REPORT ON SHRINK TEMPERATURES*

DETERMINATION OF Ts ON SUSPENDED LEATHER SPECIMENS

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ABSTRACT

This report is divided into three parts. The first discusses the determination of leather shrink temperatures using freely suspended specimens in special holders and the results of preliminary tests of the method. Part II presents the complete statistical analysis of the shrink-temperature data submitted by nine operators on 405 specimens. The test was run on an intra- and interlaboratory basis. Part III gives a complete description of the test method and operating instructions. Five figures show the apparatus in use and the construction details.

PART I — DISCUSSION

For the past eight years in our Leather Laboratories most shrink temperatures have been determined on small die-cut specimens about 3/16″ x 2¼″. Each test specimen is inserted into a special holder which, in turn, is placed in a bracket mounted on a standard 800-ml. beaker (Fig. 1). Six holders can be mounted in the bracket; therefore, six determinations can be made during each run. The water, placed in the beaker to a fixed level (600 ml.), is heated at the rate of 3 to 3½ Centigrade degrees per minute, with adequate stirring, until the leather specimen begins to shrink. The temperature of the bath at this moment is recorded as the shrink temperature (T_s). These values are the T_s of specimens that are fixed at one end, with the other end unweighted and free to move when

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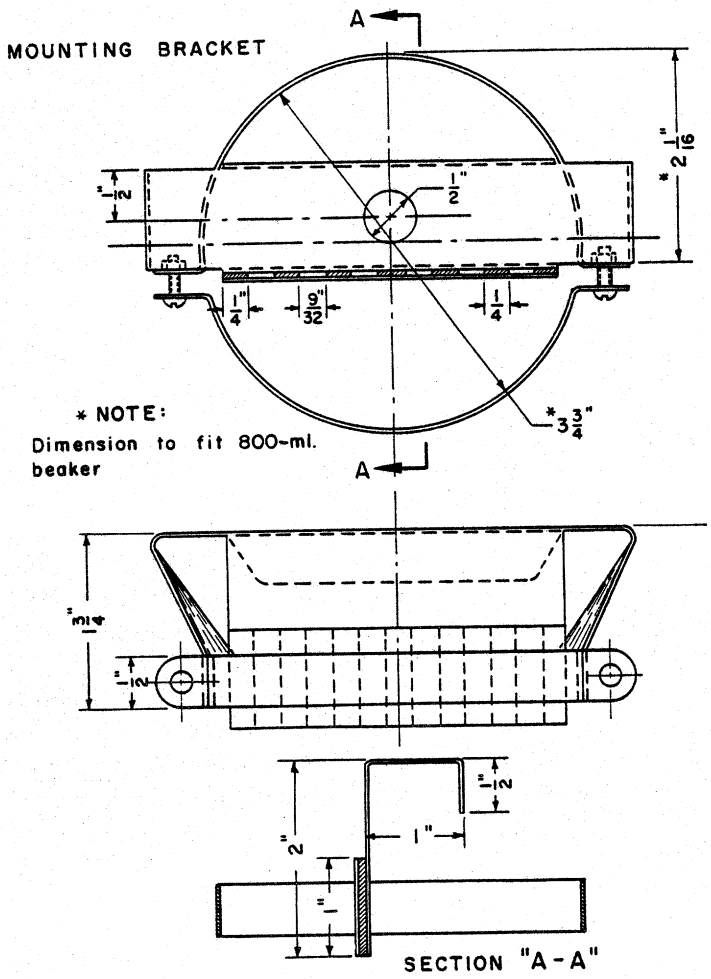


FIGURE 3.—Mounting bracket (working drawing).

in a beaker. It has been found convenient to run as many as eight specimens at one time in a glass-pipe pressure assembly complete with an internal heater, magnetic stirrer, thermometer-well, and other necessary accessories. A pressure safety device is "built-in." Barnett (3) *et al.* published their version of such apparatus in 1960. The samples can be watched during the test, and the T_s can be read exactly as in the simple beaker assembly. There is no need for special preparation of the specimens other than to make sure they are thoroughly wet. Certain precautions must be observed in handling pressure equipment. These are outlined and included with a complete set of working drawings and operating instructions, all of which are available from this laboratory on request.

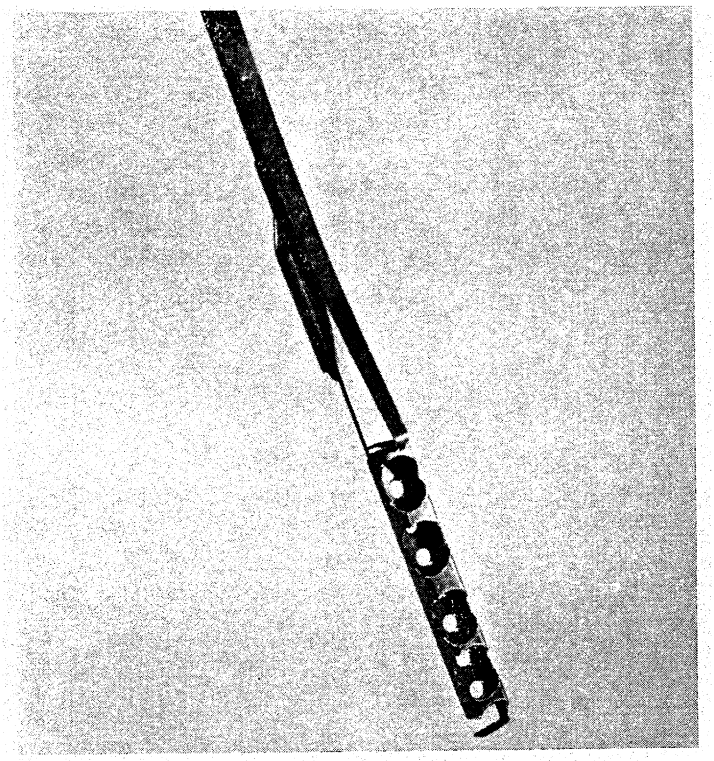


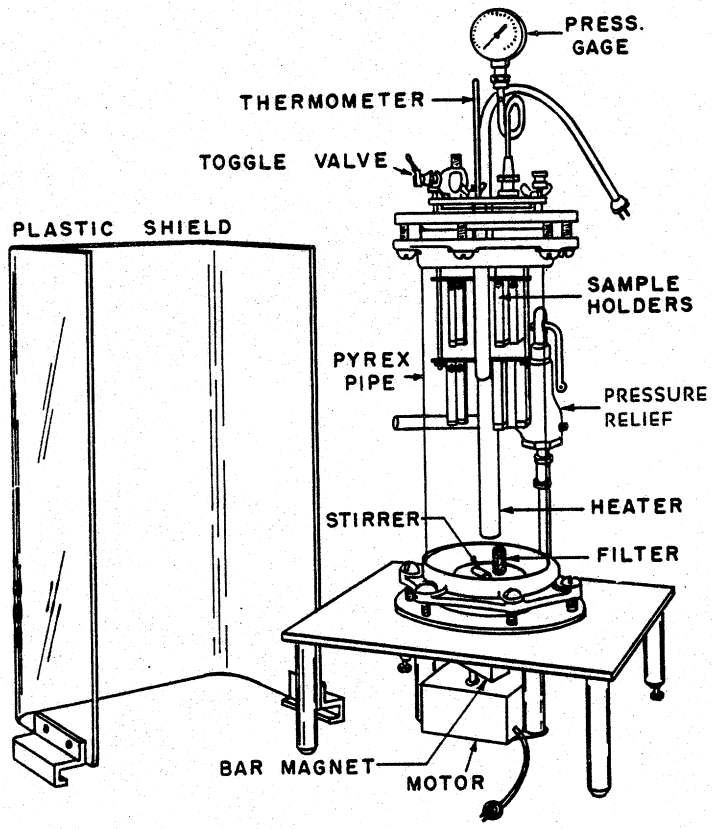
FIGURE 4.—Sample holder with stirrup base.

Many types of apparatus (2), such as the Theis meter, magnify the movement of shrinkage by mechanical means in order to detect more readily when shrinkage starts. However, such equipment can add considerable "inherent error" for which there is not always a ready correction. Such error can be much greater than that due to variability in the subjective observation such as described in this report.

The following is an interesting example: In the case of a certain garment leather that was quite flaccid when wet and somewhat difficult to handle, the T_s using the holders was 71° ± 1°C. for Operator A. For the same leather, the T_s range was 72° ± 2°C. for 96% of the observations made on 45 similar specimens by nine observers. Four of these observers had experience in using the holders; five were "first-time" observers. The same leather, cut into the proper size specimens required by Method 7011 (of KK-L-311a), using the Theis meter (with water only) and a 50-gram weight, had an average T_s of 79° ± 1°C. when run by Operator A. This is 8°C. higher than when the holders were used. The difference is appreciable and should not be discounted. It seems obvious that 71°C. is much nearer the true or at least the practical shrink temperature.

With the advent of washable leather the need for a "true" value takes on added significance.

The T_s value using the Theis meter would probably have been lower than 79°C. if less weight were used to hold the sample in place. As a matter of fact, in this particular test the average stretch due to the 50-gram weight was 7.4% while the water was cold, and the stretch increased slowly to 13.8% as the water was heated. This is 3.8% more than the Federal Specifications allow; therefore the test would have to be repeated using a smaller weight. To find the proper weight to use with each of a series of specimens could become quite a chore. Certain types of leather are affected less than others by the weights attached. For instance, one set of specimens of side leather, when checked on the Theis meter, showed practically no stretch with the 50-gram weight attached; however, the T_s was higher than the T_s recorded for a freely suspended specimen.



SHRINK - TEMPERATURE PRESSURE APPARATUS

FIGURE 5.—Pressure apparatus for shrink temperature.

A preliminary testing program at EURDD involving only persons with experience in observing shrink temperatures of leather specimens in holders was completed before an interlaboratory test was started. Some of the results of the preliminary tests were as follows:

(*a*) In a series of 200 determinations on four leathers by five observers: For all observers, the 95% confidence limits of the average T_s varied from their average by ± 0.28.

(*b*) A second series of 150 observations was made on specimens of three leathers by four operators working in pairs. Each observer read the shrink temperature of the same specimen independently of his partner, with an opaque screen between them. Partners were changed so that each person worked with every other observer for a total of 15 sets of observations. The results of this test indicated that there was no significant difference between operators in 13 sets; and in the other two sets there was a significant difference, but the actual value was not important from a practical standpoint.

The results of these two tests, in addition to its other desirable features, indicated that the method was worthy of consideration as a tentative (standard) method.

The interlaboratory test was done on a total of 450 specimens, 45 for each observer. There were four observers from EURDD and six observers from four other laboratories. To the latter, the specimens were sent along with the necessary directions and equipment. They had no previous experience with the method or equipment. The specimens were numbered and randomized for the test. The purpose was to test two types of holders on four types of leather with ten observers on an inter- and intralaboratory basis. When all the results were received, the results from one operator had to be dropped because of atypical data. The data (Table III) submitted by the remaining nine observers were tested statistically. A brief summary follows. The complete statistical analysis of the data is given in Part II.

Briefly, the results showed that all the operators were able to repeat themselves within acceptable limits on the five replications of each test. The variability between the experienced EURDD operators was much lower than the variability between the new operators. This is probably due to years of practice. For all the experienced observers the 95% confidence limits of the average T_s varied from their average by ± 0.32°C. For a similar calculation on all the new observers from the other laboratories the variation from their average was ± 1.4°C.

There is not much T_s difference between using the straight or stirrup holders although the latter tended to give slightly lower T_s readings (start of shrinkage more easily detected) except in the case of extremely flaccid specimens which were difficult to hold on the stirrup. The straight holders were much easier to load.

Based on the results of this test, the number of specimens necessary to estimate the average shrink temperatures within 1°C. with a 95% probability would be:

for experienced operators	4
for new operators	8

Additional practice by the new operators would probably bring this value down considerably.

PART II — STATISTICAL ANALYSIS OF SHRINK TEMPERATURE DATA

The statistical analysis was completed in two sections. The first (A) is the result of four operators, four types of skins, and two types of holders. There were five replications on all of these combinations, and this work was completed here at EURDD (experienced observers).

The second (B) was based on the results from three collaborators, five operators, four skins, and two types of holders. These data were also replicated five times. The types of skins and holders in this case are the same as those in the first section completed by EURDD. (One collaborator which furnished data for one operator was not used in this analysis because of atypical results.)

A. Statistical analysis of EURDD data

From the analysis of variance in Table I below, it can be seen that there was a highly significant difference between operators and between skins and a significant difference between types of holders.

TABLE I

	SS	df	ms	F
Total	10,202.	159		
1. Operators	22.55	3	7.52	8.17**
2. Skins	9,956.60	3	3,318.87	3,607.47**
3. Holders	4.22	1	4.22	4.59*
4. 1 × 2	54.85	9	6.09	6.62**
5. 1 × 3	1.93	3	.64	
6. 2 × 3	14.88	3	4.96	5.39**
7. 1 × 2 × 3	29.77	9	3.31	3.59**
Error	117.20	128	.92	

*Significant at the 5% level.
**Significant at the 1% level.

The averages of shrink temperatures for operators and the results of Duncan's Multiple Range Test are as follows:

Operators	Averages
2	82.38
1	81.65
4	81.60
3	81.38

Even though the variability between operators was highly significant, the actual range of the averages is only one degree; and since the individual shrink temperatures were measured to the nearest degree, this may have little practical significance.

The average by types of skins and the results of Duncan's Multiple Range Test are as follows:

Skins	Averages
D	89.75
C	89.05
A	76.80
E	71.40

The averages by types of holders and the results of Duncan's Multiple Range Test are as follows:

Holders	Averages
Straight	81.91
Stirrup	81.59

It can be seen that the difference is only .32 degrees, and this is another example where the difference is statistically significant but has little practical significance.

The interaction between skins times holders was highly significant, and this was due to the fact that the straight holder resulted in a higher temperature than the stirrup holder for skins A, C, and D, while the straight holder resulted in a lower temperature than the stirrup holder for skin E. These averages and the result of Duncan's Multiple Range Test are as follows:

	Holders	
Skins	Straight	Stirrup
D	89.90	89.60
C	89.60	88.50
A	77.05	76.55
E	71.10	71.70

B. Statistical analysis of data from three collaborators

Table II shows the results of the analysis of variance.

TABLE II

	SS	df	ms	F
Total	13,788.875	199		
1. Operators	244.350	4	61.088	31.50**
Locations	185.537	2	92.768	47.84**
between operators within locations	58.813	2	29.406	15.16**
2. Holders	41.405	1	41.405	21.35**
3. Skins	12,999.335	3	4,333.112	2,234.71**
4. 1 × 2	32.770	4	8.192	4.225**
5. 1 × 3	74.890	12	6.241	3.219**
6. 2 × 3	55.255	3	18.418	9.499**
7. 1 × 2 × 3	36.470	12	3.039	1.567
Error	304.400	157†	1.939	

†Three Estimates.
**Significant at the 1% level.

The difference between operators was highly significant. This was broken down by locations and by operators within location, and both sources of variability were highly significant. The averages by operators and the results of Duncan's Multiple Range Test were as follows:

Operators	Averages
2	83.60
1	82.40
3	82.38
4	81.48
5	80.275

It can be seen that the range for operators is 3.325 degrees, which is over three times that found for EURDD, and this difference would appear to have some practical significance.

There was a highly significant difference between holders, and the averages are as follows:

Holders	Averages
Straight	82.48
Stirrup	81.57

This difference is .91 degrees, which again is about three times that found for EURDD, and this would appear to have some practical significance.

The difference between skins was highly significant. The averages and the results of Duncan's Multiple Range Test are as follows:

Skins	Averages
D	90.60
C	89.10
A	76.84
E	71.56

The interaction of holders times skins was highly significant. The averages appear as follows:

	Holders	
Skins	Straight	Stirrup
D	91.20	90.00
C	90.28	87.92
A	77.16	76.52
E	71.28	71.84

It can be seen that the straight holder resulted in a higher temperature than the stirrup holder for skins A, C, and D, while the straight holder had a smaller value than the stirrup holder for skin E. These results are essentially the same as those found by EURDD.

Summary and conclusions

The following table shows the difference between holders for EURDD and the collaborators:

	Straight	Stirrup
EURDD	81.91	81.59
Collaborators	82.48	81.57

It can be seen from this table that there is essentially no difference between EURDD and the collaborators using stirrup holder. The difference for the straight holder is .57 degrees, which is statistically significant but may not have much practical significance.

The real statistical significance and practical significance appears to be in the error term in Table I and Table II. It can be seen that this difference is slightly larger than a factor of two. This error term, in both cases, represents the unexplained variability in the two separate sets of data. It is a function of this error term that determines the sample size in future experiments for a certain desired degree of accuracy with a certain probability of achieving this accuracy. This formula for the sample size is as follows:

$$n = \frac{t^2 s^2}{(x - \bar{x})^2}$$

Thus, if it was desired to determine the sample size necessary to estimate the average shrink temperature within one degree with a 95% probability of being sure of this, one would substitute in the formula (in the case of EURDD) as follows:

$$n = \frac{t^2 s^2}{(x - \bar{x})^2} = \frac{(2)^2(1)}{(1)^2} = 4$$

Where $t = 2$, $s^2 = 1$, and the desired degree of accuracy $(x - \bar{x}) = 1$, this would result in the value of $n = 4$. In the case of the collaborators, the substitution would be the same for t and $(x - \bar{x})$, but the value of s^2 would be 2, which would result in a sample size of $n = 8$, which is twice the sample size necessary for this desired degree of accuracy in the case of EURDD.

EURDD and the collaborators tested Skin B using only the straight holder, and because of this it was not included in the general analysis. In the case of EURDD the analysis was done on just this one skin. There was no significant difference between replications or between operators; however, this can be attributed, at least in part, to the fact that an interaction term had to be used for the error term which would tend to decrease the sensitivity of the test. Because of these reasons, the analysis of skin B was not computed for the collaborators.

In order to determine the homogeneity of the variance by type of holder and by EURDD and the collaborators, these variances were computed and appear in the following table:

	Straight	Stirrup
EURDD	67.90	61.18
Collaborators	66.09	62.68

It can be seen that these differences are quite negligible between EURDD and the collaborators. However, there is some slight difference between these variances in the case of type of holder.

TABLE III

INTERLABORATORY TEST DATA
SHRINK TEMPERATURES, °C.

Leathers*								
A	B	C	D	E	A	C	D	E
Straight Holders					Stirrup Holders			
Experienced observers								
77	80	90	89	72	75	89	89	74
77	81	91	90	71	75	89	89	74
75	79	90	89	72	75	86	89	73
76	79	88	89	72	74	90	87	73
76	78	90	91	71	75	89	90	73

TABLE III *(Continued)*

INTERLABORATORY TEST DATA

SHRINK TEMPERATURES, °C.

Leathers*								
A	B	C	D	E	A	C	D	E
Straight Holders					Stirrup Holders			
Experienced observers *(Continued)*								
77	82	88	90	71	77	88	88	71
78	82	89	89	71	78	88	88	71
78	79	90	89	70	77	87	88	72
78	80	88	90	71	77	89	89	71
77	79	88	89	71	76	88	89	71
78	80	90	87	72	78	89	93	72
78	79	89	90	71	78	88	92	71
77	80	92	89	72	77	91	90	70
78	79	90	92	71	77	90	92	71
78	81	90	91	72	77	89	92	71
77	79	89	93	70	77	87	90	71
76	80	88	92	71	77	87	90	72
77	80	92	88	70	76	90	89	72
76	80	91	91	71	77	88	90	71
77	80	89	90	70	78	88	88	70
Inexperienced observers								
79	84	89	90	73	78	86	88	74
78	86	89	90	72	78	91	94	73
76	84	89	90	73	79	87	90	75
77	84	90	85	73	78	89	91	74
78†	84	89	90	72	78	87	91	73
78	80	92	94	73	79	90	91	71
78	79	92	94	72	77	89	93	73
78	80	92	95	73	78	90	92	72
79	80	92	94	72	78	91	93	73
79	81	92	93	73	77	89	92	71
78	82	90	92	71	76	85	88	74
78	83	93	93	73	74	87	90	71
78	82	88	91	71	76	88	93	73
78	82	91	92	72	76	88	91	72
78	82	91	93	72	78	92	88	72
77	79	89	92	70	76	86	86	71
77	81	88	90	70	76	89	86	70
76	80	89	90	70	76	90	86	71
79	80	91	91	69	77	89	94	71
77	80	90	91	71	77	89	91	71

ing the temperature at the rate of 3° to 3½°C/minute (see Note 1). Read both thermometers every 3 minutes and record the temperature of each as well as the difference between the top and bottom (making allowance for differences shown by calibration). The stirring is adequate if none of the calculated differences exceed 1.0°C.

Note 3.—Distilled water need not be used with specimens taken during wet processing of skins.

Note 4.—Cut wet specimens from samples that exhibit excessive swelling when "wet-back."

Note 5.—If samples shrink below 60°C., start the test with water at least 10°C. below the shrink temperature.

Note 6.—Leave T_s space blank (on Data Sheet) if T_s was not carefully observed for any reason, or if there was a known deviation from the prescribed procedure. Repeat observation with substitute specimen if one was provided.

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