



Designation: D4009 – 92 (Reapproved 2023)

Standard Guide for Foam Stability of Hand Dishwashing Detergents¹

This standard is issued under the fixed designation D4009; 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 guide provides guidelines for several techniques of measuring the foam stability of light-duty hand dishwashing detergent products in the presence of artificially applied test-food soils. It is intended as a laboratory screening test to aid in the formulation of products, for quality control and as a basis between the formulator and supplier in standardizing foam stability of the detergents.

1.2 *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. 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *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. Summary of Guide

2.1 Soiled dinner plates are washed by hand in solutions of hand dishwashing detergents under standardized conditions until an end point of near-disappearance of the foam is reached, after which the number of plates washed is compared to the number of plates washed using a standard product.

3. Significance and Use

3.1 The guide, as now constituted, is not suitable for ranking of hand dishwashing products, since no basis is available at this time for correlation of the foam stability of these products

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using any particular food soil or combination of soils with consumers' ranking of performance.

3.2 The relative foam stability ranking of hand dishwashing detergent products will vary greatly depending on the type of food soils used in the test. Therefore, selection of the standard food soil to be used in a test shall be made by agreement between the interested parties on the basis of experience.

3.3 This laboratory screening guide includes flexibility in several areas so as to allow its use by the maximum number of laboratories, without purchase of significant additional equipment. It should be recognized, therefore, that differences in specific equipment may result in a reduced level of interlaboratory and inter-operator precision, and such results must be evaluated with caution.

4. Recommended Conditions

4.1 *Water Hardness*—If only one test is to be made, hard water (150 ppm, about 9 grains per gallon (gpg)) is suggested. To produce a more complete picture of product foam stability, test at two or three additional hardness levels: soft water (35 ppm, 2 gpg); moderately hard water (100 ppm, 6 gpg); or very hard water (260 ppm, 15 gpg).

4.1.1 *Calcium/Magnesium Ratio* (as CaCO_3)—It is suggested that this ratio be adjusted for different water hardness as follows:

Water Hardness Range, ppm (gpg)	Calcium/Magnesium Ratio
0 to 60 (0 to 3.5)	4:1
61 to 120 (3.6 to 7.0)	3:1
121 and over (7.1 and over)	2:1

4.2 *Water Temperature*—The water temperature at the start of the test should be adjusted to 47 °C (117 °F).

5. Materials

5.1 *Plain White Glazed Dinner Plates in Sound Condition*—200 mm to 230 mm (8 in. to 9 in.) in diameter, with 160 mm to 165 mm (6 ¼ in. to 6 ½ in.) indented bottom.

5.2 *Dishpan (conventional)*—Bottom diameter = 280 mm (11 in.), top diameter = 370 mm (14 ½ in.), depth = 140 mm (5 ½ in.). Sheet metal or plastic are suitable materials.

5.3 *Dishcloth (any conventional brand), Dish Mop, or Disposable Dishcloths*—Enough clean dish cloths (or dish mops) should be available to ensure the use of a fresh cloth (or

dish mop) for each test run in one day. A new disposable cloth should be used for each wash.

5.4 Any suitable reservoir of 4 L capacity, that can be readily loaded with test water and that can deliver its contents through a 9.5 mm ($\frac{3}{8}$ in.) (inside diameter) drainage tip. Preferably, this drainage tube is an open-shut style to permit full flow immediately upon opening.

6. Standard Soils

6.1 Four soils, representative of those commonly used for hand dishwashing tests, are described below. Other soil compositions may be used.

6.1.1 Soil A:

	wt %
Lard (not hydrogenated)	18.3
Wesson oil	9.2
Corn oil	9.2
Oleic acid (USP)	4.2
Salt	0.4
Gelatin	0.4
Flour	41.6
Water	16.7
	100.0

6.1.1.1 Prepare this mixture on the day prior to use. Discard any soil more than one day old. Prepare as follows: Weigh the lard, vegetable oil, corn oil, and oleic acid into a beaker and warm to 38 °C (100 °F). Add the salt, gelatin, and flour while mixing with a spatula. Store at 3 °C to 6 °C (38 °F to 42 °F) overnight. The following day, just prior to use and without heating, blend in the water with a large 200 mm (8 in.) spatula. Apply soil at room temperature. A bright dye may be added to aid visual inspection.

6.1.2 Soil B:

Flour	50 %
Shortening	48 %
Oleic acid (USP)	2 %

6.1.2.1 Warm the shortening with oleic acid to 38 °C (100 °F). Slowly add flour while mixing with a spatula and warming to 49 °C (120 °F). Hold soil temperature at 49 °C \pm 1.5 °C (120 °F \pm 3 °F) while soiling dishes.

NOTE 1—In order to prevent soil from melting off plates, do not exceed the proper wash water temperature of 47 °C (117 °F).

6.1.3 Soil C—Shortening.

6.1.3.1 Warm the shortening to 42 °C \pm 1.5 °C (108 °F \pm 3 °F) and maintain at this temperature during soiling. A small amount of an oil-soluble dye, can be added to the warm shortening before soiling to provide visual evidence of soil residue.

6.1.4 Soil D:

	wt %
Shortening	42.85
Spray-dried egg powder	14.3
Tap water	42.85

6.1.4.1 Prepare as follows: Weigh into the bowl of an electric household mixer, the required amount of shortening. Weigh in egg powder and then blend at low speed with the mixer to form a thick paste. Add tap water, heated to about 40 °C (104 °F), over a period of about 1 min, while mixing, and then blend for approximately 4 min to 5 min until the mixture attains a homogeneous creamy consistency. The quan-

tity prepared should be sufficient for one day's work only, soil being made up fresh daily. Approximately 450 g of shortening and 150 g of egg powder make sufficient soil for about 500 plates.

7. Preparation of Soiled Plates

7.1 Wash the plates thoroughly, by usual hand methods or in an automatic dishwasher, before starting the test and between test soilings/washings to provide clean dry plates.

7.2 Place on a balance, the beaker containing the selected soil, A, B, or C. Set to weigh 6.0 g \pm 0.1 g light. Use a spatula or spoon to remove 6.0 g soil for application to a plate. Alternatively, weigh each plate, tare, and then add soil to 6.0 g \pm 0.1 g.

7.3 Transfer Soil A or B most readily, with a small spatula. Scrape all soil from the spatula on the edge of the plate. Soil C, a liquid at 42 °C (108 °F), can be added from a small spoon or by using an automatic pipet syringe by Manostat set to deliver 6.0 g.

7.4 For Soil D use a press-down cream dispenser with adjustable plunger to deliver 2 g \pm 0.03 g of soil to each plate.

7.5 Use one's finger to spread the soil over the surface of the plate. Disposable gloves are recommended for this procedure. Soiled plates are then stacked in convenient sized piles (20 plates to 25 plates). The top plate of each pile may be inverted to prevent drying out of soil. After soiling the last plate of each stack, the residual soil on the finger is removed by wiping on the sides of the stacked, soiled plates.

7.6 Wash Soils A, B or D the same day as soiling. Test wash Soil C the day after soiling.

7.7 Keep soil well mixed by frequent stirring and keep soil covered. Do not use excess soil or soiled dishes beyond the time schedule, as noted.

8. Preparation of Dishwash Solution

8.1 The standard concentration is 0.10 % product. The standard temperature is 47 °C (117 °F). Other "as is" or solids concentrations can be used in comparison testing.

8.1.1 Prepare 4 L of test water at desired hardness and temperature and place in reservoir.

8.1.2 Draw 100 mL of test water from the reservoir.

8.1.3 Divide this into four equal portions of 25 mL each.

8.1.4 Swirl 4 g of product in a flask containing 25 mL of test water and pour into the wash pan.

8.1.5 Rinse this flask with 25 mL of test water three times, pouring each rinse into the wash pan.

8.1.6 Add the 3.9 L of test water remaining in the reservoir to the wash pan. The reservoir delivery tip is situated 610 mm (24 in.) above the bottom of the dish pan so that it delivers into the center of the pan.

9. Washing Procedure

9.1 Two alternative methods for washing the soiled plates are explained in the following:

9.1.1 *Method A*—Place the dishcloth in the wash water and the first dish is washed beginning at 20 s after the water

addition is complete. Wash one dish at a time, both front and back, using a rotating motion with the dishcloth while keeping the dish half submerged in an angular position with the bottom of the dishpan. Use a soiled dish every 30 s and continue washing to reach an end point where just half the surface of the wash solution shows a thin layer of foam. Dishes washed can be rated at whole or half dishes, depending on the operator's measurement of residual foam.

9.1.2 *Method B*—In an alternative method, slide two soiled plates edgewise into the wash bowl. Hold one of these at 45° to the horizontal, so that about half of its area is above the wash liquor surface, and most of the foam is to the front of the plate. Remove the soil from the plate by four circular strokes of a dishmop or cloth. Pass the mop or cloth through the liquor surface on each stroke. Foam is thus stirred into the wash liquor, and the soil is simultaneously emulsified. Then turn the plate and similarly clean its reverse side, but with three circular strokes. Brush back any foam adhering to the plate into the bowl with the mop or cloth, and then transfer the plate to a sink or container of hot water or detergent. Then take a third soiled plate from the pile, and slide underneath the plate already in the bowl. Wash the second plate as described above, and repeat the cycle. Foam height is gradually reduced, and eventually free liquor surface, that is liquor not covered by foam, becomes visible. Note the number of plates washed, and continue the washing operation. In general, one more plate will reduce the area of foam to about half (or less than half) of the total liquor surface. Note the number of plates washed to this half-foam end point and wash further plates until almost complete destruction of the foam occurs. This normally takes only one more plate. The result of the test is taken as the number of plates washed at the half-foam end point. Experience has shown that this end point is the one least subject to errors in judgement by operators.

10. Cleaning Procedure

10.1 After each test, the dishcloth or dishmop should be boiled in detergent solution, thoroughly rinsed out with hot tap water, and as much liquid as possible removed by squeezing. The plates that have been washed during the test should be thoroughly washed and rinsed, placed in racks and allowed to dry. Alternatively, an automatic dishwasher may be used. The wash bowl must be well cleaned after each test.

11. Reference Blend

11.1 Periodically, and specifically when using any new components in the preparation of a soil or when evaluating a new set of samples, it is prudent to test a known pair of dishwash blends to permit some comparison with previous data. This reference mix should wash the same number of dishes within the normal variation limits.

12. Arrangement of Tests

12.1 Since the absolute level of results, that is, number of plates washed to the foam end point, may be different for different operators and on different days, these possible effects should be allowed for by:

12.1.1 Testing each product the same number of times by each operator,

12.1.2 Testing products on a comparative basis, and

12.1.3 Conducting at least four replicate tests.

12.2 The products should be tested until a suitable and specified statistically determined confidence interval is reached between the appropriate comparisons.

13. Data Evaluation

13.1 The test conditions, such as, water hardness, product concentration, temperature, test method, and method of data analysis, should be specified.

13.2 Calculate the average number of plates washed by each product, including that of a reference product, over the several replicate tests. Express the foam stability of a product as a percentage of the foam stability of a reference product. Differences in foam stability may also be expressed as relative plate count averages, as long as a suitable statistical method can be adopted that will allow one to determine product differences at specified, statistically determined confidence intervals.

14. Precision and Bias

14.1 When experienced operators are employed, the 95 % confidence level of a result, average of four tests, should be approximately $\pm 5\%$.

14.2 Plate count averages and standard deviations for a given product have little, if any, direct value in assessing product differences. Product differences can be evaluated only through sufficient testing to determine the foam stability difference that is necessary to establish to ascertain whether two products are different with a specified degree of confidence.

14.3 While appropriate statistical treatments for analyzing data and evaluating confidence intervals can be found in a number of texts and papers, the following references can be recommended.

14.3.1 Mandel, J., and Lashof, T. W. "The Interlaboratory Evaluation of Testing Methods," *ASTM Bulletin*, No. 239, July 1959.

14.3.2 Snedecor, G. W., *Statistical Methods*, 5th Ed., Iowa State College Press, Ames, Iowa, 1956.