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Standard Test Method for Basic Storage Stability of a Mechanical Pump Dispenser[1]

This standard is issued under the fixed designation D6654; 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 test method covers the determination of the basic storage stability of a mechanical pump dispenser with a product.

1.2 This test method covers an evaluation of the weight lost during storage of mechanical pump dispensers (spray or flow types) with a product.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2063 Test Methods for Measurement of Torque Retention for Packages with Continuous Thread Closures

D3890 Test Method for Number of Strokes to Prime a Mechanical Pump Dispenser

D4336 Test Methods for Determination of the Output Per Stroke of a Mechanical Pump Dispenser

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

3. Significance and Use

3.1 Determining the storage of a mechanical pump dispenser for consumer usage. Products of consumer usage are of the personal care, household, insecticides, food, automotive, and institutional nature. Pharmaceutical and cosmetic products including perfume are not covered under this practice.

4. Apparatus

4.1 *Containers,* that will allow the mechanical pump dispenser to be affixed to them (see Note 1) and also be capable of containing product for a period of time at various environmental conditions (see Note 2).

NOTE 1—If possible, the actual container to be marketed should be used.

4.2 *Product,* a sufficient amount to fill the number of containers in accordance with 4.1, reference 10.2.

4.3 *Balance,* with direct reading to 0.01 g. Top loading or analytical style is recommended.

4.4 *Environments,* where the mechanical pump dispenser and product will be kept during the test period.

4.4.1 *Ambient Area,* maintained at 23 ± 3°C (73 ± 5.4°F).

4.4.2 *Oven (45°C),* maintained at 45 ± 3°C (113 ± 5.4°F).

NOTE 2—The oven temperature can be changed according to product formulation. If different temperatures are used, this should be noted in the test report discussed in 11.1.

4.4.3 *Cycle Chamber,* optional, alternating 5 to 50°C (41 to 122°F) every 24 h.

4.5 *Oven-Safe Tray,* with absorbent paper towels laid in the tray.

4.6 *Food Coloring or Dye,* optional.

5. Precautions

5.1 Appropriate handling considerations should be given to flammable, toxic, caustic, or other potentially hazardous material used. When testing at any temperature, safety should be the number one consideration and special attention should be used on the flash points of the products tested.

6. Sampling

6.1 Based upon the desired precision, sampling shall be performed in accordance with Practice E122.

6.2 In the absence of any special sampling plan, performance shall be based on not less than the number of representative specimens exposed to any of the environmental conditions as specified in Section 9.

7. Test Specimens

7.1 Test specimens shall be clean, dry, and previously unused mechanical pump dispensers.

[1] This test method is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.33 on Mechanical Dispensers.

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[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.

8. Conditioning

8.1 If possible, condition the test specimens at 23 ± 3°C (73 ± 5.4°F) for not less than 4 h. If the test specimens are not conditioned at the recommended temperature and humidity, this should be noted in the test report discussed in section 11.1.

8.2 Test pumps should be tested no sooner than 24 h after assembly when possible. If pumps are not conditioned at the recommended time, this should be noted in the test report discussed in 11.1.

9. Test Specimen Preparation

9.1 Prepare a minimum of twelve unprimed mechanical pump dispensers with product and containers to be used for evaluation.

9.1.1 Label all of the assembled units with the product name, any safety hazards for the product, name of the technician completing the test, and the test number or means of identifying the test along with the following :

9.1.1.1 Minimum three units labeled, "Upright, Ambient."

9.1.1.2 Minimum three units labeled, "Upright, Oven 45°C"

9.1.1.3 *Optional*—Minimum three units labeled, "Upright, Cycle Chamber."

9.1.1.4 Minimum three units labeled, "On-Side, Ambient."

9.1.1.5 Minimum three units labeled, "On-Side, Oven (45°C)."

9.1.1.6 *Optional*—Minimum three units labeled "On-Side, Cycle Chamber."

NOTE 3—Since the output of this test is weight loss of product for the total package of container with a mechanical pump dispenser, it may be advantages for a control package of the test container with a standard closure to be included at each condition so that the effect of the mechanical pump dispenser may be determined.

10. Procedure

10.1 If the data is to be presented as gross weight lost only, then proceed to 10.2. If the data is to be presented as weight lost as a percentage of the weight of the product, weigh each container with its intended pump at the beginning of testing and record that data on the data sheet as "Unfilled Packaging Weight." (Tare).

10.2 Fill each container with the desired amount of product. The level of product in the container should represent the level used for commercial use. To better identify leakage, food coloring or dye may be added.

10.3 Affix the mechanical pump dispensers to each container securely to prevent leakage between the container at the minimum recommended torque load in accordance with Test Method D2063.

NOTE 4—It is extremely important that the test specimens exhibit the minimal recommended torque load so that the worst case for detecting leakage is observed.

10.4 Weigh each package and record the "initial weight" data (W_i) on a data sheet with the information identifying the package.

10.5 Place all of the test specimens, as prepared in 9.1 in their respective environments as indicated by the appropriate label on each unit. Specimens labeled "On-Side" should be placed with the orifice facing down excluding trigger sprayers. Trigger sprayers may have the orifice to the side. If the mechanical pump dispensers use a protective hood or overcap, these test specimens should be stored without these protective devices in place. If the mechanical pump dispensers have a locking feature, these test specimens should be in the locked position during storage and the unlocked position during inspection and testing.

10.6 Once a week for a period of four weeks, (generally on the same day of the week as the test start day), remove all of the test specimens from their respective environments and inspect the test specimens for leakage.

10.7 Allow the temperature of the test specimens to equilibrate (generally 4-6 h) to ambient temperature prior to the actual evaluations.

10.8 Weigh each package and record the "new weight" data (W_n) on the data sheet with the initial weight information.

10.9 Calculate the following:

$$Gross\ Weight\ Loss = W_i - W_n \quad (1)$$

$$\%\ Weight\ Loss = \frac{W_i - W_n}{W_i - Tare} \times 100$$

10.10 At the end of four weeks, evaluate the unprimed mechanical pump dispensers for each of the following parameters:

10.10.1 *Final Weight Lost*—Report the "Final Weight Lost" in accordance with 10.9.

10.10.2 *Strokes to Prime*—Report in accordance with Test Method D3890. Report the findings in 11.1.

10.10.3 *Output per Stroke*—Report in accordance with Test Methods D4336. Report the findings in 11.1.

10.11 Evaluate if the spray or the flow of the product appears to be defective or impeded. Report findings in 11.1.

10.12 Retain all test specimens that are discontinued from testing because of one or more defects. Save these if future examination is required.

11. Report

11.1 Report the following information:

11.1.1 Description of the mechanical pump dispenser (supplier, type, closure size, diptube length),

11.1.2 Identification of the product used.

11.1.3 Statement to the effect that the tests were made in compliance with this test method along with the following information:

11.1.3.1 Name of the technician completing the test.

11.1.3.2 Date of report along with dates for the test period.

11.1.4 Detailed record of test on each mechanical pump dispenser as follows:

11.1.4.1 When a mechanical pump dispenser was discontinued from additional testing and the nature of its discontinuance;

11.1.4.2 Any observations or recommendations that may assist in correcting the various defects reported upon;

11.1.4.3 Data for each package on "Initial Weight", "New Weight" for weeks 1, 2, 3, and 4, and "Final Weight". The

TABLE 1 Results from Validation Test

Gross Weight Loss	Average	Std. Dev.	Max	Min
Upright , Ambient	0.02	0.01	0.03	0.01
Upright, Oven 45°C	0.25	0.02	0.29	0.23
On-Side, Ambient	0.02	0.01	0.03	0.01
On-side, Oven 45°C	0.27	0.02	0.32	0.23
% Weight Loss	Average	Std. Dev.	Max	Min
Upright , Ambient	NA	NA	NA	NA
Upright, Oven 45°C	NA	NA	NA	NA
On-Side, Ambient	NA	NA	NA	NA
On-side, Oven 45°C	NA	NA	NA	NA
Strokes-to-Prime	Average	Std. Dev.	Max	Min
Upright , Ambient	7.13	0.78	8	6
Upright, Oven 45°C	6.00	0.00	6	6
On-Side, Ambient	7.50	0.50	8	7
On-side, Oven 45°C	6.13	0.33	7	6
Output-per-Stroke	Average	Std. Dev.	Max	Min
Upright , Ambient	0.169	0.002	0.172	0.166
Upright, Oven 45°C	0.167	0.002	0.170	0.165
On-Side, Ambient	0.167	0.002	0.169	0.164
On-side, Oven 45°C	0.167	0.002	0.171	0.164

mean value and standard deviation should be calculated and reported for each variable, (for example, “Upright, Ambient”, etc.), along with the maximum and minimum values.

11.1.4.4 Calculations of “Gross Weight Loss”. The mean value and standard deviation should be calculated and reported for each variable, (for example, “Upright, Ambient,” etc.), along with the maximum and minimum values.

11.1.4.5 If this information is desired, calculations of % weight loss. The mean value and standard deviation should be calculated and reported for each variable, (for example, “Upright, Ambient”, etc.), along with the maximum and minimum values.

11.1.4.6 Data for each package on strokes to prime and output per stroke. The mean value and standard deviation should be calculated and reported for each variable, (for example, “Upright, Ambient”, etc.), along with the maximum and minimum values.

11.2 Report the overall results of the testing and draw a conclusion as to the acceptability of the mechanical pump dispenser. If the mechanical pump dispenser is acceptable, but with certain limitations, note accordingly.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is highly dependent on the particular pump style and contents tested. One laboratory has investigated one particular pump style and a hairspray product with 12 replicate tests for gross weight loss only, yielding no visual product leakage along with the results provided in Table 1.

12.1.1 Other pumps and contents will have other averages and will have more or less variability between replicate tests. Users of this test method are suggested to reference historical files of previous tests of similar pumps and contents for an estimate of within laboratory repeatability. Committee D10 believes that because of this strong product and pump style dependency, further investigation of repeatability and reproducibility is not practicable. For the precision and bias for strokes-to-prime and output-per-stroke data, the user should refer to test methods D3890 and D4336.

12.2 *Bias*—This test method has no bias because an accepted reference or referee value is not available.

13. Keywords

13.1 mechanical pump dispenser; package; storage stability