



Designation: **D3094 – 00 (Reapproved 2010) D3094 – 18**

## Standard Test Method Practice for Seepage Rate of Aerosol Products<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This ~~test method practice~~ covers the determination of approximate mass loss due to valve seepage rate<sup>2</sup> 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<sup>3</sup>)/year. This figure is partially based on air content and is subject to variations according to filling conditions. This ~~test method practice~~ 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~~ safety, health, and ~~health~~ environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Significance and Use

2.1 This ~~test method practice~~ 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 practice~~ may also be used to evaluate new valves with standard mixtures.

### 3. Apparatus

3.1 *Bath*, constant-temperature, equipped with a thermo-regulator sufficient to maintain water at  $80 \pm 2^\circ\text{F}$  ( $26 \pm 1^\circ\text{C}$ ). 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  $130^\circ\text{F}$  ( $54^\circ\text{C}$ ).

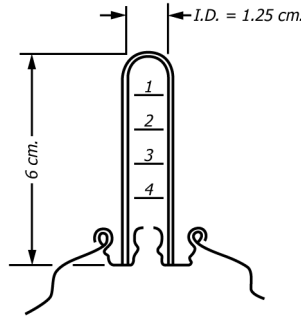
### 5. Procedure

5.1 Fill the bath with water that has been allowed to deaerate for 24 h at room temperature. Bring the bath to  $80^\circ\text{F}$  ( $26^\circ\text{C}$ ) and immerse the dispensers.

<sup>1</sup> This ~~test method practice~~ is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.33 on Mechanical Dispensers/Aerosol Products. This ~~test method practice~~ was originally developed by the Chemical Specialties Manufacturers Assn.

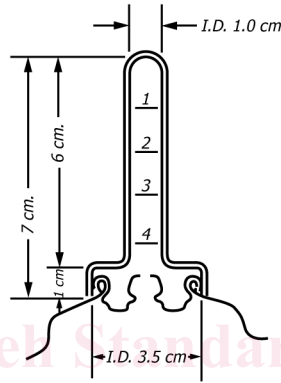
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<sup>2</sup> Data on the theoretical development of seepage concepts has been filed at ASTM Headquarters as RR:D10-1000. Contact ASTM Customer Service at service@astm.org.



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

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 ( $\text{NaNO}_3$ ) and 0.5 % triethylene 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,