Designation: D4204 – 16

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

This standard is issued under the fixed designation D4204; 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.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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, the specified number of film specimens in a test unit (n_1) 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 individual piece of film to be tested, 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 *Abbreviations:*

RR = round-robin study
q = number of samples to be used in the RR
r = total number of film assemblies that will be needed for each lab to complete the necessary testing
n_1 = specified number of film specimens in a test unit
n_2 = number of additional film specimens in each test set
p_1 = number of laboratories participating in the RR
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 = total length of film necessary to produce samples fro participating plus latent laboratories; $L_2 = (p_1 + p_2)(L_1)$
SD = standard deviation for a single source of variability for one given sample
S_1 = standard deviation for within-laboratory within-day variability of a test value
S_2 = standard deviation for within-laboratory variability of a test result
S_3 = standard deviation for between-laboratory variability of a test result
S_4 = standard deviation for within-sample variability
S_r = standard deviation of a within-laboratory single test result for one given sample on any day
S_R = standard deviation of a between-laboratory single test result for one given sample on any day

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.19 on Film, Sheeting, and Molded Products.

Current edition approved Nov. 1, 2016. Published November 2016. Originally approved in 1982. Last previous edition approved in 2012 as D4204 - 12. DOI: 10.1520/D4204-16.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

4. Significance and Use

4.1 This practice is intended to assist task groups participating in a round-robin study with the preparation of test sets of film specimens from film samples in the form of rolls on a 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 E691. 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 the number of samples q to be used in the round-robin (RR) study that would be expected to be uniform for which the standard deviation for within-sample variability (S_4) is expected to be small. The larger the value of standard deviation for within-sample variability (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 of standard deviation for within-sample variability (S_4). In the typical study, sample standard deviation for within-sample variability (S_4) is not estimated separately; the result is an overestimation of one or more of the components of variability: standard deviation for within-laboratory within-day variability (S_1), standard deviation for within-laboratory between-day variability, (S_2), standard deviation for between-laboratory variability (S_3) that the study is designed to estimate. Because of this, standard deviation for within-sample variability (S_4) is a nuisance factor to be dealt with as conveniently as possible.

5.1.2 It is preferable to confound standard deviation for within-sample variability (S_4) with the within-laboratory components of variability, of within-day and between-day components of variability (S_1 and S_2), and to obtain an estimate of between-laboratory variability (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, the standard deviation for within-sample variability (S_4) is confounded only with within-laboratory within-day variability (S_1), so that the estimate of within-laboratory between-day variability (S_2) is also not inflated by the standard deviation for within-sample variability, (S_4). The confounding of standard deviation for within-sample variability (S_4) with only within-laboratory within-day variability (S_1) is equivalent to a completely random selection of all film specimens from the film sample.

5.1.3 The best source of samples is from commercial or laboratory extrusion operations that have demonstrated the capability to produce film under conditions that 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 the specified number of test specimens (n_1) from each sample. If the range of property levels thus found is narrower than is deemed appropriate for the study, it is desirable to obtain one or more additional samples, to replace one or more of the number of samples to be used in the RR (q) collected initially.

6. Procedure

6.1 Determine the number of extra film specimens to be used for each test (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 a defective film specimen, it can be discarded and an additional film specimen, already at hand, can be substituted. Thus, the extra number of film specimens (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 the specified number of film specimens plus the determined number of extra film specimens ($n_1 + n_2$), from which one test result would be obtained.

6.2 Determine the number of extra film sets that will be prepared (p_2) for the RR. On a practical basis, it is advisable to set p_2 equal to roughly one half of the number of laboratories participating in the RR (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 Determine the length of the film specimen appropriate for one test to be conducted (L_1).

6.4 Calculate the total length of film necessary for the participating labs and latent labs (L_2) as follows:

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

6.5 To produce one sample of the total number of samples q:

6.5.1 Unwind and cut off successive lengths of film, each of length L_2, as depicted in the equation above. Lay out the length, on a clean flat surface. Place succeeding cut lengths on top of the first cut length, to form a multilayer stack of film. Build the stack first up to two layers, then up to three layers each, etc. Continue until the stack contains the number of pieces of film necessary to complete the testing plus the predetermined extra pieces of film ($n_1 + n_2$). Do this with each roll of film that is to be included in the round robin.

6.5.2 From the first of the stacks, prepare the total number of test sets ($p_1 + p_2$). Do this by starting at one end of the stack and making successive cuts through all layers at the predetermined film length for one test specimen (L_1). As each test set

is obtained, package and label the test set appropriately and mark the package with a sequential number. Keep the packaged test sets from the individual stacks segregated. Call this a *collection* of test sets.

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

6.5.4 By use of random numbers, select one packaged test set from the first collection. Repeat for each of the remaining collections, to form one group of 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 E691, 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.

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

$$S_L = S_3$$

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

8. Keywords

8.1 film; round robin; testing

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4204 - 12) that may impact the use of this standard. (November 1, 2016)

(1) Revised 5.2.