Designation: D3601 – 88 (Reapproved 2007)

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 ± 1°C (77 ± 1.8°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: (*1*) distilled water or (*2*) distilled water with a subsequent seeding of the test liquid with synthetic hard water.

FIG. 1 Boston Round Bottle.

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 metalworking 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 ± 1°C (77 ± 1.8°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 ± 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

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

1. Preparation of Emulsion

1.1 (7.2) Sample description _____
1.2 (7.2) Concentration, % _____
1.3 (7.3) Source of water used _____
1.4 (7.3) Water hardness, ppm _____
1.5 (7.2) Method of preparing emulsion _____

2. Test Data

2.1 (7.5) Temperature at start of test _____ °C
2.2 (7.6) Initial height (*I*) _____ mm
2.3 (7.9) Maximum total height at zero time (*M*) _____ mm
2.4 (7.11) Residual total height after 5 min (*R*) _____ mm
2.5 (7.10) Time to defoam to 10 mm (to nearest ½ min) _____ min
2.6 (7.12) Temperature at end of test _____ °C
2.7 (8.1) Maximum foam, $F_m = M - I$ _____ mm
2.8 (8.2) Residual foam, $F_r = R - I$ _____ mm

Caution—The round robin on this test used distilled water and a controlled synthetic hard water to make data comparative to the products under test at different places and at different times. Care must be exercised when natural waters are used that comparative samples are used in exactly the same water, taken at the same time from the same source. (For instance, well waters can change in hardness rapidly depending on the change in demand within the hour.)

NOTE—Numbers in parentheses indicate the section within the body of the method where the observations to be recorded are made.

FIG. 2 Suggested Test Form for Recording Data.

F_m, Millimetres Maximum Foam = $M - I$

		Sample Number											
		1	2	3	4	5	6	7	8	9	10	11	12
6	x_1	30	35	20	10	65[A]	65[A]	65[A]	5	60	20	15	15
	x_2	30	40	20	10	65[A]	65[A]	65[A]	10	65	20	10	10
7	x_1	39	49	20	20	101	101	101	21	101	11	10	10
	x_2	48	48	19	19	100	100	101	12	101	14	20	10
		F_r, Millimetres Residual Foam = $R = I$											
6	x_1	0	0	0	0	0	0	45	0	55	5	0	0
	x_2	0	0	0	0	0	0	45	0	66	5	5	0
7	x_1	0	0	0	0	0	0	101	0	101	7	4	10
	x_2	0	0	0	0	0	0	101	0	101	8	3	4
		T, Time to Defoam to 10-mm Net Foam Level, min											
6	x_1	0.5	0.5	0.25[B]	0	2.0	2.5	5+	0	5+	1.0	0	0
	x_2	0.25[B]	0.5	0.25[B]	0	1.5	3.0	5+	0	5+	1.25	0	0
7	x_1	0.25[B]	0.25[B]	0.25[B]	0.25[B]	1.5	2.0	5+	0.25[B]	5+	0.25[B]	0.25[B]	1.0
	x_2	0.25[B]	0.25[B]	0.25[B]	0.25[B]	1.5	2.0	5+	0.25[B]	5+	0.25[B]	0.25[B]	0.25[B]

[A] Maximum foam possible in bottle.
[B] Request was for nearest 0.5 min. Where <0.5 min is shown, it was recorded as 0.25 min. x_1 and x_2 are duplicate test results.

Sample Descriptions

Sample 1 L-1-3A in distilled water – 10 % butyl Cellosolve® (trademark of Union Carbide)
3 L-1-3B in distilled water – 10 % butyl Carbitol® (trademark of Union Carbide)
5 L-1-3C in distilled water – Sample 3 plus nonionic ether
7 L-1-3D in distilled water – 5 % commercial long oil soluble coolant
9 L-1-3E in distilled water – 3 % commercial synthetic coolant
11 L-1-3F in distilled water – 3 % commercial synthetic plus defoamer
2, 4, 6, 8, 10, 12 – Same in hard water, respectively

FIG. 3 Sample Data (Bottle Method).

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).

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.[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-cm^3 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

[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.

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

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 ± 1°C (77 ± 1.8°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 ± 1°C (77 ± 1.8°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 ½ 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°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:

$$F_m = M - I \quad (1)$$

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:

$$F_r = R - I \quad (2)$$

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 Bias [6]

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:

$$Maximum\ foam - 7.0\ mm \quad (3)$$

9.1.2 *Reproducibility*—The difference between two, single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty:

$$\text{Maximum foam—72 \% relative} \quad (4)$$

9.2 Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, no statement on bias is being made.

NOTE 10—Residual foam and defoam time values are not suitable for statistical evaluation due to the large number of "zeros," "<0.5 min" and "5^+ min" included in the data.

NOTE 11—*Referee Work*—No more than three replicate tests will normally be required.

10. Keywords

10.1 aqueous media; bottle test; foam; low shear; water based metalworking coolant

[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:D-2-1024.