Designation: D 4204 – ~~95~~00

Standard Practice for Preparing Plastic Film Specimens for a Round-Robin Study[1]

This standard is issued under the fixed designation D 4204; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This practice covers the preparation of test sets of plastic film specimens for subsequent use in an interlaboratory round-robin study to evaluate the precision of a test method.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

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[1] This practice is under the jurisdiction of ASTM Committee D~~-~~20 on Plastics and is the direct responsibility of Subcommittee D20.19 on Film and Sheeting . ~~This revision includes an ISO equivalency statement.~~

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***A Summary of Changes section appears at the end of this standard.**

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *film specimen*—one piece of a sample obtained by cutting across the width of the sample and to a length such that one test specimen can subsequently be prepared.

NOTE 2—For any sample in a laboratory, n_1 test specimens are tested to produce a single test result in a short-time period, while replicate test results are obtained over a longer time period. Thus, there are within-laboratory components of variability for both short-term and long-term testing. This practice calls these within-day and between-day components of variability, inasmuch as round-robin protocols often specify that replicate test results be obtained on different days.

3.1.2 *sample*—a quantity of film of a width appropriate to the test method under study and of a length sufficient to yield the total number of film specimens needed for the planned round-robin study.

3.1.3 *test result*—the value (usually, the arithmetic average) of the property derived from one test unit.

3.1.4 *test set*—a group of several film specimens, in a number greater than that specified for a test unit.

3.1.5 *test specimen*—the unit, usually of specified dimensions, that is to be cut from one film specimen and tested, to produce one value of the property, or properties, by the test method under study.

3.1.6 *test unit*—a specified number of film specimens from which an equal number of test specimens is to be prepared and tested in a short-time span to yield one test result for each property.

3.2 *Symbols: ~~Symbols:~~*

RR = round-robin study,
p_1 = number of laboratories participating in the RR,
q = number of samples to be used in the RR,
r = number of replicate test results to be obtained on different days by each laboratory for each sample in the RR (Note 1),
n_1 = specified number of film specimens in a test unit,
n_2 = number of additional film specimens in each test set,
p_2 = number of additional "latent" laboratories provided for in the specimen preparation procedure,
L_1 = film-specimen length appropriate for preparing one test specimen,
L_2 = length of film from a sample from which can be obtained $(p_1 + p_2)$ film specimens; $L_2 = (p_1 + p_2)(L_1)$,
SD = component standard deviation for a single source of variability for one given sample,
S_1 = SD for within-laboratory within-day variability of a test value,
S_2 = SD for within-laboratory between-day variability of a test result,
S_3 = SD for between-laboratory variability of a test result,
S_4 = SD for within-sample variability,
S_r = within-laboratory standard deviation of a single test result for one given sample on any day, and
S_R = between-laboratory standard deviation of a single test result for one given sample on any day.

4. Significance and Use

4.1 This practice is intended to assist task groups ~~entering into~~ participating in a round-robin study wit~~o~~h the prepar~~e~~ation of test sets of film specimens from film samples in the form of rolls on a cardboard core.

4.2 This practice assumes that the essential features of the round-robin protocol have already been established by following the guidance of Practice E 691. In particular, it is assumed that the following are known: (*1*) the number of film samples to be used, (*2*) the number of participating laboratories, (*3*) the number of replicate test results to be generated by each laboratory for each sample, and (*4*) the number of test specimens required to yield one test result for each sample.

4.3 In accordance with this practice, samples are partitioned into test sets so that real within-sample variability will not unduly distort the conclusions drawn from statistical analyses of the data generated in the round-robin study.

5. Sample Selection

5.1 Select q samples that would be expected to be uniform (for which S_4 would be small). The larger the value of S_4, the greater will be the adverse effect upon conclusions drawn from round-robin data regarding test method precision.

5.1.1 For any sample, the total observed variability will always contain the component S_4. In the typical study, S_4 is not estimated separately; the result is an overestimation of one or more of the components of variability (S_1, S_2, and S_3) that the study is designed to estimate. Because of this, S_4 is a nuisance factor to be dealt with as conveniently as possible.

5.1.2 It is preferable to confound S_4 with within-laboratory components of variability, S_1 and S_2, and to obtain an estimate of S_3 that is not inflated by within-sample variability, S_4. This practice is intended to accomplish this. In most cases, in accordance with this practice, S_4 is confounded only with S_1, so that the estimate of S_2 is also not inflated by within-sample variability, S_4. The confounding of S_4 with only S_1 is equivalent to a completely random selection of all film specimens from the film sample.

[2] *Annual Book of ASTM Standards,* Vols 06.03 and 14.02.

5.1.3 The best source of samples is from commercial ~~extrusion operations. If one~~ or ~~more samples must be from a noncommercial source (for example, a small-scale~~ laboratory extrusion operation~~),~~s that have demonstrated the ~~preparing laboratory should obtain backup data~~ capability to produce film under conditions t~~h~~at have shown that appreciable systematic trends in the level of the property, or properties, to be measured did not occur as the sample was being fabricated.

5.2 Before preparing test sets as described in Section 6, have one laboratory test n_1 test specimen from each sample. If the range of property levels thus found is narrower than is deemed appropriate for the study, it may be desirable to obtain one or more additional samples, to replace one or more of the q samples collected initially.

6. Procedure

6.1 Select a value of n_2 for the RR. In view of the way test sets are made up, as described subsequently, there will always be two "sacrifice" film specimens in a test set, one on top and one on bottom of the stack, that serve to protect the integrity of the film specimens in between. These two are not to be used; always take test specimens from film specimens between the top and bottom film specimens in the test set. In addition, it is usually advisable to include a minimum of one or two extra film specimens in each test set, in the event a laboratory finds an occasional film specimen with a defect which, properly, should not be used; then, when a defective film specimen is found, it can be discarded and an additional film specimen, already at hand, can be substituted. Thus, n_2 must be at least 2 and, preferably would be 3 or 4. The total number of film specimens in a test set would then be $(n_1 + n_2)$, from which one test result would be obtained.

6.2 Select a value of p_2 for the RR. On a practical basis, it is advisable to set p_2 equal to roughly one half of p_1. Then, if mailed test sets are lost in transit, if after-the-fact recheck testing is needed in one or more laboratories, or if there are late-entering laboratories into the study, additional test sets will be at hand, as needed. For test set preparation, consider the total number of laboratories to be $(p_1 + p_2)$.

6.3 Select a value of L_1 appropriate for the test to be conducted.

6.4 Calculate L_2 as follows:

$$L_2 = (p_1 + p_2)\,(L_1) \tag{1}$$

6.5 For one of the q samples:

6.5.1 Unwind and cut off successive lengths of film, each of length L_2. Lay out the first r lengths, side by side, on a clean flat surface. Place succeeding cut lengths on top of the first r cut lengths, to form r stacks of multilayers of film lengths. Build each of the r stacks first up to 2 layers each, then up to 3 layers each, etc. Continue until each of the r stacks contains $(n_1 + n_2)$ layers of film.

6.5.2 From the first of the r stacks, prepare $(p_1 + p_2)$ test sets, each containing $(n_1 + n_2)$ film specimens. Do this by starting at one end of the stack and making successive cuts through all $(n_1 + n_2)$ layers at incremental distances of L_1 along L_2. As each test set is obtained, package and label the test set appropriately and mark the package with a sequential number [1, 2, 3, ..., $(p_1 + p_2)$]. Keep the packaged test sets from the first of the r stacks segregated. Call this a *collection* of $(p_1 + p_2)$ test sets.

6.5.3 Repeat 6.5.2 for each of the r stacks, in turn, to form r segregated collections.

6.5.4 By use of random numbers, select one packaged test set from the first collection. Repeat for each of the r collections, to form one *group* of r replicate test sets for testing the first sample in one laboratory.

6.5.5 Continue the selection process of 6.5.4, to end up, finally, with $(p_1 + p_2)$ groups, each containing r replicate test sets, for testing the first sample in $(p_1 + p_2)$ laboratories.

6.6 Repeat 6.5 for each of the q samples.

6.7 Make an *assembly* by combining one group from each of the q samples; repeat this process to obtain $(p_1 + p_2)$ assemblies for testing all q samples in $(p_1 + p_2)$ laboratories.

6.8 Distribute p_1 assemblies to the p_1 participating laboratories. Retain the remaining p_2 assemblies in case they are needed subsequently.

7. Precision Estimates

7.1 After the round-robin study has been completed, analyses of variance of the data will provide estimates of component standard deviations, S_1, S_2, and S_3, for each of the q samples.

7.2 Estimates of within-laboratory and between-laboratory variability for each sample, consistent with the use of symbols in Practice E 691, are arrived at as follows. In the following equations, n is the standard number of replicate test specimens required for one test result, as dictated by the test method, which is not necessarily the same as the value of n_1 used in the round-robin study.

~~$$S_r = (S_1^2/n + S_2^2)^{½} \quad (2)$$~~

$$S_r = (S_1^2/n + S_2^2)^{\frac{1}{2}} \quad (2)$$

$$S_L = S_3$$

~~$$S_R = (S_r^2 + S_L^2)^{½}$$~~

$$S_R = (S_r^2 + S_L^2)^{\frac{1}{2}}$$

8. Keywords

8.1 film; round robin; testing

SUMMARY OF CHANGES

This section identifies the location of selected changes to this practice. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this practice. This section may include descriptions of the changes, or the reasons for the changes, or both.

D 4204 – 00:
(*1*) Editorial changes in 4.1, 5.1.3, 6.1 and 6.2.
(*2*) Added Keywords section.