Designation: D3094 – 00 (Reapproved 2010)

Standard Test Method for Seepage Rate of Aerosol Products[1]

This standard is issued under the fixed designation D3094; 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 approximate mass loss due to valve seepage rate[2] of aerosol products by the collection and measurement of gases seeping through the valve and into a special eudiometer tube, over a relatively short time period.

1.2 It can be shown that the average refrigeration-filled aerosol product seeps to the extent of approximately 3.0 mL when the corresponding mass loss is 0.10 oz (2.9 cm^3)/year. This figure is partially based on air content and is subject to variations according to filling conditions. This test method is not considered dependable when applied to pressure-filled, unpurged aerosol products.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *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. Significance and Use

2.1 This test method affords a more rapid answer to the ever-present problem of mass loss during storage. It is of particular value in determining the effectiveness of valve stake and clinch seal elastomers in contact with new formulations. This test method may also be used to evaluate new valves with standard mixtures.

3. Apparatus

3.1 *Bath,* constant-temperature, equipped with a thermoregulator sufficient to maintain water at 80 ± 2°F (26 ± 1°C). The tank should be of sufficient proportions to accommodate the necessary number of test specimens in an upright position, so that each specimen is surrounded by approximately 1 in. (25 mm) of water.

3.2 *Eudiometer Tubes* (Fig. 1 and Fig. 2), custom-ordered or hand-made, with an internal volume of 5.0 mL net (allowing for any part of the valve that might protrude into the tube). It is convenient to calibrate in 1, 2, and 3-mL divisions.

NOTE 1—For tests involving many dispensers, small test tubes and vials have been successfully substituted for the tubes in Fig. 1 and Fig. 2.

4. Test Specimens

4.1 Test specimens shall be prepared in accordance with production methods wherever possible, making certain that the clinch diameter and the depth of clinch below the curl of the mounting cup are in agreement with the specifications. New dispensers shall be pretested for leakage by heating the contents to 130°F (54°C).

5. Procedure

5.1 Fill the bath with water that has been allowed to deaerate for 24 h at room temperature. Bring the bath to 80°F (26°C) and immerse the dispensers.

5.2 Scrub the bath walls, bottom, and dispenser surfaces to remove adhering air. Give the dispensers a hard knock to release any air bubbles clinging to the valve parts.

5.3 Submerge the eudiometer tubes and fill them. Remove the air bubbles. Invert the tubes over the dispenser valves and allow them to remain for 48 h.

5.4 Give each dispenser a hard knock to free the clinging gas into the inverted eudiometer tube. Determine and record the amount of gas in each tube.

5.5 The mass loss due to seepage through the valve and O-ring seal represents only a part of the total mass loss. Leakage will also occur at the seams and seam junctures.

5.6 There is usually a 1 to 2-week adjustment period with new dispensers, during which some perturbations in seepage rate will occur. After this, a reasonably steady day-to-day rate is assumed.

NOTE 2—Many dispensers are found to rust slightly when stored under water for 2 days. This condition may be remedied by employing a bath solution containing 0.5 % sodium nitrate ($NaNO_3$) and 0.5 % triethylene

[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. This test method was originally developed by the Chemical Specialties Manufacturers Assn.

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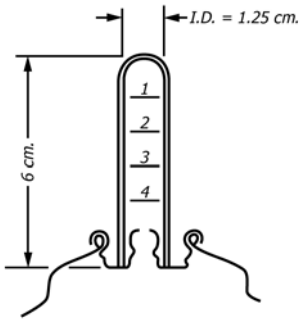
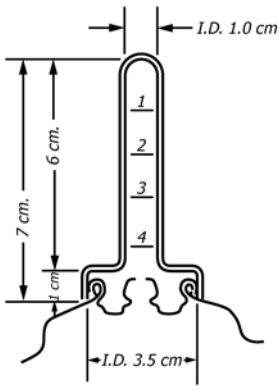
[2] Data on the theoretical development of seepage concepts has been filed at ASTM Headquarters as RR:D10-1000. Contact ASTM Customer Service at service@astm.org.



NOTE 1—The dimensions are approximate, and subject to the geometry of the valve.

FIG. 1 Suggested Tube for Evaluation of Valve and Staked Seals



NOTE 1—The dimensions are approximate, and subject to the geometry of the valve.

FIG. 2 Suggested Tube for Evaluation of Valve, and Staked and Clinched Seals

glycol in water. In a more concentrated solution, triethylene glycol exerts a softening effect upon enamel dispenser finishes.

6. Calculation

6.1 Correct the volume of gas collected in the eudiometer tube to allow for water solubility. Since the degree of solubility differs with the composition of gas, use the following equations in accordance with the chemical content of freshly diffused gas:

For all mixtures of P-11 and P-12:

$$V_c = V_o = 0.29 + (0.66\, N_{P=11}) \quad (1)$$

For difluorodichloromethane only:

$$V_c = V_o + 0.29 \quad (2)$$

For trichlorofluoromethane only:

$$V_c = V_o + 0.95 \quad (3)$$

where:

V_c = corrected column of gases in eudiometer tube,
V_o = observed volume of gases in eudiometer tube, and
$N_{P=11}$ = mole fraction or volume %/100 of trichloro- fluoromethane in the gas as it is diffused into the tube (before selective solubility changes the gas composition).

NOTE 3—Eq 2 and Eq 3 are special cases of Eq 1. Eq 1 is simplified and accurate to 0.1 mL only.

6.2 Corrections for aerosols containing several standard propellants are presented in Fig. 3. All data is based on the use of standard 5.0-mL eudiometer tubes. For example, an air-free aerosol containing 50 % each of P-11 and P-12 as propellants will diffuse an observed 2.73 mL of gas under test conditions when the seepage is 0.10 oz (2.9 cm^3)/year through the valve. The gas will be predominantly difluorodichloromethane.

7. Precision and Bias

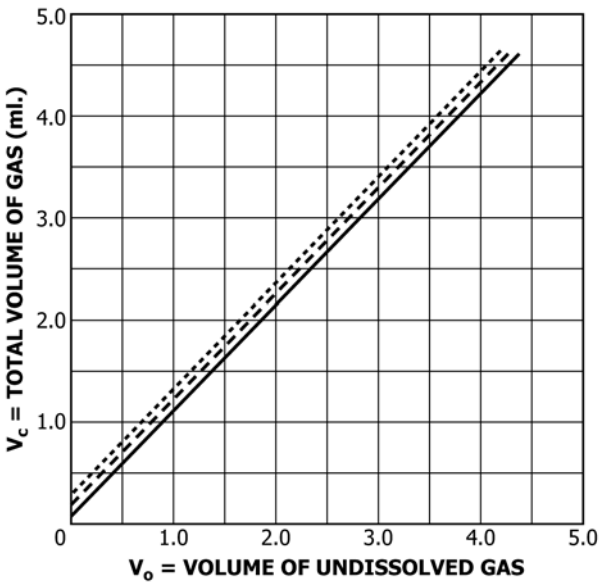
7.1 *Precision*—The precision of D3094 is highly dependent on the contents and type of aerosol packaging being tested. One laboratory conducted a seepage test on a water-based hair mousse. The results are shown in Table 1. The results of this test are dependent on variations in filling the contents (both propellant and product concentrate), the solubility of the propellant used, the head space in the particular can as well as the level of the product in the can at the time of testing.

TABLE 1 Results of Seepage Test[A]

Bubble Size	Actual Propellent Loss	No. of Units	Estimated Annual Weight Loss
4 mL	7.12 mL	1	2.53 g
5 mL	8.12 mL	4	2.88 g
6 mL	9.12 mL	5	3.24 g
7 mL	10.12 mL	2	3.59 g

[A] Actual propellent loss is Bubble Size + Soluble Gases.
Soluble Gases = 0.13 mL/mL of Water × 24 mL tube = 3.12 mL soluble gases.

7.2 *Bias*—Test Method D3094 has no bias because an accepted reference or referee value is not available.



Aerosol Liquid Phase Composition:
..... 50 % P-11 + 50 % P-12
– – – – – 67 % P-12 + 33 % P-11
——— 100 % P-12

FIG. 3 Fate of Gases Seeping Through Aerosols and into a 5.0-mL Eudiometer Tube Filled with Water