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Interlaboratory Study of ASTM F2731, Standard Test Method for Measuring the Transmitted and Stored Energy of Firefighter Protective Clothing Systems

ABSTRACT: This paper describes an interlaboratory study conducted using ASTM F2731, Standard Test Method for Measuring the Transmitted and Stored Energy of Firefighter Protective Clothing Systems. Five replications of six different composites representative of firefighting turnout gear materials were tested at six different laboratories equipped to conduct the test. Data collected were used to predict the time to second degree burn for each of the turnout composite test specimens. Statistical analysis showed good agreement between test sites. This interlaboratory study confirmed the repeatability and reliability of ASTM F2731, a test method used to measure an important property associated with the thermal protective performance of firefighter turnout materials.

KEYWORDS: firefighting, interlaboratory study, protective clothing, textiles, turnout gear, stored energy

Introduction

Skin burn injuries incurred during exposures to radiant heat at levels below flashover conditions are a cause for concern to firefighters. These burn injuries, often referred to as "stored energy burns," can involve several minutes of exposure to thermal energy levels not sufficient to degrade the outer shell of the turnout gear. They occur as a result of radiant heat transmitted from the firefighting environment and stored within the layers of the turnout suit composite. Subsequent compression of the layers exacerbates burns through rapid discharge of stored thermal energy within the layers of the garment. Studies have shown that these burns often occur in areas where moisture vapor impermeable layers, such as reflective trim, are attached to the outer shell of the turnout. The presence of moisture within the layers of the turnout suit has been shown to contribute to skin burn injuries [1,2].

An apparatus and test method for measuring the heat transmitted and stored in firefighting materials has been developed by NC State Univ. [2]. These test procedures are the basis for ASTM F2731, Standard Test Method for Measuring the Transmitted and Stored Energy of Firefighter Protective Clothing Systems [3]. As part of the development process, a limited interlaboratory study was conducted that involved two different test sites. The study showed the ability to reproduce test results generated at different test sites using a range of turnout composites [4]. The purpose of this paper is to describe the results of a more extensive interlaboratory study utilizing six different test sites.

Stored Energy Testing Apparatus

Figure 1 shows the stored energy testing (SET) apparatus used for this research. The testing apparatus consists of a specimen holder, a sensor assembly, data collection sensor, heat source, compressor assembly, and a data acquisition system/burn analysis program. The turnout composite specimen is loaded into the transfer tray and exposed to radiant heat supplied by a ceramic heating element for 120 s. The temperature

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of the heating element can be adjusted in one degree Celsius increments using a digital controller. The heat source is positioned 108 ± 5 mm away from the specimen holder. A water-cooled Schmidt-Boelter thermopile type sensor with a diameter of 25.4 mm measures the transmitted heat through the specimen. Water at 32.5 ± 1°C is pumped through the data collection sensor to cool it at a constant rate [3]. Five seconds after the exposure phase, the specimen is compressed by a compressor assembly for a period of 60 s. Throughout the process, the data collection thermal sensor records the heat transmitted through the layers of the test specimen. This information is analyzed using Henrique's burn model to predict the time to second degree burn for the specimen [2]. Additional information about individual components of the apparatus can be found in Refs [2] and [5].

Stored Energy Test Procedure

6 in. × 6 in. test specimens are cut from the layers that make up a turnout suit. These layers are then assembled into a composite that is used for the testing procedure.

The turnout composite is moisture preconditioned as follows: two pieces of 152 × 152 mm (6 in. × 6 in.) AATCC blotter paper are submerged in distilled water for 10 s. Both sheets of blotter paper are then run through a wringer with 30 lb on the rolls. One piece of blotter paper is placed on one side of the innermost separable layer of the composite (closest to the user) and the other piece is placed on the other side of this layer. The entire test composite is then placed into a sealed plastic bag. The air is removed from the bag and the specimen is placed in an environmentally controlled room (21 ± 3°C, 65 ± 10 % relative humidity) and allowed to equilibrate for 12–24 h [3].

The composite specimen is then removed from the bag and tested within 5 min. Specimens are exposed to 8.4 kW/m^2 (0.2 cal/cm^2-s) radiant heat for a period 120 s to simulate a firefighting environment

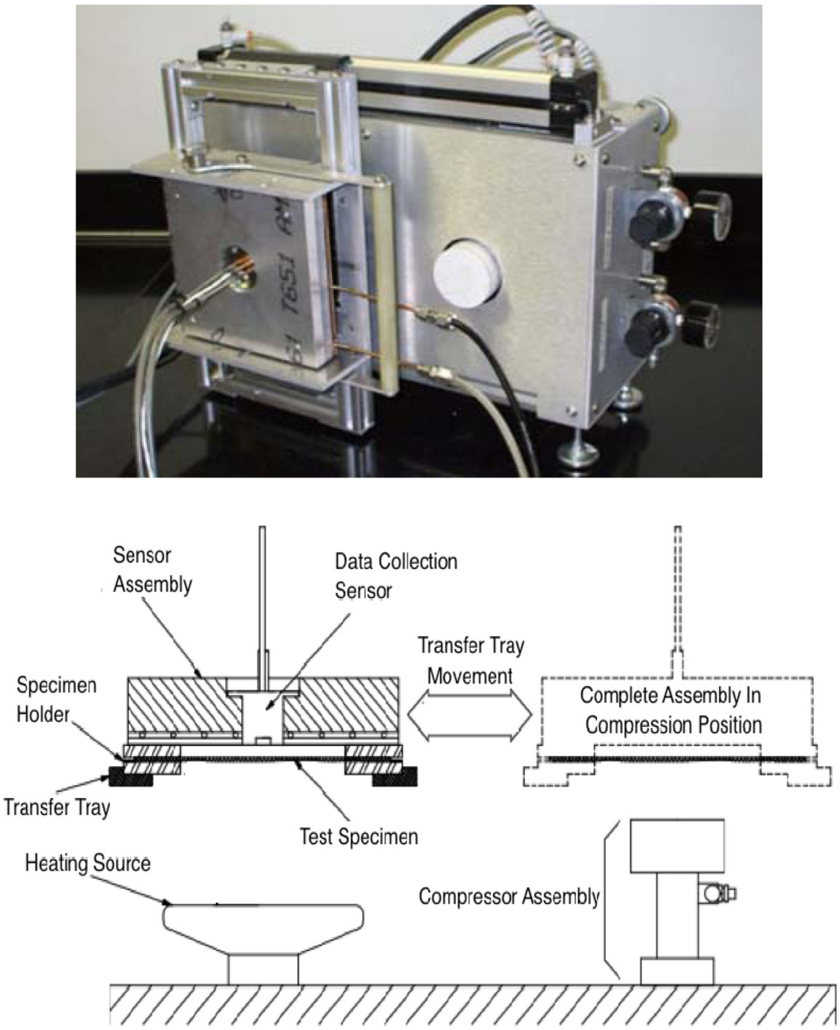

FIG. 1—*Photograph (top) and schematic (bottom) of stored energy testing apparatus [1,2].*

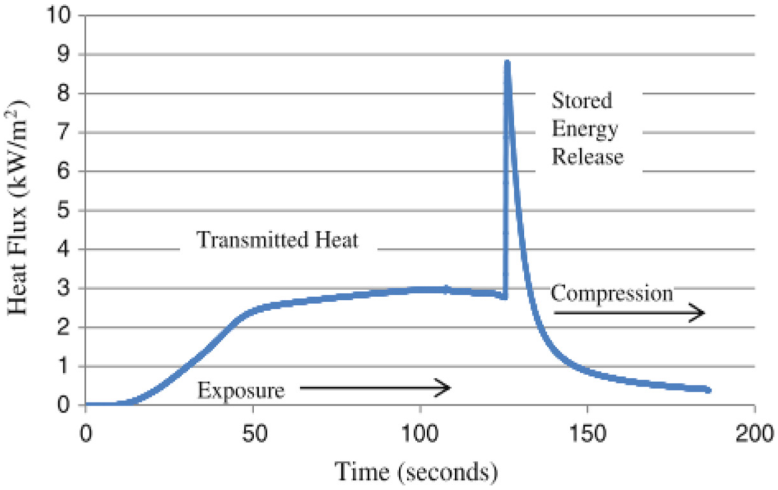

FIG. 2—*Typical measured heat flux during a stored energy test [2].*

with subflashover conditions. The data collection sensor measures the heat transmitted through the composite throughout the duration of the test. A 6.4 mm air gap (to emulate the spacer used in the thermal protective performance test [6]) is incorporated between the sensor assembly and the specimen. Two minutes after the start of the radiant heat exposure period, the turnout composite is compressed against the sensor assembly at a pressure of 13.79 kPa (2.0 psi) for a period of 60 s. The data collection sensor records the thermal energy discharged from the specimen during this time. A sample heat flux graph created by the stored energy program is shown in Fig. 2. Upon compression (≈120 s in Fig. 2), a distinct peak in the thermal flux indicates stored thermal energy discharged from the heated specimen.

Henriques burn model is used to translate the heat flux data to predict the time to second degree burn [1]. Additional description of the stored energy testing procedure can be found in Ref [3].

Interlaboratory Study

The objective of the interlaboratory study was to determine within and between laboratory variations, and assess the reproducibility of the stored energy test method. An additional objective was to identify sources contributing to variability in the test, or to the differences in results obtained at test sites.

Testing was conducted at NC State Univ. (NCSU) and at five other laboratories. Five of the six laboratories used the same testing apparatus used at NCSU. Test operators at each site were trained by NCSU on the setup of the stored energy apparatus, and on the standard testing procedures. Each laboratory site performed the testing in accordance with ASTM F2731. Testing conditions used at the test sites are summarized in Table 1.

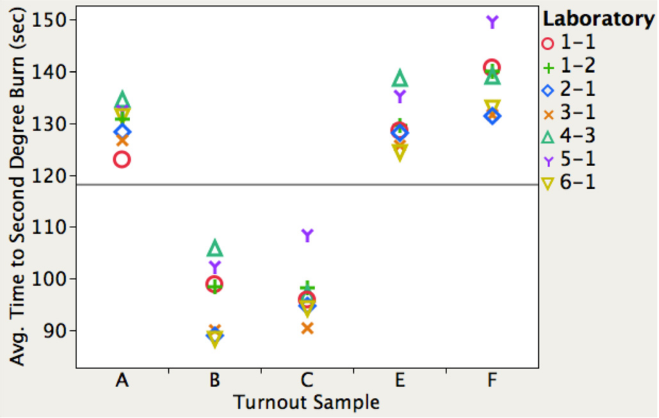

FIG. 3—*Predicted time to second degree burn (by turnout specimen).*

TABLE 1—*Test conditions for interlaboratory study.*

Preconditioning	ASTM F2731 wet preconditioning procedure [3]
Radiant heat exposure	8.5 ± 0.5 kW/m^2
Exposure period length (before compression)	120 s
Compression period length	60 s
Total data collection time	190 s

Laboratory 4 was the only participating laboratory testing site that did not use the NCSU testing apparatus. However, they used an apparatus that also conforms to the requirements specified in ASTM F2731 [3].

NCSU (laboratory site 1) conducted two separate tests. The same operator on two different apparatuses conducted these tests over a 2-day period. The first set of data was collected using the instrument provided to the other participating laboratories (except site 4). The second data set was collected using a second apparatus that also complies with the specifications of ASTM F2731 [3].

Each laboratory calibrated the heat exposure as called for by ASTM F2731. The incident heat exposure is established by exposing the thermal sensor directly to the radiant heat source for a period of at least 70 s. The flux data recorded by the sensor was averaged over a 60 s period to determine the average exposure. The temperature of the heat source was adjusted until an average thermal flux of 8.5 ± .5 kW/m^2 (0.2 ± .012 cal/cm^2-s) was achieved [3].

Test Materials

Each laboratory tested six different firefighter turnout composite specimens. The turnout composites were selected to represent materials used in NFPA 1971 compliant firefighting turnout gear [7]. All consisted of a thermal liner, moisture barrier, and outer shell layer lay-up. Some of the composites incorporated an additional outer layer, including reflective trim or fabric reinforcement attached to the outer shell. The composites used are described in Table 2.

Two different weight outer shells were utilized in the sample set. All of the turnout composites used the same breathable, vapor permeable moisture barrier. Two different thermal liners, a two layer and one layer batting design, were represented in the sample set. Three of the turnout systems were base composites, or lay-ups without an additional layer added to the outer shell material. Each base composite represented different combinations of thermal liners and outer shells. Composite B consisted of a base composite with a 6 in. × 3 in. strip of nonporous reflective trim attached across the center of the outer shell. Composite C consisted of the same base composite with a layer of nonporous fabric, used to reinforce the knee and elbow areas of turnout suits, attached to the outer shell. Composite D incorporated the

TABLE 2—*Turnout materials tested.*

Specimen	Thermal liner	Moisture barrier	Outer shell	Additional layers
A (base composite)	two-layer spunlaced para- and meta-aramid/spun yarn meta-aramid	enhanced bi-component ePTFE/meta-aramid	7.5 oz/yd^2 meta-aramid/para-aramid	none
B (base + trim)	two-layer spunlaced para- and meta-aramid/spun yarn meta-aramid	enhanced bi-component ePTFE/meta-aramid	7.5 oz/yd^2 meta-aramid/para-aramid	nonporous reflective trim
C (base + reinforcing layer)	two-layer spunlaced para- and meta-aramid/spun yarn meta-aramid	enhanced bi-component ePTFE/meta-aramid	7.5 oz/yd^2 meta-aramid/para-aramid	16 oz/yd^2 100 % para-aramid
D (base + additional outer shell)	two-layer spunlaced para- and meta-aramid/spun yarn meta-aramid	enhanced bi-component ePTFE/meta-aramid	7.5 oz/yd^2 meta-aramid/para-aramid	7.5 oz/yd^2 meta-aramid/para-aramid
E	two-layer spunlaced para- and meta-aramid/spun yarn meta-aramid	enhanced bi-component ePTFE/meta-aramid	6.0 oz/yd^2 meta-aramid/para-aramid	none
F	needle punched para- and meta-aramid/spun yarn meta-aramid	enhanced bi-component ePTFE/meta-aramid	7.5 oz/yd^2 meta-aramid/para-aramid	none

TABLE 3—*Average time to second degree burn at different laboratory sites for test turnout composites.*

	Predicted time to second degree burn, s				
Laboratory	A	B	C	E	F
1-1	123.0	98.9	95.9	128.2	140.7
1-2	130.8	98.3	98.2	129.5	140.0
2-1	128.3	88.8	94.6	128.1	131.4
3-1	126.8	89.9	90.3	125.7	131.6
4-3	134.4	105.8	96.9	138.5	138.9
5-1	132.5	102.3	108.4	135.2	149.7
6-1	131.4	88.3	94.2	124.4	133.0

same base composite with an additional layer of outer shell fabric attached to represent reinforced shoulder areas.

Each laboratory tested five replicates of each turnout composite in a random order. Predicted time to second degree burn was recorded, as was average total energy, thickness, dry weight, and wet weight of each test composite.

Results and Discussion

Table 3 compares the average predicted second degree burn time for tests conducted at the participating laboratories. In the laboratory column, the first number denotes the participating laboratory while the number after the dash denotes the testing apparatus used. Figure 3 is a graphical representation of these data. Composite specimen D (base + additional outer shell layer) is not included because no second degree burn was recorded in the allotted exposure and compression period in over 70 % of the tests performed. A detailed table of results obtained by the interlaboratory study can be found in the Appendix.

Table 4 provides a summary of statistics calculated in accordance with ASTM E691, Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method [8].

The repeatability standard deviation (s_r), an index of within–laboratory variation, was calculated using

$$s_r = \sqrt{\sum_{1}^{p} s^2/p} \qquad (1)$$

where:

p = number of labs and

s = standard deviations for each lab.

The repeatability limit (r) was estimated from the repeatability standard deviation by

$$r = 2.8 s_r \qquad (2)$$

The repeatability limit defines the maximum expected difference (with 95 % confidence) between two measurements taken by the same operator within one laboratory [8].

TABLE 4—*Precision statistics (for predicted second degree burn time).*

Specimen	$\bar{x}$ Mean, s	$S_{\bar{x}}$ Standard deviation, s	s_r Repeatability std. dev., s	s_R Reproducibility std. dev., s	r Repeatability limit, s	R Reproducibility limit, s
A	129.6	3.9	8.5	8.5	23.7	23.8
B	96.1	7.0	9.4	10.9	26.2	30.6
C	96.9	5.7	4.7	7.0	13.1	19.7
E	129.9	5.1	3.7	6.1	10.3	17.1
F	137.9	6.6	7.9	9.6	22.1	27.0
Average	118.1	5.7	6.8	8.4	19.1	23.6

The reproducibility standard deviation (s_R) indicates the between-laboratory variation. It was calculated using

$$s_R = \sqrt{(s_{\bar{x}})^2 + (s_r)^2(n-1)/n} \tag{3}$$

where:

$s_{\bar{x}}$ = standard deviations of lab average,
s_r = repeatability standard deviations, and
n = number of replications.

The reproducibility limit, calculated using Eq 4 defines the maximum expected difference (with 95 % confidence) between two measurements taken by different operators between two laboratories [8]:

$$R = 2.8 s_R \tag{4}$$

Table 4 shows that the standard deviation of the laboratory averages, a measure of overall data variation, ranged from 3.9 to 7.0 s. The largest amount of variation observed within the laboratory averages occurred in testing specimen B, the base composite turnout specimen with a layer of reflective trim added to the outer shell. Specimens that incorporate trim or reinforcement layers show a significant decrease in the predicted time to second degree burn. The composite specimen F (base composite with 1-layer needle punched thermal liner) showed the longest predicted time to second degree burn.

Test Repeatability and Reproducibility

Repeatability statistics quantify the within-laboratory variation while reproducibility statistics quantify the between-laboratory variation.

The repeatability standard deviations range from 3.7 to 9.4 s. These standard deviations range from 2.8 to 9.8 % when compared to the overall means of each test specimen. Therefore these statistics demonstrate that the test is repeatable within a single laboratory.

The reproducibility standard deviations range from 6.1 to 10.9 s. These standard deviations range from 4.7 to 11.3 % when compared to the overall means for each test specimen. These data show that there is higher variability from laboratory-to-laboratory than within a single laboratory.

Figure 4 provides a comparison of within-laboratory repeatability and between-laboratory reproducibility. The repeatability standard deviation for each composite is always slightly higher than then reproducibility standard deviation. This is an expected result because reproducibility measurements include within- and between-laboratory variation. The differences between the repeatability and reproducibility standard deviations indicate the variability added when measurements are made from laboratory to laboratory. The observation that the repeatability and reproducibility standard deviations vary from specimen to specimen indicates that the type of material tested affects the overall test variability.

The repeatability and reproducibility limits (r and R) shown in Table 4 quantify the expected maximum difference between two measurements (with 95 % certainty) taken within one laboratory and

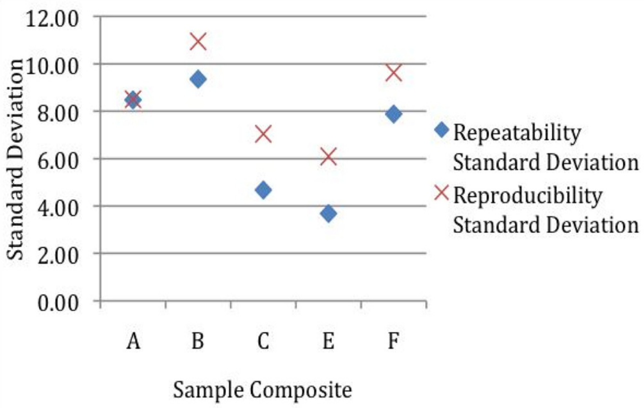

FIG. 4—*Repeatability and reproducibility standard deviations.*

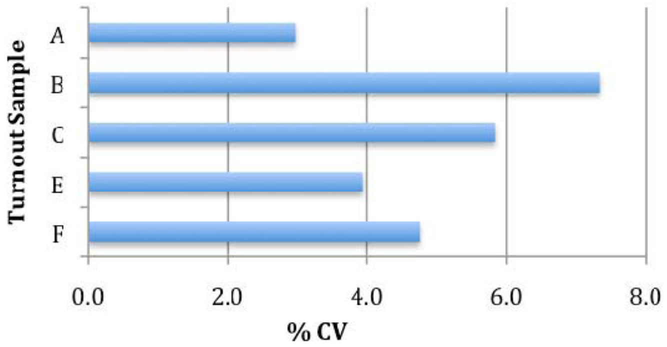

FIG. 5—*Coefficient of variation for turnout specimens.*

between laboratories [8]. These differences range from 10.3 to 26.2 s for repeatability, and 17.1 to 30.6 s for reproducibility. These values are a useful way to determine whether the amount of variation seen while performing a test in accordance with ASTM F2731 is within the range that has been established by this interlaboratory study.

To provide a mean normalized estimate of variability, the coefficient of variation of the test results obtained at each site (shown in Eq 5) was also computed. The standard deviation and means of all results across all laboratories were used to compute an overall %CV for each material:

$$C_v = \frac{\sigma}{|\mu|} \tag{5}$$

where:

σ = standard deviation and
μ = mean.

Figure 5 compares the % coefficient of variation (%CV) for each specimen. The %CV ranges from 3.0 to 7.3 % over all of the turnout composites. These statistics show that variation within the test does depend on the type of composite tested. They also indicate a significant increase in the variation of specimens when additional impermeable layers attached to the outer shell (specimens B and C). However, the %CV are all under 10 %, indicating a good overall agreement between laboratories.

Sources of Test Variability

This research investigated two possible sources of between-laboratory test variation: the variation in thermal exposure intensity and the moisture preconditioning procedure.

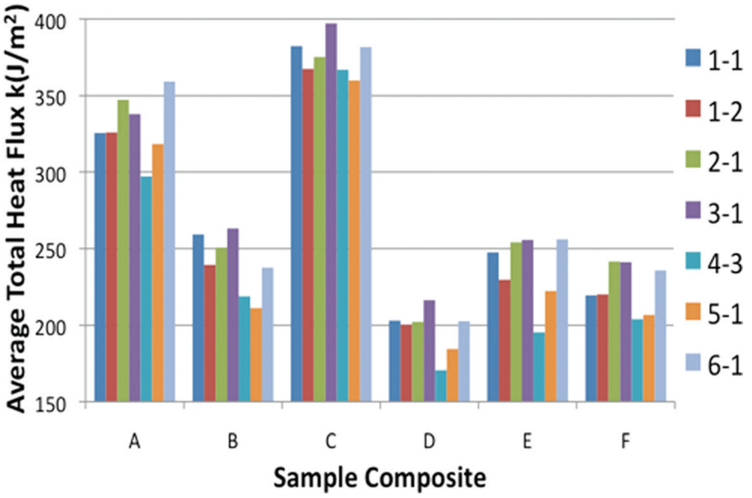

FIG. 6—*Average total energy transmitted per composite (by lab).*

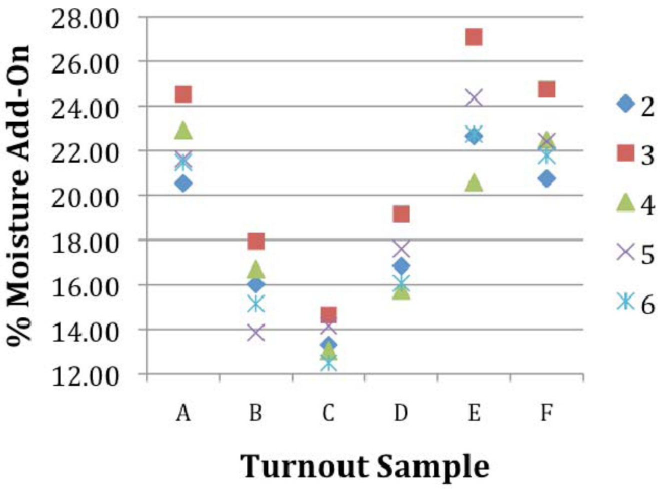

FIG. 7—*Moisture add-on versus material (per lab).*

Because the stored energy test involves low-level radiant heat exposure for a long period of time, the total energy transmitted though the sample recorded throughout the test can vary over the test duration. Figure 6 compares the average total energy through each specimen composite recorded by each laboratory. Lower total energy values are reflected in the test results by a longer predicted time to second degree burn. Higher total energy values have the opposite effect on predicted time to second degree burn (see Table 3). It can therefore be concluded that the variation in total energy contributes to differences between laboratory estimates of time to second degree burn.

Previous studies have shown that moisture present within firefighter clothing materials has a complex and pronounced effect on its the thermal protective performance [6]. The moisture preconditioning procedure therefore may be a source of laboratory-to-laboratory variation. To study this effect, the dry and wet weights (moisture preconditioned) of each turnout composite tested were recorded. To quantify sources of variability, the average % moisture add-on after conditioning was calculated for each test material at each laboratory. Figure 7 shows the variability of the % moisture add-on between laboratories.

These data indicate good agreement between labs on the amount of moisture add-on for each composite. Although slight variation are observed from specimen to specimen as well as laboratory to laboratory, this study did not show a direct correlation between moisture add-on and the time to second degree burn. However, varying amounts of moisture within the test composite undoubtedly contributes to the variation in burn times within as well as between laboratories.

Conclusions

Statistical analysis of the data obtained from this interlaboratory study showed good inter-laboratory agreement of the stored energy test method. Repeatability and reproducibility limits provide the basis on which to assess the variability of future tests run in accordance with ASTM F2731.

The study confirmed that some test variability can be attributed to differences in the turnout composite test specimens. Test specimens incorporating impermeable trim attached to the outer shell show an increase in within- and between-laboratory variability. Other sources of variability investigated were variations in thermal exposure intensity and moisture add-on after preconditioning. However, these sources of variation did not directly correlate with test values obtained, nor did they cause any outlying observations in this study.

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APPENDIX: COMPLETE RESULTS OBTAINED FROM THE INTERLABORATORY STUDY OF THE STORED ENERGY TEST METHOD ARE SHOWN IN TABLE 5 BELOW.

TABLE 5—*SET interlaboratory study data.*

	Time to second degree burn, s					
Laboratory	A	B	C	D	E	F
1-1	88.36	97.62	95.3	no burn	123.61	137.8
	128.16	95.96	100.71	no burn	128.72	132.9
	131.29	107.37	87.05	no burn	126.43	no burn
	137.95	105.02	101.13	no burn	130.47	134.53
	129.21	88.36	95.23	no burn	131.57	157.74
1-2	127.58	98.79	94.94	no burn	133.55	154.38
	127.64	88.85	96.8	143.2	128.6	132.65
	132.15	114.44	100.02	no burn	no burn	137.93
	134.92	88.11	100.62	no burn	130.92	134.95
	131.67	101.54	98.61	143.05	124.87	no burn
2-1	132.76	90.66	91.39	no burn	126.33	130.32
	122.53	90.46	93.44	no burn	129.85	131.46
	130.36	87.3	96.36	150.57	127.76	131.47
	125.15	89.18	95.45	no burn	126.19	130.35
	130.67	86.54	96.25	no burn	130.5	133.3
3-1	116.6	92.7	92.2	143.2	128.4	127.3
	130.5	88.1	89.7	138.9	128.3	133.1
	130.4	89.1	91.2	137.4	117.4	132.2
	127.6	92.3	90.8	151.9	126.8	130.9
	128.7	87.1	87.4	142.2	127.5	134.5
4-3	132.81	130.44	96.69	no burn	no burn	138.78
	135.9	123.24	92.57	no burn	137.02	138.21
	131.62	91.15	100.52	no burn	145.7	139.59
	133.13	100.78	103.35	no burn	134.84	no burn
	138.51	83.46	91.38	no burn	136.57	139.05
5-1	135.74	100.27	100.65	no burn	134.5	no burn
	no burn	104.22	112.16	no burn	136.24	no burn
	129.19	103.65	100.6	no burn	136.21	165.49
	no burn	102.61	108.41	no burn	138.59	139.88
	no burn	100.86	120.28	no burn	130.64	143.67
6-1	132.25	87.71	98.38	no burn	128.39	134.42
	130.31	89.57	90.19	no burn	119.5	131.57
	136.52	94.14	90.98	147.95	126.9	135.81
	123.91	85.06	96.35	140.31	122.1	130.26
	133.85	85.23	94.98	no burn	124.98	132.92

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