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Standard Test Method for Foam In Aqueous Media (Bottle Test)¹

This standard is issued under the fixed designation D3601; 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 measurement of the increase in volume of a low-viscosity aqueous liquid (less than 3 cSt at 40° C) due to its tendency to foam under low shear conditions.

Note 1—Foam under high shear is covered by Test Method D3519 which uses a commercial blender.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

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. For specific safety information, see 7.13.

2. Referenced Documents

2.1 ASTM Standards:²

D1126 Test Method for Hardness in Water D3519 Test Method for Foam in Aqueous Media (Blender Test)

3. Summary of Test Method

3.1 The increase in volume is determined by the increase in total height of the test fluid, including foam, after vigorous shaking of the emulsion at $25 \pm 1^{\circ}$ C (77 $\pm 1.8^{\circ}$ F).

Note 2—Water that is normally used to reduce the concentrate to a working consistency can be used. However, if this type of water is not desirable or available, two other types of water may be used to prepare the test liquid: (I) distilled water or (2) distilled water with a subsequent seeding of the test liquid with synthetic hard water.

4. Significance and Use

4.1 The results obtained by this test method are useful as guides in determining the tendency of a water-based metal-

working coolant to produce foam under low shear conditions. No correlation with changes in heat transfer, pumpability, or other factors affected by foam is intended. The foam generated by any given industrial process depends on the method by which the foam is generated and may not be directly proportional to that produced by this controlled laboratory test method. Further, the foam generated at the specified test temperature will not necessarily predict the foaming tendency of the liquid (that is, metalworking coolant) at some other use temperature.

5. Apparatus

5.1 *Water Bath*, constant-temperature, suitable to hold several bottled emulsions at $25 \pm 1^{\circ}C (77 \pm 1.8^{\circ}F)$ for 1 to 2 h.

NOTE 3—A common household dishpan is satisfactory when the test temperature is close to room temperature.

5.2 Stop Watch or Timer, capable of measuring 5 min \pm 0.2 s.

5.3 *Bottles*, clean or new, clear glass, 16-oz (narrow mouth), with screw neck.³ The 16-oz bottle is $6^{41}/_{64}$ in. (169 mm) tall and has a maximum diameter of $2^{31}/_{32}$ in. (75 mm). The outside neck is $9'_{32}$ in. (7.1 mm) and the shoulder radius is 1 in. (25.4 mm).

5.4 Syringe or Transfer Pipet.⁴

5.5 Rule, millimetre, at least 150 mm in length.

6. Materials

6.1 Distilled Water.

6.2 *Hard Water*, 20 000 ppm prepared by dissolving 29.4 g of reagent grade (ACS standard) $CaCl_2 \cdot 2H_2O$ in 1 litre of freshly boiled distilled water (used only where distilled water is used as in Note 5).

¹This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.L0.01Metal Removal Fluids and Lubricants.

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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.

³ 16-oz Round Packer, Brockway Glass Co., Mold No. 1094 or 16-oz Boston Round, as shown in Fig. 1 is suitable for this purpose. Bottle takes Cap No. 28. Standard Glass Container Series No. 80 specifies approximate weight of glass, etc., on drawing No. C-8010 obtainable from Glass Container Manufacturers' Institute Inc., 330 Madison Ave., New York, NY 10017.

⁴ Disposable pipets, Thomas Scientific Co. Saedsboro, NJ 08085-0099, No. 8937-R62, Corning No. 7077 or Fisher No. 13-671-108E with attached 25 to 50-cm ³ (1 to 2-oz) capacity rubber bulb have been found suitable for this purpose as have Becton-Dickson disposable syringes No. 21G, without needle.



FIG. 1 Boston Round Bottle.

7. Procedure

7.1 Fix a label or other means of marking total heights to the outside of the bottle.

Note 4—Waterproof medical adhesive tape makes satisfactory waterproof labels, which readily take pencil marks. $^{\rm 5}$

7.2 Using the manufacturer's recommended procedure, prepare 200 ml of emulsion at the recommended concentration and pour the emulsion into the 16-oz round bottle (or equivalent 500-cm³ bottle).

7.3 When natural water is used, record water hardness (using Test Method D1126), source, and date obtained.

Note 5—In the absence of manufacturers' recommendations, place 190 ml of distilled water in the 16-oz round bottle. Using a 10-ml syringe or pipet, accurately measure 10 ml of the coolant concentrate into this water. Immediately cap and shake to thoroughly mix this 5% emulsion or solution.

7.4 Store this test liquid in the constant-temperature bath for a minimum of 1 h and a maximum of 2 h at $25 \pm 1^{\circ}C$ (77 $\pm 1.8^{\circ}F$).

Note 6—Storage in water to level above air-liquid interface of emulsion will suffice to stabilize emulsion (or solution) temperature.

7.5 Using any suitable thermometer, measure and record the temperature of this liquid and adjust it to $25 \pm 1^{\circ}C$ (77 $\pm 1.8^{\circ}F$) if necessary. Remove the thermometer and recap the bottle.

7.6 Remove the test sample from the constant-temperature bath. Mark and record the height to the nearest 1 mm of the liquid/air interface, disregarding any foam from previous operations. (This is initial height, I.)

7.7 Place a second mark 10 mm higher than the first mark.

7.8 Vigorously shake the test sample bottle using a minimum of an 8-in. stroke and 40 shakes in less than 10 s.

7.9 Immediately mark and read to the nearest 1 mm the total height (including foam). Call this maximum total height at zero time, M. Start the timer.

Note 7—If the foam is not level, make the best average measurement possible.

7.10 Allow the bottle to stand undisturbed and record the time (to the nearest $\frac{1}{2}$ min within this rest period of 5 min) for

the foam to subside to a net foam height of 10 mm (as indicated by the mark made in accordance with 7.7).

7.11 If the foam height at this time exceeds 10 mm, record the total height to the nearest 1 mm as residual total height after 5 min, R.

7.12 Measure and record the temperature of the test liquid to the nearest $1^{\circ}C$ (1.8°F).

Note 8—The following sections should be performed only when distilled water has been used in the above procedure in accordance with Note 5.

7.13 Add 1.00 ml of 20 000 ppm hard water stock solution to the emulsion. Shake gently to assure uniform mixing of the system. Allow the emulsion to sit quiet for 5 min; then proceed as in 7.14. (Warning—Differences in hydrolyzation time can yield differences in foaming properties.)

7.14 Repeat 7.8-7.12.

8. Calculation and Report

8.1 Calculate maximum foam height, F_m , as follows:

$$F_m = M - I \tag{1}$$

where:

I = initial height (7.6), and

M = maximum total height at zero time (7.9).

8.2 *Time to Defoam to 10 mm (7.10)*— Record the time to defoam to 10 mm of net foam to the nearest 0.5 min if foam subsides to this point within the 5-min test time. Otherwise, record "5 + ."

8.3 Calculate residual foam after 5 min, F_r , as follows:

$$F_r = R - I \tag{2}$$

where:

R = residual total height to nearest 1 mm after 5 min (7.11).

NOTE 9-A suitable report form is described in Fig. 2.

9. Precision and Bias⁶

9.1 The precision of the test method as determined by the statistical examination of interlaboratory test results is as follows:

9.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$Maximum foam - 7.0 mm \tag{3}$$

⁵ One suitable transparent tape is sold under the brand "Scalefix Scales" by Bel-Art Products Inc., Pequannock, NJ, Catalog No. H-2075 (1974).

⁶ The results of the cooperative round-robin test program from which these values have been derived are filed at ASTM International Headquarters. Request RR:D02-1024.