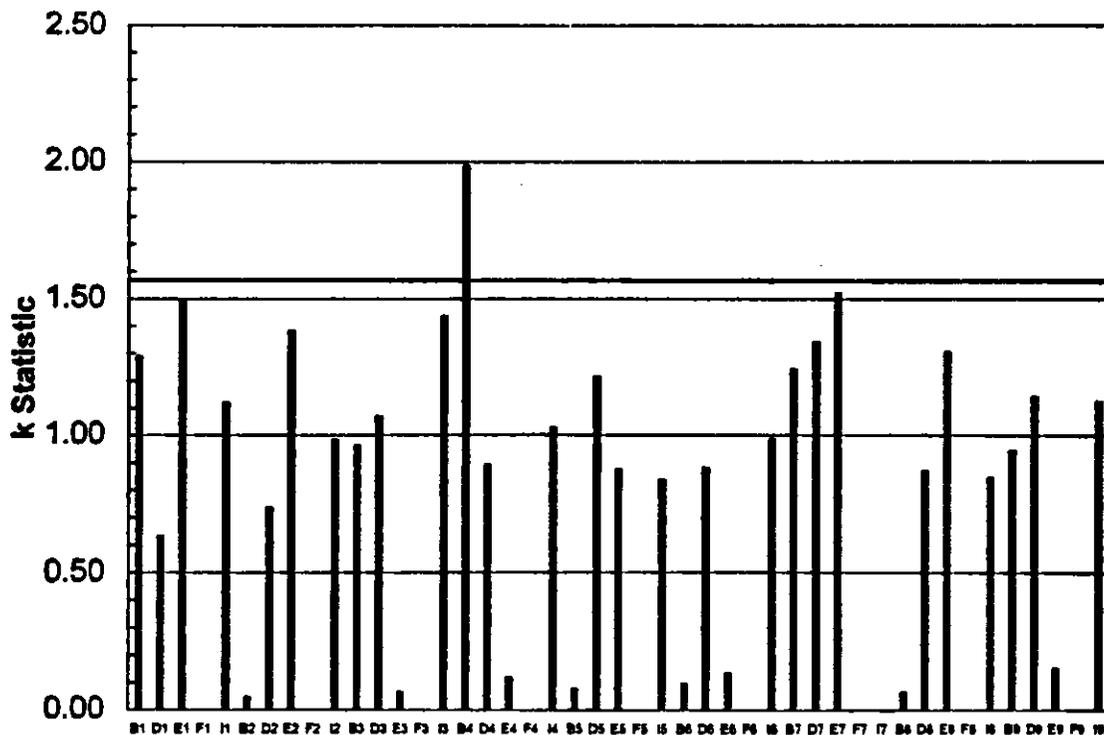


Figure 3. Within-laboratory consistency (k) statistics by fabric (B,D,E,F,I) and laboratory (1-9).

The critical k value of 1.56 was exceeded only by laboratory 4 on fabric B. The standard deviation of laboratory 4 on fabric B was 25.96, whereas the standard deviations of the remaining 8 laboratories ranged from 0.61 to 16.85. The large range of standard deviations across laboratories on fabric B is due to a few failures (combustion time scores of 120 sec) mixed with a preponderance of short, passing combustion time observations (see the top left and top right panels of Figure 1). The skewed distribution of combustion time observations for fabric B probably accounts for the large standard deviation of test determination scores observed in laboratory 4 on fabric B. Laboratory 4 did not exhibit high standard deviations as compared to other laboratories on any other fabrics. None of the other laboratories exceeded the acceptable k limit of 1.56 on any fabric, thus satisfying this essential characteristic of the ASTM E691-92 guidelines for an acceptable test standard.

Between-laboratory consistency of test determination scores is assessed by computation and graphical display of so-called h statistics for each combination of fabric and laboratory, where

$h_j = d_j/S_M$, the between-laboratory consistency statistic for laboratory j ,

$d_j = M_j - \Sigma M/9$, the deviation of laboratory j 's mean from the mean of all laboratory means,

$M_j = \Sigma X/10$, the mean of the 10 test determination scores for laboratory j , and

$S_M = (\Sigma(M_j - (\Sigma M/9))^2/8)^{1/2}$, the standard deviation of the 9 laboratory means.

Figure 4 gives the h statistics for the 9 laboratories and 5 fabrics in this study. The critical values of $h = \pm 2.23$ for the 0.5% significance level are depicted by horizontal lines in Figure 4. Data from

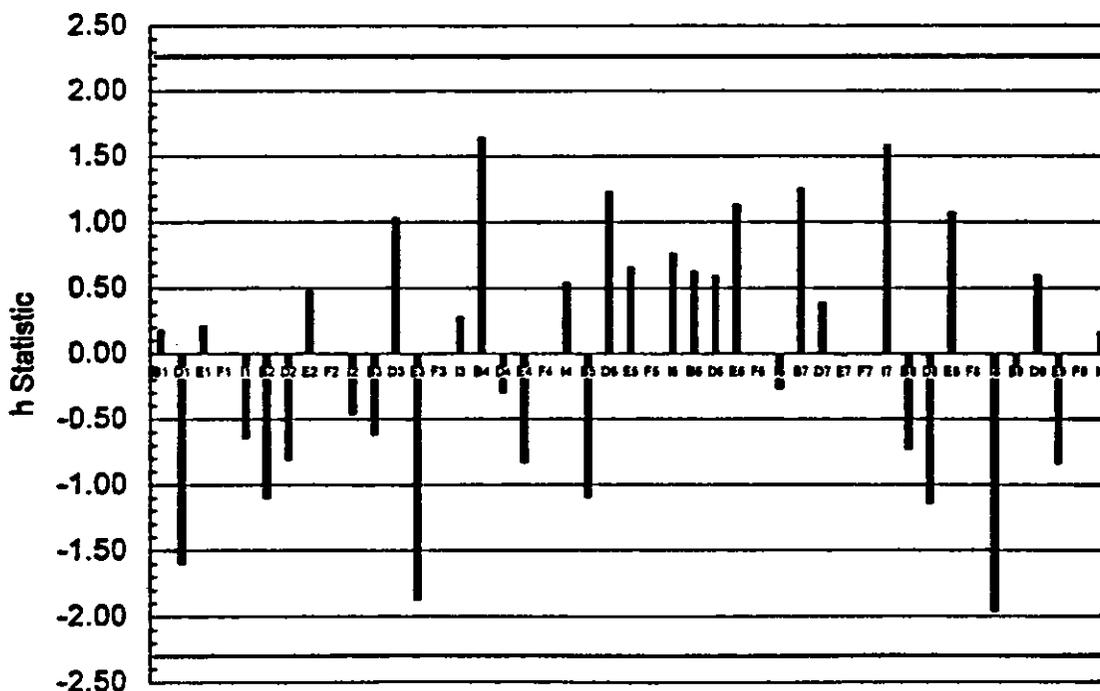


Figure 4. Between-laboratory consistency (h) statistics by fabric (B,D,E,F,I) and laboratory (1-9).

laboratories with h statistics falling outside the ± 2.23 criteria must be scrutinized, but none of the data in Figure 4 reached these criteria, satisfying this essential characteristic of the ASTM E691-92 guidelines for an acceptable test standard.

Precision Statistics

A major purpose of an interlaboratory study is to make a statement about the *precision* of results to be expected in laboratories conducting testing in accordance with a given test standard. Precision refers to the degree to which similar results are obtained under similar conditions. ASTM E691-92 distinguishes two measures of precision, *repeatability* and *reproducibility*, which are viewed as “two practical extremes of precision” (p. 491). Repeatability is defined as the variability within a given laboratory using the same well-trained operator under well-controlled conditions. Reproducibility is defined as the variability between various laboratories, each with its own well-trained operator, attempting to conduct tests under as similar conditions as possible. Factors likely to differ among laboratories include operator, equipment, calibration, and environment (temperature, humidity, barometric pressure, etc.). Each measure of precision is estimated by a formula for the standard deviation designed to measure that particular form of precision. In this report, precision statistics are presented both for the continuous combustion time data described above and for pass-fail data whereby each combustion time observation is classified *pass* or *fail* depending on whether the total combustion time reached 2 minutes.

Continuous Combustion Time Data

For a continuous variable such as the combustion time measure in the present study, the repeatability standard deviation is given by the formula

$$S_r = (\sum S^2/9)^{1/2},$$

and the reproducibility standard deviation is given by the formula

$$S_R = \text{maximum} \{S_r, [S_M^2 + S_r^2(9/10)]^{1/2}\},$$

where S_M is the standard deviation of the 9 laboratory means as defined above.

The *repeatability* and *reproducibility limits* are derived from the repeatability and reproducibility standard deviations as follows:

$$r = 2.8 S_r, \text{ the repeatability limit, and}$$

$$R = 2.8 S_R, \text{ the reproducibility limit.}$$

The latter measures provide 95% confidence intervals for the difference between 2 test determinations randomly taken under either repeatability or reproducibility conditions, respectively, where test determinations are assumed to be normally distributed with the same variance under repeatability conditions. Figures 1 (right panels) and 2 show that the distributions observed in this study are

bimodal and positively skewed, and thus not normal distributions, so the 95% confidence intervals defined by the repeatability and reproducibility limits are rough approximations in this case.

Table 1 below gives precision statistics for the continuous combustion time data collected for 9 laboratories and 5 fabrics in the interlaboratory study. Means and standard deviations of laboratory means (S_M) are included for reference. Data for fabric F are included in Table 1 for completeness, although the fact that fabric F failed all 270 tests ($3 \times 10 \times 9 = 270$ total observations) across all 9 laboratories renders that fabric meaningless for estimating precision in the interlaboratory study. The repeatability and reproducibility standard deviations do not covary with test determination means; each measure is of comparable magnitude for fabrics B and E and for fabrics D and I. For each fabric, reproducibility standard deviations (S_R) are only slightly larger than the repeatability standard deviations (S_r), indicating little between-laboratory variation in the combustion time data.

The ratios of the reproducibility limit R to the repeatability limit r for each fabric, given in the last column of Table 1, range from 1.00 to 1.19. These ratios are comparable to the ratios for other fire test methods used to regulate materials, including ratios ranging from 1.1 to 1.6 in ASTM E648 (*Standard Method for Critical Radiant Flux of Floor Covering Systems*), ratios ranging from 1.2 to 4.0 in ASTM E662 (*Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials*), and a ratio of 1.8 in ASTM E1354 (*Standard Test Method for Heat and Visible Smoke Release for Materials and Products Using an Oxygen Consumption Calorimeter*) (cited on p. 70 in [2]).

Table 1. Precision Statistics on Combustion Time (seconds) for 9 Laboratories and 5 Fabrics.

Fabric	Mean	S_M	S_r	S_R	r	R	R/r
B	9.46	3.58	13.06	13.06	36.57	36.57	1.00
E	17.59	4.47	8.02	8.82	22.45	24.70	1.10
D	48.72	21.21	29.16	34.86	81.66	97.61	1.20
I	85.94	21.53	30.01	35.69	84.03	99.94	1.19
F	120.00	0.00	0.00	0.00	0.00	0.00	—

Note: $n = 10$ test determinations per laboratory per fabric, with each test determination defined as the average of 3 observations on the same randomly sampled specimen from a test unit of the fabric.

Pass-Fail Combustion Time Data

If any of the 3 ignition tests on a given specimen of fabric in a given laboratory exhibited signs of combustion after 2 minutes, then that specimen *failed* the combustion test; otherwise, the specimen *passed* the test. (In 2 instances where only 2 ignition tests were performed on a specimen, the specimen failed if any of the 2 combustion times exceeded 2 minutes.) The proportion of failures observed out of 10 tested specimens was recorded for each of the 5 fabrics and 9 laboratories in the study, yielding 45 proportions for analysis. Although published ASTM practices do not provide guidance on estimating precision for pass-fail variables, methods for estimating repeatability and reproducibility for pass-fail variables have been published and applied elsewhere [1, 2].

For a pass-fail variable as in the present test-method study, the repeatability standard deviation is given by the formula

$$S_r = (M_p(1-M_p)/10)^{1/2},$$

and the reproducibility standard deviation is given by the formula

$$S_R = [(\sum(p_i - M_p)^2)/8]^{1/2},$$

where p_i is the proportion of failures observed in laboratory i , and M_p is the mean of the 9 laboratory proportions of failures. Although the estimate S_r may exceed the estimate S_R due to sampling error, the repeatability parameter estimated by S_r cannot exceed the reproducibility parameter estimated by S_R ; therefore, as with the continuous variable case, when S_R is less than S_r , S_R is set equal to S_r .

Finally, the *repeatability* and *reproducibility limits* are derived as in the continuous variable case by multiplying the repeatability and reproducibility standard deviations by 2.8.

Table 2 gives precision statistics for the proportion of specimen failures for the 9 laboratories and 5 fabrics in the interlaboratory study. As shown in Table 2, except for Fabric D with a mean proportion failing of .68, the reproducibility standard deviations (S_R) are only slightly larger than the repeatability standard deviations (S_r), indicating little between-laboratory variation in the pass-fail data. The larger repeatability and reproducibility standard deviations for fabric D can be explained as follows. The formula for S_r above shows that, for a given specimen sample size (10 in the present study), the repeatability standard deviation is maximal for a mean proportion failing of .5 and decreases symmetrically as the mean proportion approaches 0 or 1. Thus, for fabrics with a mean proportion near 0 or 1, such as fabrics B, E, I, and F, the repeatability standard deviation is less than for fabrics with a mean proportion near .5, such as fabric D. Since the reproducibility standard deviation must be at least as large as the repeatability standard deviation, the large repeatability standard deviation for fabric D implies an equally large, if not larger, reproducibility standard deviation.

Table 2. Precision Statistics on Proportion of Specimen Failures for 9 Laboratories and 5 Fabrics.

Fabric	Mean	S_r	S_R	r	R	R/r
B	.09	.09	.09	.25	.26	1.03
E	.04	.07	.07	.18	.18	1.00
D	.68	.15	.24	.41	.67	1.61
I	.90	.09	.12	.27	.34	1.29
F	1.00	.00	.00	.00	.00	—

Note: $n = 10$ test determinations per laboratory per fabric, with each test determination defined as 1 if any of 3 observations on the same randomly sampled specimen from a test unit of the fabric failed and 0 otherwise.

The ratios of the reproducibility limit R to the repeatability limit r for each fabric, given in the last column of Table 2, range from 1.00 to 1.61. These ratios are comparable to the ratios for other fire test methods used to regulate materials, including ratios ranging from 1.1 to 1.6 in ASTM E648 (*Standard Method for Critical Radiant Flux of Floor Covering Systems*), ratios ranging from 1.2 to 4.0 in ASTM E662 (*Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials*), and a ratio of 1.8 in ASTM E1354 (*Standard Test Method for Heat and Visible Smoke Release for Materials and Products Using an Oxygen Consumption Calorimeter*) {cited on p. 70 in [2]}.

Limitations

ASTM E177-90a (*Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods*) states that:

A process is in a *state of statistical control* if the variations between the observed test results from it can be attributed to a constant system of chance causes...By “chance causes” is meant unknown factors, generally numerous and individually of small magnitude, that contribute to variation, but that are not readily detectable or identifiable. (§17.1)

A measurement process may be described as precise when its test results are in a state of statistical control and their dispersion is small enough to meet the requirements of the testing situations in which the measurement process will be applied. (§18.2)

The within- and between-laboratory consistency statistics presented above in Figures 3 and 4 reveal no evidence of a lack of statistical control for the test results in this interlaboratory study. However, Figures 1 and 2 reveal considerable dispersion of test results for the fabrics tested. Figures 1 and 2 also reveal marked asymmetry (i.e., positive skew) of test results, ruling out the assumption of a normal distribution for the flammability test results. The normality assumption is certainly not required for a valid flammability test method, but its violation means that the nominal 95% confidence level associated with the repeatability and reproducibility limits defined above is not exactly correct for the continuous combustion time data, though the limits may still be reasonable approximations for both the continuous and pass-fail data [3, 4, 5]. The repeatability and reproducibility limits clearly depend on the type of fabrics (Tables 1 and 2) and may be expected to vary similarly across other types of fabric not included in this interlaboratory study. Fabrics with a very small (or very large) expected proportion of specimen failures are likely to yield very small repeatability and reproducibility coefficients under the draft flammability test method examined in this interlaboratory study.

The numbers of failures acceptable (1) per specimen and (2) in the complete sample of tested specimens (if any) are not specified in the draft test standard. The analysis of pass-fail data in this report therefore assumed that a given test specimen failed if and only if any of the 3 consecutive combustion time tests on that specimen failed the 120 sec criterion and computed the proportion of specimens failing out of 10 specimens tested. Furthermore, the sample size of 10 specimens chosen for this interlaboratory study was arbitrarily specified as a reasonable number to test given logistical constraints of conducting the study.

Conclusion

The 9 participating laboratories exhibited consistent combustion time means (k statistics) and variances (h statistics) across all 5 fabrics in the study (Figures 2 and 3), satisfying these essential characteristics of the ASTM E691-92 guidelines for an acceptable test standard. As shown in Figures 1 and 2, the fabrics exhibited considerable within-laboratory combustion time variability and skew (excluding fabric F, which failed every ignition test in every laboratory). For the continuous combustion time data (Table 1), repeatability standard deviations (within-laboratory) ranged from 8.02 (fabric E) to 30.01 seconds (fabric I), whereas reproducibility standard deviations (between-laboratory) ranged from 8.82 (fabric E) to 35.69 (fabric I) seconds. For the pass-fail data (Table 2), repeatability standard deviations (within-laboratory) ranged from .07 (fabric E) to .15 (fabric D), whereas reproducibility standard deviations (between-laboratory) ranged from .07 (fabric E) to .24 (fabric D). For both continuous and pass-fail combustion time data, reproducibility standard deviations were only slightly larger than repeatability standard deviations, indicating little between-laboratory variation. For fabrics with a very small (or very large) expected proportion of failures, both repeatability and reproducibility standard deviations are very small, satisfying this essential characteristic of the ASTM E691-92 guidelines for acceptable *precision* in a test method.

References

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Glossary (see ASTM E456-96)

Precision, n – the closeness of agreement between independent test results obtained under stipulated conditions. Note 1 – The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation. Note 2 – “Independent test results” means results obtained in a manner not influenced by any previous result on the same or similar test object. Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme stipulated conditions.

Repeatability, n – precision under repeatability conditions.

Repeatability conditions, n – conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

Repeatability limit, r, n – the value below which the absolute difference between two individual test results obtained under repeatability conditions may be expected to occur with a probability of approximately 0.95 (95%).

Repeatability standard deviation, S_r, n – the standard deviation of test results obtained under repeatability conditions. Note 1 – In an interlaboratory study, this is the pooled standard deviation of test results obtained under repeatability conditions.

Reproducibility, n – precision under reproducibility conditions.

Reproducibility conditions, n – conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. Note 1 – Identical material means either the same test specimens or test specimens are tested by all the laboratories as for a nondestructive test or test specimens are taken at random from a single quantity of material that is as nearly homogeneous as possible. Note 2 – A different laboratory of necessity means a different operator, different equipment, and different location and under different supervisory control.

Reproducibility limit, R, n – the value below which the absolute difference between two test results obtained under reproducibility conditions may be expected to occur with a probability of approximately 0.95 (95%).

Reproducibility standard deviation, S_R, n – the standard deviation of test results obtained under reproducibility conditions.

Test result, n – the value of a characteristic obtained by carrying out a specified test method. Note 1 – also referred to as *test determination*. Note 2 – The test method should specify that one or a number of individual observations be made and their average or another appropriate function, such as the median or the standard deviation, be reported as the test result. It also may require standard

corrections to be applied, such as correction of gas volumes to standard temperature and pressure. A test result, therefore, can be a result calculated from several observed values. In the simplest case, the test result is the observed value itself.

Test specimen, n – the portion of a test specimen needed to obtain a single test determination. Note 1 – When used for a physical test, this is sometimes called *test piece*. For a chemical test, it is sometimes called *test portion* or *test sample*. In interlaboratory evaluation of test methods and other statistical procedures, it is best to reserve the word *sample* for the whole amount of material involved and not the individual test specimens, pieces or portions being tested.

Test specimen, n – the total quantity of material (containing one or more test specimens) needed to obtain a test result as specified in the test method. See *test result*.

Appendix A

Test Results

Following are the actual test observations and determinations collected by the 9 laboratories on the 5 different fabrics. Observations are total combustion times (in seconds) for flaming, glowing, and smoldering, with times exceeding 120 sec recorded as exactly 120 sec.

Obs. Mean
(Test Result)

Fabric Laboratory Block Obs. 1 Obs.2 Obs.3

B	1	1	120	3	3	42.00
B	1	2	3	120	3	42.00
B	1	3	3	3	3	3.00
B	1	4	3	3	3	3.00
B	1	5	4	4	3	3.67
B	1	6	3	3	2	2.67
B	1	7	2	2	2	2.00
B	1	8	2	2	2	2.00
B	1	9	2	0	0	0.67
B	1	10	0	0	0	0.00
B	2	1	6	6	6	6.00
B	2	2	5	6	6	5.67
B	2	3	5	7	7	6.33
B	2	4	7	6	6	6.33
B	2	5	5	5	5	5.00
B	2	6	5	6	6	5.67
B	2	7	6	5	4	5.00
B	2	8	4	4	5	4.33
B	2	9	4	6	6	5.33
B	2	10	5	5	5	5.00
B	3	1	4	3	3	3.33
B	3	2	3	3	3	3.00
B	3	3	3	3	3	3.00
B	3	4	3	3	3	3.00
B	3	5	3	4	4	3.67
B	3	6	120	5	4	43.00
B	3	7	3	3	3	3.00
B	3	8	3	3	3	3.00
B	3	9	4	4	4	4.00
B	3	10	5	3	3	3.67
B	4	1	3	3	4	3.33
B	4	2	2	4	3	3.00
B	4	3	120	3	120	81.00
B	4	4	4	4	4	4.00
B	4	5	4	3	3	3.33
B	4	6	120	4	3	42.33
B	4	7	3	4	3	3.33
B	4	8	8	4	4	5.33
B	4	9	3	3	4	3.33
B	4	10	3	5	3	3.67
B	5	1	8	6	6	6.67
B	5	2	5	6	6	5.67
B	5	3	5	6	5	5.33
B	5	4	4	5	4	4.33
B	5	5	4	4	6	4.67
B	5	6	13	5	5	7.67

Fabric	Laboratory	Block	Obs. 1	Obs.2	Obs.3	Obs. Mean (Test Result)
B	5	7	6	4	4	4.67
B	5	8	6	5	6	5.67
B	5	9	5	5	7	5.67
B	5	10	6	5	4	5.00
B	6	1	13	15	14	14.00
B	6	2	13	12	17	14.00
B	6	3	13	12	10	11.67
B	6	4	11	11	12	11.33
B	6	5	10	11	11	10.67
B	6	6	13	12	12	12.33
B	6	7	12	13	14	13.00
B	6	8	10	11	10	10.33
B	6	9	12	10	10	10.67
B	6	10	12	12	10	11.33
B	7	1	8	7	7	7.33
B	7	2	8	7	5	6.67
B	7	3	120	9	7	45.33
B	7	4	5	6	6	5.67
B	7	5	3	5	4	4.00
B	7	6	7	5	5	5.67
B	7	7	5	10	7	7.33
B	7	8	9	6	7	7.33
B	7	9	5	120	6	43.67
B	7	10	7	4	4	5.00
B	8	1	6	6	5	5.67
B	8	2	6	6	6	6.00
B	8	3	6	9	8	7.67
B	8	4	7	7	7	7.00
B	8	5	6	8	6	6.67
B	8	6	7	8	8	7.67
B	8	7	9	7	8	8.00
B	8	8	7	8	7	7.33
B	8	9	6	6	5	5.67
B	8	10	7	8	7	7.33
B	9	1	5	5	4	4.67
B	9	2	5	7	5	5.67
B	9	3	5	4	4	4.33
B	9	4	5	3	4	4.00
B	9	5	5	4	4	4.33
B	9	6	7	5	6	6.00
B	9	7	6	3	4	4.33
B	9	8	6	7	7	6.67
B	9	9	5	6	4	5.00
B	9	10	3	120	8	43.67
D	1	1	25	0	10	11.67
D	1	2	0	0	0	0.00

Fabric	Laboratory	Block	Obs. 1	Obs.2	Obs.3	Obs. Mean (Test Result)
D	1	3	2	120	2	41.33
D	1	4	3	4	3	3.33
D	1	5	120	0	0	40.00
D	1	6	120	3	3	42.00
D	1	7	2	5	5	4.00
D	1	8	4	5	0	3.00
D	1	9	0	0	0	0.00
D	1	10	3	4	0	2.33
D	2	1	11	6	6	7.67
D	2	2	6	10	120	45.33
D	2	3	7	120	6	44.33
D	2	4	12	10	120	47.33
D	2	5	13	8	13	11.33
D	2	6	8	5	120	44.33
D	2	7	7	120	5	44.00
D	2	8	4	8	6	6.00
D	2	9	2	2	4	2.67
D	2	10	120	5	9	44.67
D	3	1	120	120	120	120.00
D	3	2	120	6	120	82.00
D	3	3	*	4	5	4.50
D	3	4	120	120	5	81.67
D	3	5	8	8	120	45.33
D	3	6	6	120	120	82.00
D	3	7	3	120	120	81.00
D	3	8	8	9	120	45.67
D	3	9	12	120	3	45.00
D	3	10	120	4	120	81.33
D	4	1	3	0	15	6.00
D	4	2	10	120	120	83.33
D	4	3	5	3	0	2.67
D	4	4	120	0	6	42.00
D	4	5	120	3	0	41.00
D	4	6	120	120	5	81.67
D	4	7	4	4	2	3.33
D	4	8	11	3	120	44.67
D	4	9	0	120	8	42.67
D	4	10	0	3	120	41.00
D	5	1	16	120	120	85.33
D	5	2	120	16	20	52.00
D	5	3	19	22	19	20.00
D	5	4	17	19	17	17.67
D	5	5	120	120	7	82.33
D	5	6	120	120	16	85.33
D	5	7	120	14	120	84.67
D	5	8	120	120	120	120.00

Fabric	Laboratory	Block	Obs. 1	Obs.2	Obs.3	Obs. Mean (Test Result)
D	5	9	120	16	120	85.33
D	5	10	16	47	120	61.00
D	6	1	120	24	19	54.33
D	6	2	23	120	25	56.00
D	6	3	19	21	22	20.67
D	6	4	22	22	120	54.67
D	6	5	21	120	18	53.00
D	6	6	120	120	120	120.00
D	6	7	22	18	24	21.33
D	6	8	21	120	27	56.00
D	6	9	120	20	26	55.33
D	6	10	21	25	120	55.33
D	7	1	120	5	13	46.00
D	7	2	7	120	2	43.00
D	7	3	7	9	120	45.33
D	7	4	8	6	10	8.00
D	7	5	8	120	120	82.67
D	7	6	120	120	120	120.00
D	7	7	120	15	120	85.00
D	7	8	5	9	9	7.67
D	7	9	6	120	120	82.00
D	7	10	120	120	10	83.33
D	8	1	10	6	7	7.67
D	8	2	12	11	10	11.00
D	8	3	8	120	7	45.00
D	8	4	9	11	9	9.67
D	8	5	120	120	10	83.33
D	8	6	11	10	10	10.33
D	8	7	15	13	10	12.67
D	8	8	120	10	8	46.00
D	8	9	7	7	8	7.33
D	8	10	10	11	8	9.67
D	9	2	8	120	8	45.33
D	9	3	8	14	120	47.33
D	9	5	103	120	120	114.33
D	9	6	10	120	120	83.33
D	9	7	10	14	10	11.33
D	9	7	120	23	12	51.67
D	9	8	8	9	10	9.00
D	9	8	9	120	120	83.00
D	9	9	120	120	12	84.00
D	9	10	10	120	12	47.33
E	1	1	10	17	20	15.67
E	1	2	18	15	20	17.67
E	1	3	9	20	25	18.00
E	1	4	120	10	26	52.00

Fabric	Laboratory	Block	Obs. 1	Obs. 2	Obs. 3	Obs. Mean (Test Result)
E	1	5	15	10	24	16.33
E	1	6	14	12	17	14.33
E	1	7	8	10	10	9.33
E	1	8	10	20	14	14.67
E	1	9	17	15	20	17.33
E	1	10	10	14	10	11.33
E	2	1	15	19	17	17.00
E	2	2	44	17	21	27.33
E	2	3	17	17	16	16.67
E	2	4	120	10	12	47.33
E	2	5	12	17	13	14.00
E	2	6	13	14	12	13.00
E	2	7	19	15	14	16.00
E	2	8	16	16	13	15.00
E	2	9	13	18	16	15.67
E	2	10	18	14	17	16.33
E	3	1	10	9	9	9.33
E	3	2	9	8	8	8.33
E	3	3	10	11	11	10.67
E	3	4	9	9	9	9.00
E	3	5	9	9	9	9.00
E	3	6	9	9	9	9.00
E	3	7	9	9	9	9.00
E	3	8	8	9	9	8.67
E	3	9	11	10	9	10.00
E	3	10	10	10	9	9.67
E	4	1	15	13	13	13.67
E	4	2	15	16	14	15.00
E	4	3	12	11	12	11.67
E	4	4	14	15	15	14.67
E	4	5	11	12	12	11.67
E	4	6	15	15	15	15.00
E	4	7	12	15	14	13.67
E	4	8	14	17	15	15.33
E	4	9	10	12	14	12.00
E	4	10	12	14	14	13.33
E	5	1	14	14	15	14.33
E	5	2	19	24	16	19.67
E	5	3	17	38	18	24.33
E	5	4	18	16	17	17.00
E	5	5	18	16	16	16.67
E	5	6	18	20	18	18.67
E	5	7	14	80	19	37.67
E	5	8	16	19	19	18.00
E	5	9	24	19	17	20.00
E	5	10	17	18	21	18.67

							Obs. Mean (Test Result)
Fabric	Laboratory	Block	Obs. 1	Obs.2	Obs.3		
E	6	1	24	22	24		23.33
E	6	2	24	21	23		22.67
E	6	3	26	23	24		24.33
E	6	4	23	25	24		24.00
E	6	5	23	23	23		23.00
E	6	6	20	23	20		21.00
E	6	7	24	22	21		22.33
E	6	8	18	23	23		21.33
E	6	9	21	23	23		22.33
E	6	10	22	28	22		24.00
E	7	1	14	120	12		48.67
E	7	2	11	11	11		11.00
E	7	3	15	15	15		15.00
E	7	4	15	52	15		27.33
E	7	5	13	12	13		12.67
E	7	6	12	13	23		16.00
E	7	7	21	13	13		15.67
E	7	8	9	10	9		9.33
E	7	9	9	9	9		9.00
E	7	10	11	10	8		9.67
E	8	1	120	18	18		52.00
E	8	2	16	17	17		16.67
E	8	3	25	22	29		25.33
E	8	4	19	18	17		18.00
E	8	5	20	17	18		18.33
E	8	6	21	19	18		19.33
E	8	7	18	23	20		20.33
E	8	8	18	19	18		18.33
E	8	9	21	20	19		20.00
E	8	10	18	18	19		18.33
E	9	1	17	13	14		14.67
E	9	2	13	10	14		12.33
E	9	3	14	15	14		14.33
E	9	4	13	14	18		15.00
E	9	5	14	11	13		12.67
E	9	6	12	16	15		14.33
E	9	7	13	14	14		13.67
E	9	8	12	12	12		12.00
E	9	9	14	10	13		12.33
E	9	10	15	13	12		13.33
F	1	1	120	120	120		120.00
F	1	2	120	120	120		120.00
F	1	3	120	120	120		120.00
F	1	4	120	120	120		120.00
F	1	5	120	120	120		120.00
F	1	6	120	120	120		120.00

Fabric Laboratory Block			Obs. 1	Obs.2	Obs.3	Obs. Mean (Test Result)
F	1	7	120	120	120	120.00
F	1	8	120	120	120	120.00
F	1	9	120	120	120	120.00
F	1	10	120	120	120	120.00
F	2	1	120	120	120	120.00
F	2	2	120	120	120	120.00
F	2	3	120	120	120	120.00
F	2	4	120	120	120	120.00
F	2	5	120	120	120	120.00
F	2	6	120	120	120	120.00
F	2	7	120	120	120	120.00
F	2	8	120	120	120	120.00
F	2	9	120	120	120	120.00
F	2	10	120	120	120	120.00
F	3	1	120	120	120	120.00
F	3	2	120	120	120	120.00
F	3	3	120	120	120	120.00
F	3	4	120	120	120	120.00
F	3	5	120	120	120	120.00
F	3	6	120	120	120	120.00
F	3	7	120	120	120	120.00
F	3	8	120	120	120	120.00
F	3	9	120	120	120	120.00
F	3	10	120	120	120	120.00
F	4	1	120	120	120	120.00
F	4	2	120	120	120	120.00
F	4	3	120	120	120	120.00
F	4	4	120	120	120	120.00
F	4	5	120	120	120	120.00
F	4	6	120	120	120	120.00
F	4	7	120	120	120	120.00
F	4	8	120	120	120	120.00
F	4	9	120	120	120	120.00
F	4	10	120	120	120	120.00
F	5	1	120	120	120	120.00
F	5	2	120	120	120	120.00
F	5	3	120	120	120	120.00
F	5	4	120	120	120	120.00
F	5	5	120	120	120	120.00
F	5	6	120	120	120	120.00
F	5	7	120	120	120	120.00
F	5	8	120	120	120	120.00
F	5	9	120	120	120	120.00
F	5	10	120	120	120	120.00
F	6	1	120	120	120	120.00
F	6	2	120	120	120	120.00

Obs. Mean
(Test Result)

Fabric Laboratory Block Obs. 1 Obs.2 Obs.3

F	6	3	120	120	120	120.00
F	6	4	120	120	120	120.00
F	6	5	120	120	120	120.00
F	6	6	120	120	120	120.00
F	6	7	120	120	120	120.00
F	6	8	120	120	120	120.00
F	6	9	120	120	120	120.00
F	6	10	120	120	120	120.00
F	7	1	120	120	120	120.00
F	7	2	120	120	120	120.00
F	7	3	120	120	120	120.00
F	7	4	120	120	120	120.00
F	7	5	120	120	120	120.00
F	7	6	120	120	120	120.00
F	7	7	120	120	120	120.00
F	7	8	120	120	120	120.00
F	7	9	120	120	120	120.00
F	7	10	120	120	120	120.00
F	8	1	120	120	120	120.00
F	8	2	120	120	120	120.00
F	8	3	120	120	120	120.00
F	8	4	120	120	120	120.00
F	8	5	120	120	120	120.00
F	8	6	120	120	120	120.00
F	8	7	120	120	120	120.00
F	8	8	120	120	120	120.00
F	8	9	120	120	120	120.00
F	8	10	120	120	120	120.00
F	9	1	120	120	120	120.00
F	9	2	120	120	120	120.00
F	9	3	120	120	120	120.00
F	9	4	120	120	120	120.00
F	9	5	120	120	120	120.00
F	9	6	120	120	120	120.00
F	9	7	120	120	120	120.00
F	9	8	120	120	120	120.00
F	9	9	120	120	120	120.00
F	9	10	120	120	120	120.00
I	1	1	25	120	120	88.33
I	1	2	0	0	120	40.00
I	1	3	0	0	0	0.00
I	1	4	25	31	120	58.67
I	1	5	10	120	120	83.33
I	1	6	10	120	120	83.33
I	1	7	0	120	120	80.00
I	1	8	8	30	120	52.67

Fabric	Laboratory	Block	Obs. 1	Obs. 2	Obs. 3	Obs. Mean (Test Result)
I	1	9	15	120	20	51.67
I	1	10	120	120	27	89.00
I	2	1	120	38	24	60.67
I	2	2	120	120	11	83.67
I	2	3	120	120	5	81.67
I	2	4	4	120	120	81.33
I	2	5	10	120	36	55.33
I	2	6	18	120	13	50.33
I	2	7	29	7	50	28.67
I	2	8	120	20	120	86.67
I	2	9	120	120	120	120.00
I	2	10	120	16	120	85.33
I	3	1	120	120	120	120.00
I	3	2	120	120	0	80.00
I	3	3	*	30	0	15.00
I	3	4	120	120	120	120.00
I	3	5	8	120	120	82.67
I	3	6	120	120	27	89.00
I	3	7	120	120	120	120.00
I	3	8	16	23	6	15.00
I	3	9	120	120	120	120.00
I	3	10	120	120	120	120.00
I	4	1	120	120	120	120.00
I	4	2	4	120	120	81.33
I	4	3	120	120	120	120.00
I	4	4	41	120	120	93.67
I	4	5	120	120	120	120.00
I	4	6	6	120	120	82.00
I	4	7	44	120	120	94.67
I	4	8	120	40	3	54.33
I	4	9	5	120	5	43.33
I	4	10	105	120	3	76.00
I	5	1	27	120	120	89.00
I	5	2	12	120	120	84.00
I	5	3	120	120	120	120.00
I	5	4	16	120	120	85.33
I	5	5	120	120	120	120.00
I	5	6	120	120	120	120.00
I	5	7	15	120	12	49.00
I	5	8	7	120	120	82.33
I	5	9	120	120	120	120.00
I	5	10	11	120	120	83.67
I	6	1	120	120	120	120.00
I	6	2	120	120	120	120.00
I	6	3	120	120	25	88.33
I	6	4	15	120	120	85.00

Fabric	Laboratory	Block	Obs. 1	Obs.2	Obs.3	Obs. Mean (Test Result)
I	6	5	25	26	26	25.67
I	6	6	120	120	26	88.67
I	6	7	24	42	120	62.00
I	6	8	120	24	28	57.33
I	6	9	120	30	24	58.00
I	6	10	120	68	25	71.00
I	7	1	120	120	120	120.00
I	7	2	120	120	120	120.00
I	7	3	120	120	120	120.00
I	7	4	120	120	120	120.00
I	7	5	120	120	120	120.00
I	7	6	120	120	120	120.00
I	7	7	120	120	120	120.00
I	7	8	120	120	120	120.00
I	7	9	120	120	120	120.00
I	7	10	120	120	120	120.00
I	8	1	8	12	9	9.67
I	8	2	120	120	9	83.00
I	8	3	120	7	9	45.33
I	8	4	120	9	120	83.00
I	8	5	120	5	5	43.33
I	8	6	6	15	120	47.00
I	8	7	10	8	6	8.00
I	8	8	52	8	13	24.33
I	8	9	120	19	10	49.67
I	8	10	4	3	5	4.00
I	9	1	120	35	120	91.67
I	9	2	120	35	120	91.67
I	9	3	5	120	108	77.67
I	9	4	120	120	20	86.67
I	9	5	8	120	3	43.67
I	9	6	10	35	32	25.67
I	9	7	120	120	120	120.00
I	9	8	120	120	120	120.00
I	9	9	120	120	120	120.00
I	9	10	120	120	120	120.00



UNITED STATES
CONSUMER PRODUCT SAFETY COMMISSION
WASHINGTON, DC 20207

Memorandum

Date: 14 September 2001

TO : Dale Ray, Upholstered Furniture Project Manager
Directorate for Economics

THROUGH: Susan Ahmed, Ph.D., Associate Executive Director
Directorate for Epidemiology

TRR for RA

Russell Roegner, Ph.D., Director *TRR*
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Division of Hazard Analysis

SUBJECT : Statistical estimation of the reduction in fire losses from the adoption of the
CPSC draft small open-flame standard

Statistical estimation of the reduction in fire losses from the adoption of the CPSC draft small open-flame standard

1. Introduction

The Consumer Product Safety Commission (CPSC) is presently considering a mandatory standard to reduce the hazard associated with small open-flame ignitions of upholstered furniture. As part of its regulatory analysis, CPSC is evaluating the costs and benefits of the standard.¹ The benefits of the standard derive from the reduction in fire losses associated with upholstered furniture.² Reductions in both small open-flame ignited and smoking material ignited fire losses are expected under the standard. This memo presents estimates of these reductions and examines the statistical validity and significance of them.

This memo is organized as follows. Section 2 reviews the relevant experimental data. Section 3 discusses measures of ignition propensity. Section 4 reviews general issues related to statistical significance. Section 5 provides the estimates of the reductions in small open-flame and smoking material ignition fire losses and the associated statistical significance summaries. Section 6 summarizes the findings of the memo and presents conclusions. Two appendices provide additional details on the experimental data and the statistical analysis.

2. Experimental Data

The CPSC laboratory has been conducting experiments on the flammability of upholstered furniture for some time. These experiments have included “full-scale,” “mockup,” and “component” experiments. Full-scale experiments use actual furniture and are intended accurately to reproduce the hazard scenario. It is impossible for any experiment to represent perfectly the range of the hazard scenarios, but such full-scale experiments are believed to provide realistic results. Mockup and component experiments use small-scale models of the furniture and are meant to efficiently examine a wide range of experimental factors. The estimates of the reduction in fire losses considered in this memo are all based on full-scale experiments. Mockup and component experimental results are used to support the validity and applicability of the conclusions.

The estimates are based on four distinct experimental studies. Appendix 1 provides details on the four studies and their use in the present analysis. All of the studies generally attempted to choose chairs representative of relevant market distributions. However, there were specific objectives, such as looking at UFAC compliance and effectiveness, and specific constraints, such as the availability of samples, that might bear on the generalizability of the results.

¹ See Preliminary Analysis for a Mandatory Standard Addressing Small Open-Flame Ignitions of Upholstered Furniture (draft), C.L. Smith, April 2001.

² For estimates of historical fires losses associated with upholstered furniture see Upholstered Furniture Fires Loss Estimates 1980 –1998, K. Ault and M. Levenson, February 2001.

Three of the four studies involved chairs designed for the U.S. market. Such chairs are not required to meet standards for small open-flame ignition resistance. The results from these studies are used to estimate the ignition propensity of furniture currently produced for the U.S. market. The fourth study involved chairs designed for sale in the U.K. These chairs should meet U.K. ignition resistance standards. The chairs were tested with the small open-flame mockup test of the CPSC draft standard. Twenty-one of twenty-seven chairs passed this test. The results of the passing chairs are used to estimate the ignition propensity of furniture representative of the U.S. market with the adoption of the CPSC draft standard.

The validity of reduction estimates depends on the extent that the two classes of furniture represent the market conditions with and without the standard. The three studies involving U.S. market furniture provide results that are reasonably representative of the present market conditions. However, what might be questioned is the extent that the U.K. chairs represent the market conditions with the adoption of the standard. The relevant U.K. standards include tests of the upholstery fabric and filling for resistance to both open flame and smoking material ignition. The primary component of the CPSC draft standard involves a test of the small open-flame ignition resistance of the upholstery fabric.

However, although the U.K. standard contains additional tests over the CPSC draft standard, CPSC staff believes the approaches to meeting the U.K. and CPSC tests are similar. CPSC staff believes that manufacturers will make use of flame retardant (FR) treatments of the upholstery fabric, as is commonly done in the U.K. The presence of FR chemicals was detected in the upholstery fabric of all the 21 U.K. chairs used in the reduction estimates.³ The U.K. chairs do differ in a significant regard from furniture that would be produced under the CPSC standard. When U.K. chairs use polyurethane foam filling, the foam is FR treated in order to meet the U.K. standard. Twelve of the 21 passing U.K. chairs had polyurethane foam in contact with the upholstery fabric. In each of these cases, the foam contained FR chemicals.

Twenty-two fabrics from the U.K. chairs were tested for small open-flame ignition in mockups with FR treated foam and with non-FR treated foam. Table 1 summarizes the results. For 20 of the 22 chairs, there was agreement in the mockup results with FR-treated and with non-FR-treated foam. The agreement in results was statistically significant.⁴ In another experiment to examine the effect of FR treated foam on time to ignition in small open-flame application, the FR foam did not appreciably change the time to ignition of the two fabrics tested.⁵ The relevant data regarding the effect of FR treated foam on smoking material ignitions are much more limited.

³ For a review of the chemical and material analysis of the U.K. chairs see UK Chair and Mockup Test Results, L. Fansler, October 2000.

⁴ A Fisher exact test of the independence of two binary variables was performed. The p-value was 0.004.

⁵ Ignition Times Tests With Flame Retardant Foams and Polyester Batting, L. Fansler, October 1996.

Table 1: Small open-flame mockup results for the 22 U.K. chair fabrics.

		Non-FR Treated Foam Mockup		
		No Ignitions	Ignitions	Total
FR Treated Foam Mockup	No Ignitions	16	1	17
	Ignitions	1	4	5
	Total	17	5	22

3. Measures of Ignition Propensity

To properly address the effect of the draft small open-flame standard on ignition propensity, the specific measure of ignition propensity must be considered. Assume that the present fire losses associated with an ignition source, such as cigarettes, is C . The value of C may come from national databases and surveys or other independent analyses. There is a certain probability of a piece of furniture being involved in a cigarette-ignited fire without the draft standard. Call this value p . Likewise, there is a probability of such an event with the draft standard. Call this value q . The fire losses with the adoption of the

draft standard is $\frac{q}{p}C$ and the reduction in losses from the standard is $\left(1 - \frac{q}{p}\right)C$. The key

quantity we are interested in is $r = \left(1 - \frac{q}{p}\right)$.

Each of the two probabilities in r depends on the probability that a cigarette will encounter the furniture and the probability that it will ignite given it has encountered the furniture. Since the probability of a cigarette encountering the furniture does not depend on the draft standard, only the ratio of the probability of ignition without and with the standard is necessary. For the remainder of the memo, p and q will represent such probabilities, respectively.

The full-scale studies described in the previous section are used to estimate p and q . Depending on the study and particular chair, several locations on a chair were tested and in some studies, repetitions of the test were performed. The probabilities of interest, p and q , can each be estimated in two ways. Consider a single chair. The first estimate, referred to as the discrete estimate, is equal to the value of one if any of the multiple tests for the chair resulted in ignition and equal to the value of zero if there were no ignitions. The second estimate of the probability, referred to as the continuous estimate, uses the fraction of ignitions for the chair. For example, if a chair was tested 12 times and there were 4 ignitions, then the discrete estimate would be one and the continuous estimate would be 1/3. An overall estimate of the probability for a class of chairs is formed by averaging the values of the individual chair estimates in the class.

The continuous measure may better represent the probability because it gives the relative frequency of ignitions of multiple tests, which is a common and intuitive interpretation of a probability. It also is not affected by the number of tests performed. Ideally, one would

weight the results of the tests of the various chair locations to reflect the areas of the chair more likely to encounter the ignition source. Since it is unlikely any reliable information is available for such weighting, an unweighted measure is a good compromise.

It is known that cellulosic and thermoplastic fabrics have very different smoking material ignition propensities.⁶ In order to account for these differences for the smoking material estimates, separate estimates of both p and q are calculated for each of the two classes of fabrics and combined weighted by their respective market shares to give the expected probabilities for the market. For small open-flame estimates, this weighting is not performed because the difference between these two classes of fabric is less significant and because limited full-scale tests are available.

4. Statistical Significance

Statistical significance testing is a common and accepted procedure to demonstrate rigorously that an effect is real and not just an artifact of random variation. In the present case, we are interested in the effect of the CPSC draft standard on small open-flame and smoking material ignitions. Statistical significance testing starts by defining a *null hypothesis* about the effect. The null hypothesis is a statement about the effect that you wish to demonstrate is false. For example in the present case, the null hypothesis might be that there is no effect of the draft standard on smoking material ignitions.

Based on statistical considerations, a data dependent decision rule is created. The rule has the property that there is a certain accepted probability that the null hypothesis is declared false, given that it is actually true. This probability, called the significance level, is stated in the design of the experiment stage. Different fields and applications have accepted values for the significance level, but the typical values are 0.01, 0.05, and 0.1. The smallest level in which the null hypothesis is rejected is called the p-value. Thus, a p-value of 0.04 implies that the null hypothesis can be rejected at the 0.05 level. The smaller the p-value, the more evidence there is against the null hypothesis. Note that failure to reject the null hypothesis does not imply that it is true.

The specific statement of the null hypothesis affects the p-value. For example, consider the following two forms of the null hypothesis for the present problem.

Null Hypothesis I: The draft standard results in no change or an increase in smoking material ignitions.

Null Hypothesis II: The draft standard results in no change in smoking material ignitions.

Rejecting either of these statements supports the adoption of the draft standard. The first statement is known as a one-sided test, because only evidence that there is a decrease in ignitions is taken as evidence against the null hypothesis. The second statement is known as a two-sided test, because evidence of a decrease or an increase in ignitions is taken as evidence against the null hypothesis.

⁶ Cigarette – Open Flame Relationship (Draft), R. Khanna, 15 June 2001.

There is an important practical difference between the two hypotheses. For the two-sided test, a larger decrease in cigarette ignitions is needed to reject the null hypothesis than in the one-sided test.⁷ Another way of saying this is that a p-value for the one-sided test is typically one half the size of the comparable p-value for the two-sided test. Because the two-sided test results in a larger and, therefore, less significant p-value, it is considered more conservative and often used by default.

Ultimately, hypothesis testing is designed to protect one from making wrong decisions. Therefore, the proper choice of the two null hypotheses depends on the intended decisions. If an increase in smoking material ignitions results in the same decision as if there is no effect, then the one-sided test is appropriate. A possible justification for this decision rule is that if we see no effect or an increase in ignitions, we will not go forward with the draft standard.

If an increase in smoking materials ignitions results in a different decision than if there is no effect then the two-sided test is appropriate. A possible justification for this decision rule is that we have justified the draft standard based on small open-flame considerations and need to evaluate the effect, positive or negative, of the standard on smoking material ignitions.

There is an opinion among statisticians that statistical significance testing is overused. In particular, statistical significance has no bearing on the size of an effect. A very small effect with no practical significance can be statistically significant. Additionally, too much emphasis is often placed on certain values of the p-value such as 0.05. Results just slightly less significant than 0.05 may improperly be ignored.

The use of standard errors and confidence intervals often provides an improved alternative to statistical significance testing. Standard errors give a measure of the variation of an estimated effect. For example, suppose that the estimated effect on the reduction in smoking material ignitions is 60% with a standard error of 25%. The interval of values defined by the estimated effect plus and minus two standard errors often provides an approximate *95% confidence interval*. Such an interval contains the true value 95% of the time. Unlike statistical significance testing, confidence intervals provide likely values of the effect and thus provide a measure of the size of the effect. Perhaps the common preference for statistical significance testing over confidence intervals is that the former provides a yes/no answer, whereas the latter requires some interpretation.⁸

⁷ The reason for this is that in the two-tailed test, the probability of falsely rejecting the null hypothesis is divided in two to account for both a decrease and an increase.

⁸ There is an equivalence between confidence intervals and statistical significance testing. If the value of zero falls outside the 95% confidence interval, then the null hypothesis that there is no effect is rejected at a level of 0.05.

5. Reduction Estimates

Tables 2 and 3 provide the estimates and associated statistical significance of the reduction in fire losses from small open-flame and smoking materials ignitions. The information is provided for both the continuous and discrete measures defined in Section 3. An appendix provides details on the derivation of these values.

Table 2: Statistical summary of the reduction in small open-flame ignition fire loss, *r*.

	Continuous Measure	Discrete Measure
95% Confidence Interval	88 ± 12%	76 ± 19%
P-Value of One-Sided Test	0.000	0.000
P-Value of Two-Sided Test	0.000	0.000

Table 3: Statistical summary of the reduction in smoking materials ignition fire loss, *r*.

	Continuous Measure	Discrete Measure
95% Confidence Interval	77 ± 27%	57 ± 57%
P-Value of One-Sided Test	0.000	0.026
P-Value of Two-Sided Test	0.000	0.051

For small open-flame, the ignition propensity of the passing U.K. chairs is overwhelmingly statistically significantly less than that of the U.S. market chairs. This is true based on both the continuous and discrete measures. For smoking material ignition, the effect is again overwhelmingly statistically significant when the continuous measure is used. Based on the discrete measure, the effect on smoking material ignition is significant at a 0.05 level using the one-sided test and just fails to be significant at a 0.05 level for the two-sided test. Since certain approximations are used in these calculations, as described in the appendix, the actual significance of the two-sided test may be less than or greater than 0.05.

6. Conclusions

The reduction in U.S. fire losses resulting from the CPSC draft standard for small open-flame ignition resistance of upholstered furniture has been estimated using experimental data on the full-scale testing of furniture. The estimates are based on the comparison of data from studies on U.S. furniture and a study on U.K. furniture. Results from the U.K. furniture that passed the CPSC draft standard test were used to represent the ignition propensity of U.S. furniture with the adoption of the draft standard.

The role of differences between the U.K. standard and the CPSC draft standard was considered. The U.K. standard includes a cigarette ignition test of the upholstery fabric and ignition test of _____ which are not part of the CPSC draft standard. However, CPSC staff believes similar FR treatments of the upholstery as are used in the U.K. would be used in the U.S. to meet the draft standard. As for the filling

material, the practical consequence of the U.K. filling test is that foam filling is usually FR treated. In only 12 of the 21 U.K. chairs used in the estimation was foam in contact with the upholstery fabric in some test location. CPSC staff believes that the FR treatment of the foam will not have an effect on presence of combustion, but chiefly on the rate of combustion. Experimental results comparing the small open-flame ignition propensity of upholstery fabrics with FR treated and with non-FR treated foam filling show practically no difference. CPSC staff believes the U.K. furniture offers the best representation available of the U.S. furniture with the adoption of the draft standard.

Two measures of ignition propensity were considered. The measure referred to as the continuous measure was argued to be the better measure of the requisite probability of ignition. Using this measure, it was shown that the reductions in both small open-flame and smoking material ignitions were overwhelmingly statistically significant.

Appendix 1: Summary of studies used in the loss reduction estimation.

Study (Year)	Summary Report	Original Purpose	Chair Sample	Present Use
CPSC 40 Chair Test Program (1984)	Analysis of CPSC 40 Chair Test Program, May 1984, P. Fairall	To evaluate the cigarette ignition propensity of UFAC Phase 2 furniture.	40 chairs from 40 UFAC manufacturers. Chairs were chosen to represent the market in terms of fabric type and construction.	16 predominately cellululosic and 24 predominately thermoplastics chairs are used as part of a sample of representative furniture of the U.S. market without the CPSC draft standard
Small Open Flame Chair Study (1995)	Upholstered Furniture Flammability Testing: Full Scale Open Flame Data Analysis, February 1996.	To evaluate the small open-flame ignition propensity of currently available U.S. residential furniture.	27 chairs from 3 manufacturers. Each manufacturer provided 9 chairs. Of the 9 chairs, 3 were intended for the U.S. market, 3 for the California market, and 3 for the UK market.	9 U.S. market chairs are used as a sample of representative furniture of the U.S. market without the CPSC draft standard.
Cigarette Ignition Chair Study (1996)	Upholstered Furniture Flammability Testing: Cigarette Ignition Data Analysis, July 1996, G. Stafford and L. Fansler.	To evaluate the cigarette ignition propensity of currently available U.S. residential furniture.	58 pieces of upholstered furniture, 40 were from UFAC manufacturers and 18 from non-UFAC manufacturers. Chairs were chosen to represent the market in terms of fabrics and furniture styles.	34 predominately cellululosic and 22 predominately thermoplastics chairs are used as part of a sample of representative furniture of the U.S. market without the CPSC draft standard.
UK Chair Study (2000)	UK Chair and Mockup Test Results, October 2000, L. Fansler	To evaluate the small open-flame and cigarette ignition propensity of furniture designed to ignition resistant.	27 chairs manufactured in the U.K. purchased from a test lab, retailer and a manufacturer. The chairs were intended to meet U.K. standards.	21 chairs that passed the CPSC draft standard test are used as a sample of representative furniture meeting the draft standard.

Appendix 2: Statistical significance calculations details.

Table A2: Summary statistics for small open-flame data.

Furniture Sample	
With Standard	Without Standard
$n = 21$	$n = 9$
$x = 5$	$x = 9$
$f = 0.24$	$f = 1.0$
$\bar{y} = 0.12$	$\bar{y} = 1.0$
$s(y) = 0.27$	$s(y) = 0.00$

Table A3: Summary statistics for smoking materials data.

		Furniture Sample	
		With Standard	Without Standard
Fabric Type	Cellulosic	$n = 11$ $x = 3$ $f = 0.27$ $\bar{y} = 0.064$ $s(y) = 0.12$	$n = 50$ $x = 26$ $f = 0.52$ $\bar{y} = 0.248$ $s(y) = 0.33$
	Thermoplastic	$n = 10$ $x = 0$ $f = 0$ $\bar{y} = 0$ $s(y) = 0$	$n = 46$ $x = 3$ $f = 0.065$ $\bar{y} = 0.014$ $s(y) = 0.060$

$n =$ Number of chairs

$x =$ Number of chairs with at least one ignition

$f = x/n$

$\bar{y} =$ Mean of the fraction of ignitions⁹

$s(y) =$ Standard deviation of the fraction of ignitions

The statistical analysis given in Section 5 is based on common practices of uncertainty analysis used in science and engineering.¹⁰ Such an analysis is based on variance calculations and normal approximations. The variances are based on exact formulas when available or Taylor series approximations when exact formulas do not exist. The 95 % confidence interval for an estimate e is approximated by $e \pm 1.96se(e)$ where $se(e)$, the standard error of e , is equal to the square root of the variance of e . The one- and two-

⁹ To calculate \bar{y} and $s(y)$, the fraction of ignitions for each chair in the category is calculated. \bar{y} and $s(y)$ are the mean and standard deviation of these values.

¹⁰ For a review of such practices see P. Bevington and D. Robinson, Data Reduction and Error Analysis for the Physical Sciences, 1991.

sided p-values are based on the appropriate tail probability of a standard normal distribution for the value $e/se(e)$.

The variance of the reduction estimate defined in Section 3, $r = \left(1 - \frac{q}{p}\right)$, is

$$\text{var}(r) \approx \left[\text{var}(q)/q^2 + \text{var}(p)/p^2\right](q/p)^2.$$

For the small open-flame estimates, the values of p and q and their variances are based on the summary statistics in Table A2. For the continuous measure of p and q , the values of p and q are equal to the values of \bar{y} from the “Without Standard” and “With Standard” columns, respectively. The associated variances are equal to the corresponding values of $s(y)/\sqrt{n}$. For the discrete measure, the values of p and q are equal to the values of f from the “Without Standard” and “With Standard” columns, respectively. The associated variances are equal to the corresponding values of $f(1-f)/n$.¹¹

For the smoking materials estimates, the values of p and q are estimated separately for cellulosic and thermoplastics fabrics and are combined based on market share weights. Based on the notation given in Table A4,

$$p = w_c p_c + w_i p_i \text{ and } q = w_c q_c + w_i q_i$$

and the associated variances are equal to

$$\text{var}(p) = w_c^2 \text{var}(p_c) + w_i^2 \text{var}(p_i) \text{ and}$$

$$\text{var}(q) = w_c^2 \text{var}(q_c) + w_i^2 \text{var}(q_i).$$

Table A4: Notation for smoking material estimates.

Symbol	Meaning
w_c and w_i	The market share weights for cellulosic and thermoplastics fabrics scaled to sum to 1. The weights used are 0.36 and 0.64. ¹²
p_c and p_i	The estimated ignition propensities furniture for cellulosic and thermoplastics fabrics without the draft standard
q_c and q_i	The estimated ignition propensities furniture for cellulosic and thermoplastics fabrics with the draft standard

The values of $p_c, p_i, q_c,$ and q_i and the associated variances are calculated from the information in Table A3 in a similar manner as for the small open-flame estimates.

¹¹ For the case, when $f=0, f=0.5/n$ is used in the variance calculation of the discrete measure.

¹² See C. Smith April 2001. The values are assumed to be known with little relative uncertainty.



UNITED STATES
CONSUMER PRODUCT SAFETY COMMISSION
WASHINGTON, DC 20207

Memorandum

5/31/00

TO : Linda Fansler, LSM
THROUGH: Warren Porter, Jr., Director, LSC *WJP*
FROM : Shing-Bong Chen, Chemist, LSC *SBC*
SUBJECT : Chemical Analysis of Flame Retardant on Foams and Fiber Fillers from UK Chairs

Backgrounds:

Foam and fiber fill samples of 27 UK chairs were received from Mechanical/Combustion Division (LSM) for material and chemical analysis. The information provided by LSM was that foam samples might contain both melamine and phosphorus based flame retardant chemicals.

This memorandum describes the methods of material and chemical identification using Fourier Transform Infrared Spectrophotometer (FTIR) and Gas Chromatograph/Mass Spectrometry (GC/MS). The phosphorus (P) determination of all foams and fiber filler were done by Inductive Coupled Plasma (ICP).

Methods:

- a. Infrared analysis provided identification of the filling materials.
The spectra were obtained by Nicolet Magna-IR 560 spectrophotometer using ASI DuraSamplIR ATR cell. The materials identification was done by the use of search program against Aldrich Condensed Phase Library and Hummel Polymer Library.
- b. ICP analysis provided both the presence and amount of phosphorus in the filling materials.
Weighted foam/fiber was digested with 2 ml of concentrated nitric acid for 3-4 hours at 120°C. The digest was then diluted with water to 10 ml solution for phosphorus determination by ICP.
- c. Water extraction of foam and GC/MS analysis provided both the identification and the amount of melamine present in the filling materials.
A 10 milliliters of water was added to weighed (~0.25 grams) foam sample in a test tube and was heated to gentle boiling for one hour. A 5 milliliters of hot extract was transfer

to a beaker, the white crystals formed after evaporation of the water. Melamine was determined gravimetrically. The aqueous solution was used to confirm the existing of melamine by GC/MS.

- d. GC/MS analysis of the methylene chloride extract of the filling materials provided information on the presence of phosphate ester flame retardant chemicals. A small portion (0.1 – 0.2 grams) of material was soaked in methylene chloride for a few minutes before GC/MS analysis to examine for phosphorus flame retardant.
- e. GC/MS analysis -
The Total Ion Chromatograph (TIC) was obtained using HP-6890 GC-MSD system. A diluted methylene chloride or water solution was analyzed by GC/MS. The compound identifications were done by using search program against Wiley Library database of chemical mass spectra.

GC conditions

Column, J & W DB-1, 0.25 mm ID, 30 m, 0.1 µm

Carrier Gas, Helium, 1.0 ml/min

Injection, 0.5 – 1 µl split injection

Injector Temperature, 275 °C

Oven Temperature, 50 °C for 2.5 min, 50 °C to 200 °C at 50 °C/min (water extract)

200 °C for 1 min, 200 °C to 240 °C at 20 °C/min
(methylene chloride Extract).

Results:

- The 145 samples of filling materials analyzed from 27 UK chair samples are shown in Table 1.
- The foam filling materials were all polyurethane and accounted for 54 of the 145 samples. Two samples contained melamine, and sixteen samples contained phosphate esters (Fyrol PCF). The remaining thirty six samples appear to be treated with both flame retardants of melamine and phosphate esters.
- The fiber filing materials were predominantly polyester and accounted for 63 of the 145 samples. These 63 samples did not appear to contain flame retardant.
- The fiber filling materials that were mixture of polyester and other fibers or other fibers accounted for 28 samples. These samples contained a maximum of 0.06 percent phosphorous, indicating the presence of little or no flame retardant.

Table 1. Material Identifications, % Melamine and % Phosphorus from UK Chairs

Chair #	Location/Material	Product by FTIR	% P by ICP	Fyrol PCF by GC/MS	% Melamine by gravimetric	Melamine by GC/MS
1	seat-fiber fill batting	polyester	0.00	nd	nd	nd
1	side-foam	polyurethane	0.66	x	15.22	x
1	side-fiber fill	polyester	0.01	nd	nd	nd
1	back-fiber fill batting	polyester	0.01	nd	nd	nd
2	seat-foam	polyurethane	0.81	x	17.28	no
2	seat-gauge	nylon + polyester	0.03	nd	nd	nd
2	side-foam	polyurethane	0.99	x	15.47	x
2	side-fiber fill batting	polyester	0.02	nd	nd	nd
2	back-loose fiber	polyester	0.01	nd	nd	nd
3	seat-loose fiber fill	polyester	0.01	nd	nd	nd
3	side-fiber batting	polyester	0.01	nd	nd	nd
3	side-foam	polyurethane	0.76	x	17.77	x
3	side-fiber pad	polyester	0.01	nd	nd	nd
3	back-loose fiber	polyester	0.01	nd	nd	nd
4	seat-loose fiber batting	polyester	0.01	nd	nd	nd
4	side-fiber batting	polyester	0.01	nd	nd	nd
4	side-foam	polyurethane	0.73	x	16.80	x
4	back-fill fiber batting	polyester	0.00	nd	nd	nd
5	seat-gauge	nd	0.01	nd	nd	nd
5	seat-fiber batting	polyester	0.01	nd	nd	nd
5	seat-foam	polyurethane	0.92	x	11.57	x
5	side-fiber batting	polyester	0.01	nd	nd	nd
5	side-foam	polyurethane	0.99	x	18.98	x
5	back-loose fiber fill	polyester	0.01	nd	nd	nd
6	seat-gauge	nylon + polyester	0.02	nd	nd	nd
6	seat-fiber fill	polyester	0.01	nd	nd	nd
6	seat-foam	polyurethane	0.90	x	2.00	no
6	side-foam	polyurethane	0.64	x	13.21	x
6	back-foam	polyurethane	0.34	x	11.98	x
6	back-fiber batting	polyester	0.01	nd	nd	nd
7	seat-gauge	nd	0.02	nd	nd	nd
7	seat-foam	polyurethane	0.89	x	0.90	no
7	side-fiber	polyester	0.01	nd	nd	nd
7	back-fiber	polyester	0.01	nd	nd	nd

x = Chemicals were identified by GC/MS.

nd = not determined

no = Fyrol PCF or melamine was not detected.

Foam samples are shaded.

Table 1. Continued

Chair #	Location/Material	Product	% P	Fyrol PCF	% Melamine	Melamine
		by FTIR	by ICP	by GC/MS	by gravimetric	by GC/MS
8	seat-fiber batting	polyester	0.01	nd	nd	nd
8	seat-gauge	nylon + polyester	0.02	nd	nd	nd
8	seat-foam	polyurethane	0.99	x	1.21	no
8	side-fiber batting	polyester	0.01	nd	nd	nd
8	side-gauge	nd	0.02	nd	nd	nd
8	side-foam	polyurethane	0.95	x	9.10	x
8	back-fiber batting	polyester	0.01	nd	nd	nd
8	back-foam	polyurethane	1.49	x	1.06	no
9	seat-fiber batting	polyester	0.01	nd	nd	nd
9	seat-gauge	nd	0.02	nd	nd	nd
9	seat-foam	polyurethane	1.04	x	1.33	no
9	side-fiber batting	polyester	0.01	nd	nd	nd
9	side-foam	polyurethane	0.94	x	9.80	x
9	back-fiber batting	polyester	0.00	nd	nd	nd
9	back-nonwoven	nd	0.00	nd	nd	nd
9	back-loose fiber	polyester	0.01	nd	nd	nd
9	back-nonwoven	nd	0.01	nd	nd	nd
10	seat-fiber	polyester	0.01	nd	nd	nd
10	seat-gauge	nylon + polyester	0.01	nd	nd	nd
10	seat-foam	polyurethane	1.06	x	1.12	no
10	side-fiber batting	polyester	0.01	nd	nd	nd
10	side-gauge	nd	0.00	nd	nd	nd
10	side-foam	polyurethane	0.94	x	11.84	x
10	back-loose fiber fill	polyester	0.00	nd	nd	nd
11	seat-loose fiber fill	polyester	0.03	nd	nd	nd
11	side-foam	polyurethane	0.99	x	15.44	x
11	side-fiber batting	polyester	0.01	nd	nd	nd
11	back-fiber loose fill	polyester	0.00	nd	nd	nd
12	seat-fiber loose fill	polyester	0.02	nd	nd	nd
12	side-foam	polyurethane	0.80	x	14.89	x
12	back-fiber loose fill	polyester	0.01	nd	nd	nd
13	seat-gauge	nylon + polyester	0.02	nd	nd	nd
13	seat-foam	polyurethane	1.11	x	0.73	no
13	side-loose fiber batting	polyester	0.01	nd	nd	nd
13	back-fiber	polyester	0.01	nd	nd	nd
14	seat-gauge	polyester	0.01	nd	nd	nd
14	seat-foam	polyurethane	0.92	x	10.87	no
14	side-foam	polyurethane	0.54	x	14.55	x
14	back-fiber loose fill	polyester	0.00	nd	nd	nd

x = Chemicals were identified by GC/MS.

nd = not determined

no = Fyrol PCF or melamine was not detected.

Foam samples are shaded.

Table 1. Continued

Chair #	Location/Material	Product by FTIR	% P by ICP	Fyrol PCF by GC/MS	% Melamine by gravimetric	Melamine by GC/MS
15	seat-fiber fill batting	polyester	0.00	nd	nd	nd
15	seat-gauge	nd	0.01	nd	nd	nd
15	seat-foam	polyurethane	0.92	x	0.42	no
15	side-fiber loose fill	polyester	0.00	nd	nd	nd
15	back-fiber loose fill	polyester	0.00	nd	nd	nd
16	seat-foam	polyurethane	0.65	x	4.63	x
16	seat-thin layer fiber fill	polyester	0.01	no	nd	nd
16	seat-fiber pad	polyester	0.00	nd	nd	nd
16	side-foam	polyurethane	0.65	x	11.64	x
16	side-fiber fill	polyester	0.00	nd	nd	nd
16	side-fiber pad	polyester	0.00	nd	nd	nd
16	back-thin foam	polyurethane	0.58	x	12.10	x
16	back-thick foam	polyurethane	0.74	x	10.76	x
16	back-fiber fill(thin layer)	polyester	0.00	nd	nd	nd
16	burlap	nd	0.01	nd	nd	nd
17	seat-gray foam	polyurethane	0.73	x	1.99	no
17	seat-pink foam	polyurethane	0.02	no	14.18	x
17	seat-fiber fill pad	polyester	0.00	nd	nd	nd
17	back-fiber fill pad	polyester	0.00	nd	nd	nd
17	arm-5/8 foam	polyurethane	0.17	x	16.96	x
17	arm-2 1/2 foam	polyurethane	0.65	x	9.90	x
18	seat-gray foam	polyurethane	0.46	x	0.56	no
18	seat-gauge fabric	nd	0.00	nd	nd	nd
18	seat-fiber fill	polyester	0.00	nd	nd	nd
18	arm(top)-a foam	polyurethane	0.63	x	21.87	x
18	arm(top)-b foam	polyurethane	0.18	x	9.86	x
18	back-pink foam	polyurethane	0.00	no	11.41	x
18	back-fiber fill	polyester	0.00	nd	nd	nd
19	side-small foam	polyurethane	1.28	x	2.36	no
19	side-large foam	polyurethane	1.40	x	3.21	no
19	seat-foam	polyurethane	0.21	x	11.17	x
19	back-fiber fill, 100% polyester	polyester	0.01	nd	nd	nd
19	side-fiber batting(thin layer)	polyester	0.01	nd	nd	nd
20	seat-thin foam	polyurethane	0.21	x	16.67	x
20	side-blue/green foam	polyurethane	0.19	x	15.50	x
20	back-yellow foam	polyurethane	0.66	x	12.79	x
20	back-blue foam	polyurethane	0.21	x	14.39	x

x = Chemicals were identified by GC/MS.
 nd = not determined
 no = Fyrol PCF or melamine was not detected.
 Foam samples are shaded.

Table 1. Continued

Chair #	Location/Material	Product	% P	Fyrol PCF	% Melamine	Melamine
		by FTIR	by ICP	by GC/MS	by gravimetric	by GC/MS
	side-foam	polyurethane	0.70	nd	2.38	no
21	side-fiber fill, thin layer	polyester	0.00	nd	nd	nd
21	seat-fiber fill, 100% polyester	polyester	0.01	nd	nd	nd
21	back-loose fiber fill	polyester	0.00	nd	nd	nd
22	side-pillow foam	polyurethane	0.17	nd	8.99	nd
22	side-foam	polyurethane	0.69	nd	10.19	nd
22	side-fiber fill	polyester	0.00	nd	nd	nd
22	side-fiber pad	polyester	0.01	nd	nd	nd
22	back-foam	polyurethane	0.15	nd	8.55	nd
22	back-fiber fill	polyester	0.00	nd	nd	nd
22	seat-foam	polyurethane	0.75	nd	0.89	no
22	seat-knit gauge fabric	nylon + polyester	0.00	nd	nd	nd
23	side-foam	polyurethane	0.77	nd	17.68	nd
23	seat-green foam	polyurethane	0.69	nd	3.75	nd
23	seat-knit gauge	nylon + polyester	0.01	nd	nd	nd
23	seat-fiber fill, topper	polyester	0.00	nd	nd	nd
23	back-yellow foam	polyurethane	0.35	nd	5.06	nd
23	back-white foam	polyurethane	0.76	nd	16.35	nd
24	back fiber-pad	nylon	0.01	no	nd	nd
24	back--cotton batting	cellulose	0.04	no	nd	nd
24	back-sisal pad	Grilamid	0.06	no	nd	nd
24	side-fiber pad	nylon	0.01	nd	nd	nd
24	side-batting	polyester	0.04	no	nd	nd
24	side-sisal	Grilamid	0.02	no	nd	nd
25	side-fiber pad	nylon	0.01	no	nd	nd
25	side cotton batting	cellulose	0.03	no	nd	nd
25	side-sisal	cellulose + glue	0.02	no	nd	nd
26	side-fiber pad	nylon	0.01	nd	nd	nd
26	side-cotton batting	cellulose	0.04	no	nd	nd
26	side-sisal	cellulose + glue	0.03	no	nd	nd
27	seat-pink foam	polyurethane	0.55	nd	15.13	nd
27	seat-fiber fill,	polyester	0.00	nd	nd	nd
27	side-blue foam	polyurethane	0.54	nd	8.74	nd
27	side-fiber batting	polyester	0.00	nd	nd	nd
27	back-fiber fill	polyester	0.00	nd	nd	nd

x = Chemicals were identified by GC/MS.

nd = not determined

no = Fyrol PCF or melamine was not detected.

Foam samples are shaded.



United States
CONSUMER PRODUCT SAFETY COMMISSION
Washington, D.C. 20207

MEMORANDUM

DATE: June 8, 2000

TO : Linda Fansler, LSE

THROUGH: Warren Porter, LSC *WKP*

FROM : David Cobb, LSC *David Cobb*
Bharat Bhooshan, LSC *Bharat Bhooshan*

SUBJECT : Analysis of UK Upholstery Fabrics for FR Treatment Chemicals

BACKGROUND:

Upholstery fabrics from 27 UK chairs were analyzed flame retardant chemicals (FRC). Samples were analyzed for hexabromocyclododecane (HBCD), decabromo diphenyl ether (DB), antimony (Sb), and phosphorus (P). Twenty three of the UK chair fabrics were backcoated. Backcoated fabrics typically contain Sb and either DB or HBCD. The 4 remaining fabrics were immersion treated. Immersion treated fabrics typically contain an organic phosphorus FRC. The results are contained in table (1).

EXTRACTIONS/DIGESTIONS:

DB was extracted from fabric samples by placing a 20-30 milligram (mg) aliquot of sample in a test tube to which 5 milliliters (ml) of tetrahydrofuran (THF) were added. The test tubes were placed on a shaker and gently agitated for 48 hours at room temperature.

HBCD was extracted from fabric samples by placing a 20-30 mg aliquot of sample in a test tube to which 5 ml of acetonitrile were added. The test tubes were placed on a shaker and gently agitated for 48 hours at room temperature.

Sb was extracted from fabric samples by placing a 50-100 mg aliquot of sample in a test tube to which 10 ml of 4.0 N hydrochloric acid (HCl) was added. The test tubes were stirred on a vortex after 2 hour extraction time, then analyzed for Sb.

Aliquots of the immersion treated fabrics were digested in 2 ml of nitric acid prior to analysis for P content.

ANALYSIS:

DB and HBCD were analyzed by high pressure liquid chromatography (HPLC). The conditions used were as follows:

HBCD

Column: Symmetry C18 2.1 x 100mm
Eluant: 90% acetonitrile, 10% water
Flow: 0.4 ml/min
Detector: Photodiode Array (UV-Vis)
Wavelength: 206 nm
Volume injected: 10 μ l

DB

Column: Symmetry C18 2.1 x 100mm
Eluant: 100% acetonitrile
Flow: 0.4 ml/min
Detector: Photodiode Array (UV-Vis)
Wavelength: 228 nm
Volume injected: 5 μ l

The retention time for HBCD was about 3.2 minutes. The retention time of DB was about 5.0 minutes. Calculations of HBCD and DB were done by measuring the peak areas of standards and samples at this retention time, and performing linear regressions of peak area versus amount of HBCD or DB injected.

Sb and P were measured using an inductively coupled plasma atomic emission (ICP) spectrometer.

Table 1: Results of Analysis

Sample	Antimony %	DB %	HBCD %	Phosphorus %
UK1 Chair	0.68		6.3	
UK1 Extra	1.15		4.0	
UK2 Chair	2.12	6.6		
UK2 Extra	1.68	5.6		
UK3 Chair, Rust	0.63		5.0	
UK3 Chair, Plaid	1.11	5.1		
UK3 Extra, Rust	0.33			
UK4 Chair	3.71	8.3		
UK4 Extra	2.90	6.6		
UK5 Chair	3.07	7.2		
UK5 Extra	4.52	10.0		
UK6 Chair	1.17		10.7	
UK6 Extra	1.14		8.5	
UK7 Chair	1.81	6.4		
UK8 Chair	1.70	4.0		
UK9 Chair	0.49	3.4		
UK9 Extra	0.77	4.8		
UK10 Chair	0.0	0	0	
UK10 Extra	0.63	4.6		
UK11 Chair	1.14	5.7		
UK12 Chair, Rust	2.20			
UK12 Chair, Plaid	1.14		Plaid Back, 10.4 Plaid Cushion, 8.4	
UK12 Extra, Plaid	1.61			
UK13 Chair	2.36	8.6		
UK13 Extra	2.47	12.4		
UK14 Chair, Pink-Blue	3.73	8.2		
UK14 Extra, Pink-Blue	3.13	7.5		
UK15 Chair	2.02	4.3		
UK15 Extra	1.73	4.2		
UK16 Chair	2.20	9.5		
UK17 Chair	1.66	6.3		
UK18 Chair	4.13	10.2		
UK19 Chair	0.28	1.4		
UK20 Chair	1.86	7.5		
UK21 Chair	2.37	7.3		
UK22 Chair	2.64	4.1		
UK23 Chair	3.28	0	0	
UK24 Chair				1.25
UK25 Chair				1.51
UK26 Chair				1.44
UK27 Chair				1.35



United States
CONSUMER PRODUCT SAFETY COMMISSION
Washington, D.C. 20207

MEMORANDUM

DATE: Oct 25, 2001

TO : Linda Fansler, Division of Electrical Engineering (LSE)

Through : Andrew G. Stadnik, Associate Executive Director *Andrew G. Stadnik, Rb.*
Directorate of Laboratory Sciences, LS
Warren Porter, Division Director, Division of Chemistry (LSC), *W.P.*
Directorate of Laboratory Sciences

FROM : David Cobb, Division of Chemistry, LSC *David Cobb*

SUBJECT : Chemical Analysis of Barrier Fabrics

BACKGROUND:

Three of the barrier fabrics tested in accordance with the barrier test plan¹ were treated with flame retardant chemicals (FRC). According to the manufacturers, these fabrics were treated with phosphonic acid, (3-{{hydroxymethyl}amino}-3-oxopropyl)-, dimethyl ester (PA). Chemical analysis of the fabrics was requested to determine FRC load.

METHODS/RESULTS

Fabrics/Sampling:

The three barrier fabrics are identified as follows:

A = FR cotton, 6.5 oz/yd²
B = FR cotton, 6.0 oz/yd²
C = FR cotton, 7.0 oz/yd²

Aliquots from each barrier fabric were obtained throughout the length and width of the roll. Aliquots were obtained from the wedges of each cutout used for flame testing.

Analysis:

PA is usually covalently bonded to the fabric, and can not be analyzed directly. PA contains phosphorus (P), which can be analyzed. The fabric samples were acid digested and analyzed using inductively coupled plasma spectrometry (ICP) to determine the total P content. The fabric samples were also exposed to deionized water to determine the amount of water extractable P. The water extracts were analyzed using ICP to determine P, and high-pressure liquid chromatography (HPLC) to determine phosphate (PO_4^{-3}) ion. HPLC Instrument conditions used were as follows:

Column: IC-Pak, Anion HR 4.6 X 75 mm
Eluant: Borate/Gluconate in 12 % acetonitrile
Flow: 1 ml/min
Detector: Conductivity

Table 1 is a summary of the results.

Table 1. Barrier Fabric Analysis

Fabric		Acid Digest, total P %	Water extractable P %	Phosphate P %
A	Avg. P%	0.56	0.66	0.61
	Range	0.37-0.68	0.40-0.96	0.34-0.84
	Std Dev	0.086	0.148	0.126
B	Avg. P%	1.44	1.29	0.53
	Range	0.86-2.32	0.79-1.72	0.30-0.76
	Std Dev	0.339	0.221	0.123
C	Avg. P%	2.30	2.38	0.78
	Range	1.55-2.84	1.39-3.16	0.37-0.96
	Std Dev	0.219	0.252	0.089

Note: P levels found in specimens of fabric C that passed the crib test averaged 2.78%

Water Soak Analysis:

Fabric cutouts were water soaked² to determine the effect of soaking on flammability testing. The water soaks were analyzed to determine the amount P and PO_4^{-3} that leached from the fabrics. The results are contained in Table 2.

Table 2. Water Soak Analysis

Fabric	% P	Phosphate P %
A	0.54	0.47
B	1.26	0.38
C	2.09	0.58

Cover Fabric Analysis:

Two cover fabrics were also analyzed for P and PO_4^{-3} . One of the fabrics was the standard UK cover fabric that is 100% FR polyester. The other cover fabric was a 100% cotton treated with PA. The results are contained in table 3.

Fabric	Acid Digest, total P %	Water extractable P %	Phosphate P %
UK Cover	0.48	0.002	<0.001
Cover 2, 100% cotton, PA treated, 7.5 oz/yd ² , bright blue	1.10	0.026	0.065

Other Analyses:

Some of barrier fabric acid digests were analyzed for boron (B) and antimony (Sb) using ICP to determine if any other FRCs were present. Sb was not detected, and only trace levels (<0.01%) of B were detected.

DISCUSSION

The spreadsheets showing all the P and PO_4^{-3} results are contained in Attachment A. The following results were noted:

1. Barrier fabric sample C had the highest P levels, which averaged 2.3%. Barrier Fabric A had the lowest P levels which averaged around 0.6%. The P in all 3 barrier fabrics was water extractable. The water extractable P levels averaged nearly the same as the total P. PA treated fabrics the lab has analyzed in the past had P levels of 1.2 to 1.4%, with water extractable P levels of less than 0.1%.
2. Nearly all the water extractable P in barrier fabric A was in the form of PO_4^{-3} ion. 30-40% of the water extractable P in fabric B and C was in the form of PO_4^{-3} ion.
3. Based on the P and PO_4^{-3} ion levels detected, it appears that fabric A does not contain PA. Fabrics B and C may have been treated with PA, but nearly all the P detected was water extractable so it does not appear the treatment was applied in the conventional manner in which PA is covalently bound to the fabric.

REFERENCES

1. Memorandum to A. Stadnik from L. Fansler, LSE *Draft Test Plan for Barrier Tests*, March 8, 2001
2. Draft Standard for Upholstered Furniture, R. Khanna, ES, CPSC revised February 19, 2001.

Barrier Fabrics P Analysis May 16, 2001

Std ppm	Measured ppm
0	0.0139
1	0.751
5	4.947
10	10.9
25	26.68
50	52.68
100	107.6

Regression Output:

Constant	-0.245337
Std Err of Y Est	0.461399
R Squared	0.999885
No. of Observations	7
Degrees of Freedom	5
X Coefficient(s)	1.074813
Std Err of Coef.	0.005146

Sample	Measured P, ppm	Actual P, ppm	Sample Wt (mg)	Volume (ml)	%P
CL-1a	12.21	11.59	19.5	10	0.594276
CL-2a	24.57	23.09	41.2	10	0.56039
CL-3A	23.1	21.72	37.6	10	0.57767
CL-1B	18.39	17.34	27.7	10	0.625928
CL-1C	19.01	17.92	27.8	10	0.644427
CL-1D	25.72	24.16	38.3	10	0.630757
CL-1E	29.57	27.74	42.9	10	0.646621
CL-1F	28.05	26.33	40.4	10	0.651629
CL-2B	25.02	23.51	37.5	10	0.626846
CL-2C	27.96	26.24	39.4	10	0.666043
CL-2D	25.08	23.56	35.8	10	0.658172
CL-2E	24.64	23.15	40.3	10	0.574521
CL-2F	21.91	20.61	36.7	10	0.561668
CL-3B	21.28	20.03	37.6	10	0.532634
CL-3C	20.72	19.51	31.4	10	0.621211
4844-1A	126.1	117.55	48.7	10	2.413778
4844-2A	101.5	94.66	42.4	10	2.232625
4844-3A	115.4	107.60	47.4	10	2.269953
5244-1A	40.24	37.67	24.8	10	1.518844
5244-2A	46.95	43.91	28.9	10	1.519387
5244-3A	61.4	57.35	33.7	10	1.701913
LPC CHK	111.5	103.97			

Barrier Fabrics P Analysis May 17, 2001

Std ppm	Measured ppm	Regression Output:	
0	0.0126	Constant	-0.01943
1	0.7541	Std Err of Y Est	0.228343
5	4.824	R Squared	0.999969
10	10.48	No. of Observations	7
25	25.55	Degrees of Freedom	5
50	50.91	X Coefficient(s)	1.017103
100	101.6	Std Err of Coef.	0.002547

Sample	Measured P, ppm	Actual P, ppm	Sample Wt (mg)	Volume (ml)	%P
CL-3D	21.47	21.13	34.1	10	0.620
CL-3E	21.88	21.53	38.7	10	0.556
CL-3F	17.73	17.45	28.5	10	0.612
4844-1B	80.26	78.93	34	10	2.321
4844-1C	95.97	94.38	39.6	10	2.383
4844-1D	95.99	94.39	46	10	2.052
4844-1E	100.5	98.83	41.5	10	2.381
4844-1F	88.86	87.38	37.3	10	2.343
4844-2B	72.77	71.57	29.6	10	2.418
4844-2C	66.51	65.41	26.3	10	2.487
4844-2D	109.5	107.68	45.3	10	2.377
4844-2E	80.88	79.54	32.5	10	2.447
4844-2F	61.89	60.87	26.8	10	2.271
4844-3B	82.48	81.11	33.6	10	2.414
4844-3C	102.3	100.60	43.5	10	2.313
4844-3D	92.54	91.00	36.2	10	2.514
4844-3E	85.81	84.39	34.6	10	2.439
4844-3F, 1:3	33.14	32.60	45	30	2.173
5244-1B	48.68	47.88	30.3	10	1.580
5244-1C	35.09	34.52	21.9	10	1.576
5244-1D	37.98	37.36	24.8	10	1.506
5244-1E	34.19	33.63	23.2	10	1.450
5244-1F	30.49	30.00	19.8	10	1.515
5244-2B	76.95	75.68	45.3	10	1.671
5244-2C	68	66.88	39.6	10	1.689
5244-2D	77.7	76.41	46.4	10	1.647
5244-2E	60.53	59.53	38.7	10	1.538
5244-2F	55.3	54.39	31.9	10	1.705
5244-3B	58.6	57.63	35.4	10	1.628
5244-3C	60.53	59.53	40.4	10	1.474
5244-3D	86.51	85.07	47	10	1.810
5244-3E	50.27	49.44	29.9	10	1.654
5244-3F	46.84	46.07	30	10	1.536
LPC CHK	105.2	103.45			
0.1 PPM	0.0252	0.04			
0.2 PPM	0.0834	0.10			
0.5 PPM	0.3258	0.34			
5244 3d	35.61	35.03	555	10	1.262
4844-3a	35.72	35.14	332.6	10	2.113
cl-1c	11.06	10.89	390.2	10	0.558

Barrier Fabrics P Analysis May 17, 2001

Sample	%P	Average		Average		Fabric Wt P		Within	Within
		within sub	%RSD	within sample	%RSD	(mg/cm ²)	(mg/cm ²)	Sub	Sample
								P	P
						(mg/cm ²)	(mg/cm ²)	(mg/cm ²)	(mg/cm ²)
4844-1A	2.414	2.316	0.058	2.347	0.049	20.364	0.492	0.472	0.478
4844-1B	2.321					20.364	0.473		
4844-1C	2.383					20.364	0.485		
4844-1D	2.052					20.364	0.418		
4844-1E	2.381					20.364	0.485		
4844-1F	2.343					20.364	0.477		
4844-2A	2.233	2.372	0.042			20.364	0.455	0.483	
4844-2B	2.418					20.364	0.492		
4844-2C	2.487					20.364	0.506		
4844-2D	2.377					20.364	0.484		
4844-2E	2.447					20.364	0.498		
4844-2F	2.271					20.364	0.463		
4844-3A	2.270	2.354	0.053			20.364	0.462	0.479	
4844-3B	2.414					20.364	0.492		
4844-3C	2.313					20.364	0.471		
4844-3D	2.514					20.364	0.512		
4844-3E	2.439					20.364	0.497		
4844-3F	2.173					20.364	0.443		
5244-1A	1.519	1.524	0.032	1.595	0.061	23.758	0.361	0.362	0.379
5244-1B	1.580					23.758	0.375		
5244-1C	1.576					23.758	0.374		
5244-1D	1.506					23.758	0.358		
5244-1E	1.450					23.758	0.344		
5244-1F	1.515					23.758	0.360		
5244-2A	1.519	1.628	0.049			23.758	0.361	0.387	
5244-2B	1.671					23.758	0.397		
5244-2C	1.689					23.758	0.401		
5244-2D	1.647					23.758	0.391		
5244-2E	1.538					23.758	0.365		
5244-2F	1.705					23.758	0.405		
5244-3A	1.702	1.634	0.073			23.758	0.404	0.388	
5244-3B	1.628					23.758	0.387		
5244-3C	1.474					23.758	0.350		
5244-3D	1.810					23.758	0.430		
5244-3E	1.654					23.758	0.393		
5244-3F	1.536					23.758	0.365		
CL-1A	0.594	0.632	0.033	0.609	0.066	22.061	0.131	0.139	0.134
CL-1B	0.626					22.061	0.138		
CL-1C	0.644					22.061	0.142		
CL-1D	0.631					22.061	0.139		
CL-1E	0.647					22.061	0.143		
CL-1F	0.652					22.061	0.144		
CL-2A	0.560	0.608	0.080			22.061	0.124	0.134	
CL-2B	0.627					22.061	0.138		
CL-2C	0.666					22.061	0.147		
CL-2D	0.658					22.061	0.145		
CL-2E	0.575					22.061	0.127		
CL-2F	0.562					22.061	0.124		
CL-3A	0.578	0.587	0.063			22.061	0.127	0.129	
CL-3B	0.533					22.061	0.118		
CL-3C	0.621					22.061	0.137		
CL-3D	0.620					22.061	0.137		
CL-3E	0.556					22.061	0.123		
CL-3F	0.612					22.061	0.135		

May 18, 2001 Barrier Testing DI Water Extractions

Std ppm	Measured ppm	Regression Output:	
0	0.0734	Constant	-0.19489
1	0.7173	Std Err of Y Est	0.291434
5	4.431	R Squared	0.999947
10	9.87	No. of Observations	7
25	24.43	Degrees of Freedom	5
50	49.88		
100	99.06	X Coefficient(s)	0.993853
		Std Err of Coef.	0.00325

Sample	Measured P, ppm	Actual P, ppm	Sample Wt (mg)	Volume (ml)	%P	Avg P per sub	RSD per Sub	Avg P per Sample	RSD per Sample	Avg Total P	% Water Extractable P
CL-1A	10.33	10.59	344.8	200	0.614	0.600	0.031	0.594	0.031	0.609	97.6
CL-1B	11.54	11.81	401.8	200	0.588						
CL-1C	11.06	11.32	390.2	200	0.580						
CL-1D	8.293	8.54	276.5	200	0.618						
CL-2A	12.42	12.69	418.1	200	0.607	0.592	0.035				
CL-2B	13.41	13.69	480.2	200	0.570						
CL-2C	13.32	13.60	444.6	200	0.612						
CL-2D	12.8	13.08	452.6	200	0.578						
CL-3A	15.85	16.14	535.8	200	0.603	0.591	0.032				
CL-3B	10.74	11.00	362.2	200	0.608						
CL-3C	9.321	9.57	326.2	200	0.587						
CL-3D	9.209	9.46	334.5	200	0.566						
4844-1A	36.02	36.44	524	200	1.391	1.934	0.188	2.082	0.111	2.347	88.7
4844-1B	37.29	37.72	358.7	200	2.103						
4844-1C	33.52	33.92	323.5	200	2.097						
4844-1D	39	39.44	367.6	200	2.146						
4844-2A	40.17	40.61	374.9	200	2.167	2.192	0.016				
4844-2B	51.79	52.31	478.5	200	2.186						
4844-2C	37.13	37.56	334.6	200	2.245						
4844-2D	42.7	43.16	397.6	200	2.171						
4844-3A	35.72	36.14	332.6	200	2.173	2.120	0.057				
4844-3B	35.95	36.37	340.8	200	2.134						
4844-3C	34.41	34.82	357.5	200	1.948						
4844-3D	62.29	62.87	565.6	200	2.223						
5244-1A	23.54	23.88	372.2	200	1.283	1.276	0.028	1.293	0.029	1.595	81.0
5244-1B	21.99	22.32	342.3	200	1.304						
5244-1C	20.23	20.55	317.5	200	1.295						
5244-1D	15.04	15.33	250.5	200	1.224						
5244-2A	23.57	23.91	362.3	200	1.320	1.302	0.042				
5244-2B	21.73	22.06	334.6	200	1.319						
5244-2C	18.25	18.56	275.5	200	1.347						
5244-2D	17.66	17.97	293.9	200	1.223						
5244-3A	22.96	23.30	354.8	200	1.313	1.299	0.013				
5244-3B	24.16	24.51	374.4	200	1.309						
5244-3C	29.3	29.68	465.1	200	1.276						
5244-3D	35.61	36.03	555	200	1.298						
LPC CHK											
100 PPM P	102.4	103.23									

May 18, 2001 Barrier Testing DI Water Extractions
 PO₄ Analysis by HPLC

Std (ppm)	Area	Regression Output:	
32.3	498127	Constant	-30394.62
62.5	1080564	Std Err of Y Est	37771.86
142.9	2505509	R Squared	0.99919
0	0	No. of Observations	4
		Degrees of Freedom	2
		X Coefficient(s)	17693.64
		Std Err of Coef.	356.2016

Sample	Area PO ₄	Conc PO ₄ , ppm	Sample Wt (mg)	Volume (ml)	%PO ₄	%P	Avg P per sub	RSD per Sub	Avg P per Sample	RSD per Sample	Avg Total P	% Phosphate P from total P
CL-1A	518804	31.04	344.8	200	1.800	0.588	0.588	0.024	0.583	0.028	0.609	95.7
CL-1B	609095	36.14	401.8	200	1.799	0.587						
CL-1C	573350	34.12	390.2	200	1.749	0.571						
CL-1D	423114	25.63	276.5	200	1.854	0.605						
CL-2A	589186	35.02	418.1	200	1.675	0.547	0.575	0.036				
CL-2B	725393	42.72	480.2	200	1.779	0.581						
CL-2C	667364	39.44	444.6	200	1.774	0.579						
CL-2D	700020	41.28	452.6	200	1.824	0.595						
CL-3A	820607	48.10	535.8	200	1.795	0.586	0.586	0.028				
CL-3B	560135	33.38	362.2	200	1.843	0.601						
CL-3C	494857	29.69	326.2	200	1.820	0.594						
CL-3D	480568	28.88	334.5	200	1.727	0.563						
4844-1A	818176	47.96	524	200	1.830	0.597	0.827	0.191	0.847	0.127	2.347	36.1
4844-1B	810448	47.52	358.7	200	2.650	0.865						
4844-1C	808448	47.41	323.5	200	2.931	0.956						
4844-1D	857586	50.19	367.6	200	2.730	0.891						
4844-2A	870867	50.94	374.9	200	2.717	0.887	0.877	0.047				
4844-2B	1052400	61.20	478.5	200	2.558	0.835						
4844-2C	814038	47.73	334.6	200	2.853	0.931						
4844-2D	893084	52.19	397.6	200	2.625	0.857						
4844-3A	831961	48.74	332.6	200	2.931	0.956	0.837	0.142				
4844-3B	820034	48.06	340.8	200	2.821	0.920						
4844-3C	665126	39.31	357.5	200	2.199	0.718						
4844-3D	1125185	65.31	565.6	200	2.309	0.754						
5244-1A	649867	38.45	372.2	200	2.066	0.674	0.644	0.146	0.607	0.103	1.595	38.0
5244-1B	529511	31.64	342.3	200	1.849	0.603						
5244-1C	434786	26.29	317.5	200	1.656	0.540						
5244-1D	485571	29.16	250.5	200	2.328	0.760						
5244-2A	561185	33.43	362.3	200	1.846	0.602	0.602	0.020				
5244-2B	530554	31.70	334.6	200	1.895	0.618						
5244-2C	413348	25.08	275.5	200	1.821	0.594						
5244-2D	441716	26.68	293.9	200	1.816	0.593						
5244-3A	580794	34.54	354.8	200	1.947	0.635	0.574	0.078				
5244-3B	531194	31.74	374.4	200	1.695	0.553						
5244-3C	697618	41.15	465.1	200	1.769	0.577						
5244-3D	769864	45.23	555	200	1.630	0.532						

**May 22, 2001 Barrier Testing DI Water Extractions and Acid Digestions
P Analysis by ICP**

Std Actual Measured	Regression Output:	
ppm ppm		
0 -0.1649	Constant	0.258028
1 0.5292	Std Err of Y Est	0.630369
5 3.396	R Squared	0.99946
10 8.262	No. of Observations	7
25 17.02	Degrees of Freedom	5
50 34.17		
100 67.77	X Coefficient(s)	0.676315
	Std Err of Coef.	0.00703

Sample	Measured Actual	Vol (ml)	Wt (mg)	%P	Avg P per	RSD per
	ppm ppm				sub	Sub
Acid Digestions						
4844-6a	48.45 71.26	10	25.4	2.805	2.772	0.052
4844-6b	41.74 61.34	10	21.6	2.840		
4844-6c	28.63 41.95	10	15.5	2.707		
4844-6d	28.53 41.80	10	15.2	2.750		
4844-6e	44.67 65.67	10	24.1	2.725		
4844-6f	45.6 67.04	10	23.9	2.805		
DI Water Extractions						
4844-6-a	61.74 90.91	200	589.5	3.084	2.789	0.297
4844-6-b	63.5 93.51	200	592.1	3.159		
4844-6-c	55.8 82.12	200	579.8	2.833		
4844-6-d	26.16 38.30	200	318	2.409		
4844-6-e	40.27 59.16	200	435.7	2.716		
4844-6-f	39.14 57.49	200	453.7	2.534		

PO₄ Analysis by HPLC

Std (ppm)	Area	Regression Output:	
16.4	332886	Constant	-36113.2
32.3	530039	Std Err of Y Est	53071.22
62.5	1096948	R Squared	0.99963
142.9	2539936	No. of Observations	5
333.3	6108808	Degrees of Freedom	3
		X Coefficient(s)	18367.69
		Std Err of Coef.	203.9391

Sample	Area PO ₄	Conc PO ₄ ppm	Sample Wt (mg)	Volume (ml)	%PO ₄	%P	Avg P per sub	RSD per Sub	Avg Total P	% Phosphate P from total P
4844-6-a	1145001	64.30	589.5	200	2.182	0.712	0.784	0.099	2.772	28.3
4844-6-b	1267001	70.95	592.1	200	2.396	0.782				
4844-6-c	1187336	66.61	579.8	200	2.298	0.750				
4844-6-d	762883	43.50	318	200	2.736	0.893				
4844-6-e	917799	51.93	435.7	200	2.384	0.778				
4844-6-f	857544	48.65	453.7	200	2.145	0.700				

Std Actual	Measured	Regression Output	Std Actual Measured
ppm	ppm	ppm	ppm
0	-0.0022	Constant	0
1	0.8945	Std Err of Y Est	1
5	5.574	R Squared	5
10	10.96	No of Observations	10
25	25.98	Degrees of Freedom	25
		X-Coefficients	
		Std Err of Coef	
		0.019006	1.02732
			0.019006

Sample	Measured	Actual	Vol (ml)	Wt (mg)	%P	Avg P per Sample	RSD per Sample	Maximum	Minimum
Acid Digestions	ppm	ppm				Sample	Sample		
CL1L	9.012	6.63	10	23.5	0.367	0.517	0.004	0.367	0.675
CL1C	13.91	13.40	10	35.6	0.376				
CL4R	12.77	12.28	10	30.8	0.402				
CL4C	11.02	10.50	10	28.1	0.408				
CL1L	12.57	12.10	10	29.8	0.405				
CL1C	17.98	17.49	10	24.4	0.482				
CL1R	15.82	15.08	10	28.1	0.536				
CL21L	18.44	17.81	10	30	0.594				
CL21C	18.42	17.79	10	32.6	0.546				
CL29R	21.56	20.86	10	37.9	0.560				
CL29C	14.42	13.90	10	29.2	0.530				
CL31L	17.83	17.22	10	32.2	0.535				
CL31C	19.57	18.91	10	35	0.540				
CL36R	32.2	31.20	10	46.2	0.675				
CL36C	18.39	18.73	10	30.9	0.605				
CL41L	20.9	20.11	10	32.1	0.626				
CL41C	24.01	23.23	10	31.4	0.621				
CL48R	21.8	21.58	10	31.7	0.624				
CL48C	21.83	21.4	10	28	0.594				
CL49L	22.78	22.22	15	26	1.100				
CL49C	20.86	20.34	15	35.4	0.862	1.303	0.415	0.862	2.320
CL49R	21.14	20.62	15	30.6	1.011				
CL49L	31.25	30.61	15	34.6	1.319				
CL49C	29.38	28.77	15	33.9	1.273				
CL49R	18.5	18.03	15	23.9	1.009				
CL49L	24.64	24.08	15	33	1.044				
CL49C	14.85	14.40	15	22.9	0.944				
CL49R	24.71	24.15	15	34.4	1.188				
CL49L	24.44	23.89	15	36.3	0.960				
CL49C	24.48	23.93	15	35	0.987				
CL49R	42.8	42.25	15	38	1.482				
CL49L	31.51	30.96	15	37.3	1.241				
CL49C	44.08	43.28	15	31.2	2.074				
CL49R	39.86	39.10	15	39.8	1.470				
CL49L	38.1	38.36	15	24.8	2.320				
CL49C	23.12	22.57	15	37.6	1.527				
CL49R	43.08	42.29	15	34.1	0.827				
CL49L					1.865				

Std Actual	Measured	Regression Output
ppm	ppm	ppm
0	0.012	Constant
1	1.088	Std Err of Y Est
5	5.403	R Squared
10	10.97	No of Observations
25	25.32	Degrees of Freedom
		X-Coefficients
		Std Err of Coef
		0.020007
		1.012181
		0.020007

Water Extractions Cl

Std Actual	Measured	Regression Output
0	0.2111	Constant
1	1.16	Std Err of Y Est
5	5.829	R Squared
10	12.43	No. of Observations
25	28.42	Degrees of Freedom

Std Actual	Measured	Regression Output
0	0.0006	Constant
1	0.0194	Std Err of Y Est
5	5.417	R Squared
10	11.07	No. of Observations
25	25.75	Degrees of Freedom

Water Extractions 3244

Std Actual	Measured	Regression Output
0	0.0006	Constant
1	0.0194	Std Err of Y Est
5	5.417	R Squared
10	11.07	No. of Observations
25	25.75	Degrees of Freedom

Sample	Measured	Actual	Vol (ml)	Wt (mg)	% ^a	Avg P per Sample	RSD per Sample	Minimum	Maximum	HPLC P
CL1L	10.72	8.19	50	97.6	0.471	0.707	0.174	0.400	0.965	0.387
CL1C	18.56	16.08	50	121.9	0.600					0.600
CL4R	11.00	8.45	50	98	0.482					0.414
CL6C	8.616	8.36	50	104.6	0.400					0.339
CL11L	24.37	21.20	50	122	0.566					0.756
CL11C	22.36	18.43	50	110.7	0.554					0.749
CL18R	17.82	15.25	50	110.7	0.656					0.622
CL16C	14.85	16.24	50	103.4	0.780					0.724
CL21C	25.14	21.87	50	153	0.714					0.672
CL20R	20.03	17.37	50	112.3	0.774					0.646
CL28C	13.7	11.80	50	115.6	0.510					0.453
CL31L	25.76	22.42	50	113.8	0.775					0.629
CL31C	24.4	21.22	50	157.1	0.714					0.629
CL38R	27.8	24.30	50	136.6	0.760					0.653
CL39C	25.51	22.20	50	136	0.828					0.643
CL41L	30.78	26.82	50	146.3	0.816					0.740
CL41C	28.73	25.04	50	128.7	0.865					0.823
CL48C	13.06	11.78	50	138.6	0.403					0.344
CL48R	18.06	16.19	50	128.2	1.652	1.258	0.280	0.788	1.720	0.714
CL48L	23.49	21.8	50	144	1.652					0.714
CL48C	23.49	21.8	50	144	1.652					0.714
CL48R	23.49	21.8	50	144	1.652					0.714
CL48L	23.49	21.8	50	144	1.652					0.714
CL48C	23.49	21.8	50	144	1.652					0.714
CL48R	23.49	21.8	50	144	1.652					0.714
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