

FED. TEST METHOD STD. NO. 536A/GEN

May 9, 1975

SUPERSEDING

Fed. Test Method Std. No. 536

February 15, 1956

FEDERAL TEST METHOD STANDARD

SOAP AND SOAP-PRODUCTS  
(INCLUDING SYNTHETIC DETERGENTS);  
SAMPLING AND TESTING

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FSC 7930

INFORMATION SHEET

ON

FEDERAL STANDARDS

This Federal Standard is issued in loose leaf form to permit the insertion or removal of new or revised sections and methods.

All users of Federal Standards should keep them up to date by inserting revised or new sections and methods as issued and removing superseded and canceled pages.

New and revised material and cancellations will be issued under Change Notices which will be numbered consecutively and will bear the date of issuance. Change Notices should be retained and filed in front of the Numerical Index of the Standard until such time as they are superseded by a reissuance of the entire Standard.

## FED. TEST METHOD STD. NO. 536A/GEN

## FEDERAL TEST METHOD STANDARD

SOAP AND SOAP-PRODUCTS (INCLUDING SYNTHETIC DETERGENTS):  
SAMPLING AND TESTING

This standard was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

## 1. SCOPE AND NUMBERING SYSTEM

1.1 Scope. This standard covers methods of sampling and testing which shall be used officially in the sampling and routine testing of soaps and soap products including those containing synthetic detergents, purchased under Federal specifications. The Government reserves the right to use any additional information to determine whether the material ordered meets the requirements of the applicable purchase specification.

1.2 Numbering system. The Federal Test Methods herein are identified by numbers consisting of the basic Federal Test Method Standard number, a slant mark (/), and a whole number, e.g., FED. STD. TEST METHOD 536/101. Revision numbers, if any, are placed after a decimal point, e.g., the second revision is FED. STD. TEST METHOD 536/101.2.

1.3 Supersession. Most of the Federal Test Methods listed in the prior issue of this standard have been superseded by ASTM test methods. These superseded Federal Test Methods are cross referenced alphabetically and numerically to applicable sections of the superseding ASTM standards. Active Federal Test Methods are so designed and are printed herein. One Federal Test Method is canceled without supersession. The superseding ASTM test methods are listed separately in numeric sequence with their titles.

## 2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issues in effect on date of invitation for bids or request for proposal, form a part of this standard to the extent specified herein.

Federal Specification:

RR-S-366 - Sieve, Test.

(Activities outside the Federal Government may obtain copies of Federal Specifications and Standards as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(Single copies of this standard and of Federal Specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Services Administration Regional Offices in Boston, New York, Atlanta, Chicago, Kansas City, MO, Ft. Worth, Denver, San Francisco, Los Angeles, Seattle, WA, or from Specification Sales, GSA, Bldg. 197, Washington Navy Yard, Washington, D.C. 20407. Additional copies may be purchased from the Specification Sales Office, Washington, D.C. 20407.

(Federal Government activities may obtain copies of Federal Specifications and standards from established distribution points in their agencies.)

Laws and Regulations:

26 CFR 212 - Formulas for Denatured Alcohol and Rum.

(The Code of Federal Regulations (CFR) and the Federal Register (FR) are for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. When indicated, reprints of certain regulations may be obtained from the Federal agency responsible for issuance thereof.)

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2.2 Other publications. The following documents form a part of this standard to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials (ASTM) Standards:

- ASTM D - Standard Methods for Sampling and Chemical Analysis of Soaps and Soap Products.
- ASTM D - Standard Methods of Chemical Analysis of Soaps Containig Synthetic Detergents.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. GENERAL NOTES

3.1 Neutral ethyl alcohol (95 percent). Where this reagent is specified, it is permissible to use either formula 3A or 3C, denatured ethyl alcohol, 95 percent, neutral to phenolphthalein, described in Subpart D of 26 CFR 212.

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## ALPHABETICAL LIST OF TEST METHODS

Title of Method	Fed. Std. Test Method No.	STATUS: ACTIVE, CANCELED OR SUPERSEDED BY: ASTM Method No.
Acid, Free	302.3 5301.2	D460 #18 D820 #7-8
Acid Number of fatty acids	1001.1	D460 #45
Alcohol-insoluble matter	301.3	D460 #16-17
Alcohol-soluble matter	5701.1	D820 #13-14
Alkali, free	302.3 5301.2	D460 #18 D820 #7-8
Alkali, combined	401.3	D460 #21-22
Alkaline salts	304.3 5901.1	D460 #20 D820 #17
Anhydrous soap	401.3	D460 #21-22
Borax	1201.1	D460 #53-55
Carbonates (gravimetric method)	1401.2	D460 #61
Carbonates (volumetric method)	1402.1	D460 #62-65
Chlorides	501	D460 #50-52
Chlorides in alcohol-soluble matter	6201.1	D820 #39-41
Cleaning efficiency test	6701	ACTIVE
Combined sodium and potassium oxides	1901	D460 #23-26
Detergent, synthetic	6501	D820 #46
Fatty matter (determination of)	6101.1	D820 #35-38
Fatty matter (fatty and rosin acids), preparation of	801.1	D460 #41-42
Free alkali or free acid	302.3 5301.2	D460 #18 D820 #7-8
Glycerol (in the absence of sugars)	1702	D460 #79-81
Glycerol (in the presence of sugars)	1703.1	D460 #82-83
Hydrocarbons, volatile	1801	D460 #88-92
* Inorganic salts, neutral	6601	D820 #47
* Insoluble matter in alcohol	301.3	D460 #16-17
Insoluble matter in water	303.1 5801	D460 #19 D820 #15
Iodine number of fatty acids	1101.2	D460 #47-49
Moisture and volatile matter (oven method)	201.1 5201	D460 #11 D460 #11
Moisture (distillation method)	202.2	D460 #12-15
Neutral inorganic salts	6601	D820 #47

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## ALPHABETICAL LIST OF TEST METHODS (Cont.)

Title of Method	Fed. Std. Test Method No.	STATUS: ACTIVE, CANCELED OR SUPERSEDED BY: ASTM Method No.
Phosphates, total	1501.1	D820 #21-28
Potassium carbonate and free alkali in potash-paste soaps	305.2	D460 #27
Pyrophosphate, tetrasodium	1502.1	D460 #74-76
Qualitative test for synthetic detergent	5103	CANCELED
Rosin	701.3 6301.2	D460 #37-40 D820 #42-45
Sampling, methods of	101.2 5101.1	ACTIVE ACTIVE
Sieve tests	2101	ACTIVE
Silica present as alkaline silicates	1301	D460 #56-57
Soap anhydrous	401.3	D460 #21-22
Soap anhydrous, salt-free	5601.1	D820 #9-12
Sodium Silicate	6001	D820 #18-20
Starch	1704 6401	D460 #84-85 D460 #84-85
Sudsing test	2001.2	ACTIVE
Sugars	1705.1	D460 #86-87
Sugars (Qualitative)	1701	ACTIVE
Sulfates	1601.1	D460 #77-78
Synthetic Detergent	6501	D820 #46
Titer of fatty acids	901	D460 #43-44
Unsaponified plus unsaponifiable matter	601.2	D460 #30-32
Unsaponifiable matter	602.1	D460 #33-35
Unsaponified saponifiable matter	603.1	D460 #36
Volatile Hydrocarbons	1801	D460 #88-92
Volatile matter and moisture	201.1 5201	D460 #11 D460 #11

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## NUMERICAL LIST OF TEST METHODS

Title of Method	Fed. Std. Test Method No.	STATUS: ACTIVE, CANCELED OR SUPERSEDED BY ASTM Method No.
SOAPS AND SOAP PRODUCTS:		
Sampling	101.2	ACTIVE
Moisture and Volatile Matter (oven method)	201.1	D460 #11
Moisture (distillation method)	202.2	D460 #12-15
Alcohol-insoluble matter	301.3	D460 #16-17
Free Alkali or free acid	302.3	D460 #18
Matter insoluble in water	303.1	D460 #19
Alkaline salts	304.3	D460 #20
Free alkali and potassium carbonate in soaps	305.2	D460 #27
Total anhydrous soap and combined alkali	401.3	D460 #21-22
Chlorides	501	D460 #50-52
Unsaponified plus unsaponifiable matter	601.2	D460 #30-32
Unsaponifiable matter	602.1	D460 #33-35
Unsaponified saponifiable matter	603.1	D460 #36
Rosin	701.3	D460 #37-40
Preparation of total fatty matter	801.1	D460 #41-42
Titer test	901	D460 #43-44
Acid Number of fatty acids	1001.1	D460 #45
Iodine number (Wijs method) of fatty acids	1101.2	D460 #47-49
Borax	1201.1	D460 #53-55
Silica present as alkaline silicates	1301	D460 #56-57
Carbonates (Gravimetric Method)	1401.2	D460 #61
Carbonates (Volumetric Method)	1402.1	D460 #62-65
Total Phosphates	1501.1	D820 #21-28
Tetrasodium pyrophosphate	1502.1	D460 #74-76
Sulfates	1601.1	D460 #77-78
Sugars (Qualitative)	1701	ACTIVE
Glycerol in the absence of sugars	1702	D460 #79-81
Glycerol in the presence of sugars	1703.1	D460 #82-83
Starch	1704	D460 #84-85
Sugars	1705.1	D460 #86-87
Volatile Hydrocarbons	1801	D460 #88-92
Combined sodium and potassium oxides	1901	D460 #93-96

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## NUMERICAL LIST OF TEST METHODS (Cont.)

Title of Method	Fed. Std. Test Method No.	STATUS: ACTIVE, CANCELED OR SUPERSEDED BY: ASTM Method No.
SOAPS AND SOAP PRODUCTS (CONT.):		
Sudsing test	2001.2	ACTIVE
Sieve tests	2101	ACTIVE
SOAPS AND SOAP PRODUCTS CONTAINING SYNTHETIC DETERGENTS:		
Sampling	5101.1	ACTIVE
Qualitative test for synthetic detergent	5103	CANCELED
Moisture and Volatile matter (oven method)	5201	D460 #11
Free alkali or free acid	5301.2	D820 #7-8
Anhydrous, salt-free, soda soap	5601.1	D820 #9-12
Alcohol-soluble matter	5701.1	D820 #13-14
Matter insoluble in water	5801	D820 #15
Alkaline salts	5901.1	D820 #17
Silica present as alkaline silicates	6001	D820 #18-20
Fatty matter	6101.1	D820 #35-38
Chlorides in alcohol-soluble matter	6201.1	D820 #39-41
Rosin	6301.2	D820 #42-45
Starch	6401	D460 #84-85
Synthetic detergent	6501	D820 #46
Neutral inorganic salts (calculation)	6601	D820 #47
Cleaning efficiency test	6701	ACTIVE



## ASTM NUMERICAL INDEX

Title of Method	Fed. Std. Test Method No.	SUPERSEDING ASTM STANDARD AND SECTION(S)
Moisture and Volatile matter (Oven Method)	201.1	D460 #11
Moisture and Volatile matter (Oven Method)	5201	D460 #11
Moisture (Distillation Method)	202.2	D460 #12-15
Alcohol insoluble matter	301.3	D460 #16-17
Free acid	302.3	D460 #18
Insoluble matter in water	303.1	D460 #19
Alkaline salts	304.3	D460 #20
Alkali, combined	401.2	D460 #21-22
Combined sodium and potassium oxides	1901	D460 #23-26
Free alkali and potassium carbonates	305.2	D460 #27
Unsaponified plus unsaponifiable matter	601.2	D460 #30-32
Unsaponifiable matter	602.5	D460 #33-35
Unsaponified matter	603.1	D460 #36
Rosin	701.3	D460 #37-40
Preparation of total fatty matter	801.1	D460 #41-42
Titer test	901	D460 #43-44
Acid number of fatty acids	1001.1	D460 #45
Iodine number of fatty acids	1101.2	D460 #47-49
Chlorides	501	D460 #50-52
Borax	1201.1	D460 #53-55
Silica	1301	D460 #56-57
Carbonates (Gravimetric method)	1401.2	D460 #61
Carbonates (Volumetric method)	1402.1	D460 #62-65
Tetrasodium pyrophosphates	1502.1	D460 #74-76
Sulfates	1601.1	D460 #77-78
Glycerol in the absence of sugars	1702	D460 #79-81
Glycerol in the presence of sugars	1703.1	D460 #82-83
Starch	6401	D460 #84-85
Sugars	1705.1	D460 #86-87
Volatile hydrocarbons	1801	D460 #88-92
Free acid	5301.1	D820 #7-8
Anhydrous, salt-free soda soap	5601.1	D820 #9-12
Alcohol soluble matter	5701.1	D820 #13-14

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## ASTM NUMERICAL INDEX (Cont.)

<u>Title of Method</u>	<u>SUPERSEDED Fed. Std. Test Method No.</u>	<u>SUPERSEDING ASTM STANDARD AND AND SECTION(S)</u>
Insoluble matter in water	5801	D820 #15
Alkaline salts	5901.1	D820 #17
Silica as alkaline silicates	6001	D820 #18-20
Phosphates	1501.1	D820 #21-28
Fatty matter	6101.1	D820 #35-38
Chlorides in alcohol-soluble matter	6201.1	D820 #39-41
Rosin	6301.2	D820 #42-45
Synthetic detergent	6501	D820 #46
Neutral inorganic salts	6601	D820 #47
Acid number of fatty acids	1001.1	D460 #45-46

## SAMPLING AND ACCEPTANCE PROCEDURES

Scope: This section describes the sampling procedures for soap and soap products, including synthetic detergents, and the procedures for acceptance or rejection of lots of these products.

## 1. SELLER'S OPTION

1.1 The seller shall have the option of being represented at the time of sampling and when he so requests shall be furnished with a duplicate sample.

## 2. DEFINITIONS OF SAMPLING TERMS

2.1 Lot. A lot is a delivery or a portion of a delivery composed of material of any one type, grade, class, or composition. A lot is either accepted or rejected as a whole on the basis of tests carried out on a portion of the lot.

2.2 Sample. The sample is the combination of the portions of material taken from a lot for the purpose of inspection and/or test.

2.3 Composite sample. The composite sample is a homogeneous mixture prepared from the portions constituting the sample.

2.4 Specimen. The specimen for test is a portion of the sample or composite sample on which a specified test is prepared. In some cases, a single specimen is used for more than one test.

2.5 Test result. The test result is a single numerical value which may be the average or the median\* of all determinations carried out for a particular chemical or physical property on a particular specimen.

\* The median of an odd number of values is the middle-most when the values are written in increasing order of magnitude. Thus the median of 12.1, 12.3, 12.5, 12.6, and 13.7 is 12.5.

2.6 Acceptance of lot. The acceptance of a lot is the approval of a lot as conforming to the contract and/or specification.

2.7 Rejection of lot. The rejection of a lot is the disapproval of a lot as not conforming to the contract and/or specification.

## 3. GENERAL SAMPLING PROCEDURES

3.1 Apparatus.

3.1.1 Universal food cutter with 12-tooth blade, or equivalent.

3.1.2 Trier for paste soap. A half-round metal tube 1/2 to 1/2 inch in diameter. The length may be 2 to 4 feet, depending upon the size of packages to be sampled. One end is tapered to a point, the taper to be not more than 1 inch long. The other end is attached to a D- or T-shaped handle.

3.1.3 Sample tube for liquid product. A glass tube 3/8 to 1/2 inch in inside diameter. The length may be convenient for the size of packages to be sampled. One end is constricted by a short taper (not more than 1 inch long) to about 1/4 inch. The other end is constricted sufficiently so that it can be used as a finger valve.

3.1.4 Airtight and watertight sample containers - two pound capacity.

3.1.5 Airtight and watertight sample containers - sufficient capacity to hold samples taken as specified in 3.3.1, 3.3.2, and 3.3.3.

3.1.6 Spatula - 10- or 12-inch blade.

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3.2 General directions.

3.2.1 Each case or large container shall be carefully identified by the manufacturer's batch (or crutcher) number, until ultimate action is taken as to acceptance or rejection of material offered for delivery. Individual samples shall not be mixed and shall be placed in separate airtight and watertight containers, covered and sealed to prevent any moisture change. Samples shall be labeled completely for identification and sent to the laboratory for preparation and test.

3.2.2 In taking the portions from the case to be sampled, as many portions shall be removed from the layers next to the edges of the package case as from the middle. Portions shall be taken in such a manner that the sampling is thoroughly representative of all positions of the case.

3.3 Size of sample.

3.3.1 Small packages containing cake soap, powder, flakes, paste, or liquid.

3.3.1.1 Lots of less than 1,000 cases. Take a minimum of 10 cases at random throughout the lot and take 1 bar or 1 package from each case. In the case of lots containing less than 10 cases, sample all cases.

3.3.1.2 Lots of 1,000 cases or more. Take 1 percent of the total number of cases at random through the lot and take 1 bar or 1 package from each case. In the case of very large lots where the total sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 pounds.

3.3.2 Barrels, tierces, or similar large packages containing powder or flakes.

3.3.2.1 Lots of less than 1,000 packages. Take a minimum of 10 packages at random throughout the lot and take about 1 pound from each package. In the case of lots containing less than 10 packages, sample all packages.

3.3.2.2 Lots of 1,000 packages or more. Take 1 percent of the total number of packages at random throughout the lot and take 1 pound from each package. In the case of very large lots where the total sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced so that amount drawn shall not exceed 20 pounds.

3.3.3 Large packages containing paste or liquid soap. The number of packages to be sampled shall be as directed in 3.3.2.1 and 3.3.2.2.

3.3.3.1 Paste soap products in large packages are conveniently sampled with the metal trier. Insert the trier diagonally through the package, turn it two or three times and withdraw with the material. Take enough such portions from each package to make about 1 pound, and accumulate in a clean, dry container.

3.3.3.2 Liquid soap products in large packages are conveniently sampled with the glass tube. The product should be at a temperature of 20° to 30°C. Mix the package thoroughly and insert the tube slowly, with the upper end open so that the material gradually fills the tube as it is slowly lowered. When the tube is full, close the upper end with a finger and withdraw the tube. Take enough such portions from each package to make about 1 pound (or 1 pint liquid measure) and accumulate in a clean and dry container.

Note. If the consistency is such that neither the metal trier nor the glass tube can be used, take grab portions with a dipper or some similar and convenient tool.

#### 4. PREPARATION OF COMPOSITE SAMPLES

##### 4.1 Types.

4.1.1 Cake soap. In the case of material that can be easily disintegrated and mixed, run the cakes, constituting the sample, through a suitable chopper. With material that cannot be handled as above, quarter the cakes, constituting the sample, by cutting at right angles in the center, and shave equally from all freshly cut surfaces. Mix all portions promptly and keep in an airtight, watertight container.

4.1.2 Powdered and chip soaps. Rapidly disintegrate and mix the portions; if desired, quarter down to about 1 pound and weigh out all portions for analysis at once. The unused portion of the sample shall be preserved in an airtight, watertight container in a cool place.

4.1.3 Liquid soap. No preparation of the sample, other than thorough mixing, is necessary unless it is received during very cold weather, when it should be allowed to stand at least 1 hour after it has warmed up to room temperature (20° to 30° C) before it is tested, particularly for its lathering qualities.

4.1.4 Paste soap products. Mix thoroughly by kneading with a spatula on a clean, dry, nonabsorbent, impervious surface, and quarter down to about 1 pound. Weigh out all portions for analysis promptly and preserve the remainder in an airtight, watertight container by storing in a cool place.

#### 5. PRELIMINARY TESTS

5.1 Note the color, odor, and condition of the sample. If the sample is received in original cans or cartons, note the weight of the contents of each can or carton.

#### 6. ACCEPTANCE OR REJECTION PROCEDURE

6.1 Acceptance of lot. The lot shall be considered to meet all the requirements of the contract and/or the specification, if the test result for each property for which requirements are specified meets the corresponding requirements.

6.2 Rejection of lot. The lot shall be considered as having failed to meet the requirements of the contract and/or specification, if the test result for one or more of the properties, for which requirements are specified fails to meet the corresponding requirements.

SUGARS (QUALITATIVE)

Definition: This test detects the presence of sugars in the sample.

Scope: Applicable to soaps and soap products.

1. REAGENTS

1.1 Fehling solution. Mix equal volumes of (a) and (b) solutions immediately before use.

- (a) Copper sulfate solution. Dissolve 35 grams of copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in water and dilute to 500 milliliters.
- (b) Alkaline tartrate solution. Dissolve 173 grams of Rochelle salt ( $\text{NaKC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ ) and 50 grams of sodium hydroxide in water and dilute to 500 milliliters.

2. PROCEDURE

2.1 Add a decided excess of hydrochloric acid to a solution of the soap, heat on a steam bath for 15 minutes, cool, filter from fatty acids, and test a portion of the filtrate (which has been neutralized with sodium hydroxide solution) by boiling for 2 minutes with an equal volume of boiling Fehling solution. The formation of red cuprous oxide indicates the presence of sugar.

SUDSING TEST

Definition: This method determines the lathering power of the sample.

Scope: Applicable to soaps and soap products including those containing synthetic detergents.

1. APPARATUS

1.1 Graduated cylinder - 250-milliliter, glass-stoppered.

2. PROCEUDRE

2.1 Dissolve 1.0 + 0.01 gram of the sample in 500 milliliters of distilled water at 20 + 2°C. Place 50 milliliters of the solution in the graduated cylinder. Stopper the cylinder and shake by inversion, rotating the cylinder about its midpoint without translational motion for 1 minute at such a rate that 30 inversions are completed. Place the cylinder in an upright position on a table, remove the stopper, wait 5 seconds, and read the net volume of foam (total volume minus the volume of liquid).

## SIEVE TESTS

Definition: This method determines the relative degree of fineness of the sample.  
Scope: Applicable to soaps and soap products excluding those containing synthetic detergents.

## 1. SIEVES

1.1 Fed. Spec. RR-S-366. The sieves used in these tests shall conform to Federal Specification RR-S-366.

## 2. POWDERED PRODUCTS NOT CONTAINING ABRASIVES INSOLUBLE IN WATER

2.1 Transfer 100 + 1 grams of the well-mixed sample, without previous drying, to the first sieve (coarsest), previously dried, and sift, tapping the sieve frame from time to time and catching all of the material passing through it on the second dry sieve (next coarsest). The sifting on the first sieve is continued until the weight of the residue retained on it is not reduced by more than 0.1 gram on further sifting for 1 minute, tapping the sieve frame as before. Calculate the final weight of residue to percentage retained on the coarsest sieve. Sift the material on the second sieve, tapping the sieve frame from time to time and catching all of the material passing through it on the dry third (finest) sieve. The sifting on the second sieve is continued until the weight of the residue retained on it is not reduced by more than 0.1 gram on further sifting for 1 minute, tapping the frame as before. Add the final weight of the residue retained on the first sieve to the final weight of the residue retained on the second sieve, and calculate the sum to percentage of residue retained on the second sieve. Sift the material on the third sieve, tapping the sieve frame from time to time, until the weight of the residue retained on it is not reduced by more than 0.1 gram on further sifting for 1 minute, tapping the frame as before. Add the final weight of the residue retained on the second sieve (sum of weights retained on the first and second sieves) to the final weight of the residue retained on the third sieve and calculate the sum to percentage of residue retained on the third sieve. The series of sieves may be nested together so that the material passing through a sieve is transferred directly to the next sieve in the series.

## 3. CAKE SOAPS, PASTE SOAPS, AND POWDERS CONTAINING ABRASIVES INSOLUBLE IN WATER

3.1 For convenience in weighing, a 3-inch sieve is recommended.

3.2 Dry for 1 hour in an oven at 105° to 110°C. the standard sieves specified, cool, and weigh to the nearest 0.05 gram. Weigh an amount of sample containing 5 to 10 grams of insoluble siliceous material or other abrasive, transfer to a beaker, add about 200 milliliters of water and digest on a steam bath about 1 hour to dissolve the soluble matter. Pour the solution through the coarsest sieve, wash the insoluble matter from the beaker onto the sieve with hot water and wash with water, catching all of the liquid and solid matter passing through the sieve in clean beakers or dishes. The washing with water shall be continued until 200 milliliters of the liquid passing through the sieve into a clean 400-milliliter beaker fails to show any particles collected about the middle of the bottom of the beaker, after the liquid has been vigorously stirred and the beaker placed on a black surface. Dry the sieve and residue for 1 hour at 105° to 110°C., cool, and weigh. Calculate the percentage of residue retained on the coarsest sieve, based on the insoluble siliceous material. (If the material forms lumps or aggregates on washing with water, a camel's-hair brush may be used on the sieve.)

3.3 In a similar manner transfer all of the material (liquid and solid) that has passed through the coarsest sieve to the next coarsest sieve and wash with water until 200 milliliters of the liquid passing through the sieve into a clean, 400-milliliter beaker fails to show any particles collected about the middle of the bottom of the beaker after the liquid has been vigorously stirred and the beaker placed on a black surface. Dry the sieve for 1 hour at 105° to 110°C., cool, and weigh. Add the weight of the residue retained on the coarsest sieve to the weight of the residue found on the second sieve and calculate the sum to percentage of residue retained on the next coarsest sieve, based on the insoluble siliceous material.



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May 9, 1975

SAMPLING

Scope: See method 101.2.

1. Sampling and acceptance procedures of soaps containing synthetic detergents shall be the same as specified in method 101.2 for soaps and soap products.

FED. STD. TEST METHOD 536/6701  
 May 9, 1975

CLEANING EFFICIENCY

Definition: This method describes a cleaning efficiency test to determine the cleaning efficiency of a hard surface cleaner.

Scope: This method is applicable to all cleaning compounds including soap products and synthetic detergents.

1. PROCEDURE. This procedure shall be carried out five times for each cleaning solution tested, using five separate test panels.

1.1 Panel precleaning. Cut an 18 inch piece from the supply of cove base (see 3.8) to fit the washing tray of the Gardner Washability machine (see 3.1). The 18 inch panel shall then be scrubbed by hand using a cellulose sponge (see 3.3) saturated with a solution consisting of a 0.5 wt percent solution of linear alkylaryl sodium sulfonate (85 percent active) (see 3.14) in tap water. Rinse the panel under cool running tap water and then drain for 10 minutes. Blot the remaining water with a paper towel. Oven dry for 15 minutes at 100°C and then allow to cool at room-temperature. Determine the reflectance of the panel (see 2.1) and record.

1.2 Panel soiling.

1.2.1 Preparation of soiling mixture.

<u>Soiling mixture (see 3)</u>	<u>Parts by weight</u>
Metallic brown	20
Odorless Kerosene (VV-K-220)	12
Stoddard solvent (P-D-680, type I)	12
White mineral oil (USP)	1
Lubricating oil (SAE 10)	1
Hydrogenated vegetable shortening	1

Combine oils and shortening and heat to 41° + 1°C to melt. Add half the kerosene. With a spatula, mix in the metallic brown. Add remaining kerosene and Stoddard solvent, and mix for 2 hours with a magnetic stirrer.

1.2.2 Soiling procedure.

While the bulk lot of soil is continuously stirred to insure uniformity, a sample of approximately 5 g is removed and applied along entire length of the vinyl panel. A Byrd applicator or "Doctor" blade (see 3.4), set at 0.008 inch clearance and 2 inches width, is used to provide an even spread.

After soil application, air-dry panels at least 30 but not more than 60 minutes, then cure panels on smooth surface in a forced draft oven at 100 + 3°C for 60 minutes. Remove the panels from oven and cool to room-temperature, using the panels within 24 hours.

1.3 Panel washing.

1.3.1 Preparation of test detergent. Synthetic hard water shall be prepared according to the following formula:

0.132 gram CaCl<sub>2</sub>·2H<sub>2</sub>O (CP).

0.1475 gram MgSO<sub>4</sub>·7H<sub>2</sub>O (CP).

Accurately weigh the salts and dissolve them in a small portion of distilled or deionized water, quantitatively transfer them to a one liter volumetric flask and make up to volume. Dry detergents shall be used in this hard water at 0.5 weight percent concentrations or at a recommended concentration, and liquid products shall be tested at the recommended use concentration.

1.3.2 Washing procedure. The sponge (see 3.3) is cut to size when dry to fit the sponge box. The thickness of the sponge shall be 1 inch when wet. Total weight of the box and dry sponge shall be 1 pound + 1/2 ounce. Soak panel for 60 seconds in wash solution sufficient to cover entire panel. Wet sponge with 25 + 3°C tap water and

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squeeze damp-dry. Add 50 ml of detergent solution to sponge and insert sponge in box. Center test panel in washability pan with two properly sized pieces of vinyl molding. Start apparatus set at one stroke per second (one cycle back and forth is two strokes), while dripping detergent solution from a pipet onto test panel center at a rate of 12 ml of solution during the wash period of 100 strokes. Stop apparatus, remove and rinse test panel under a light stream of  $25^{\circ} \pm 3^{\circ}\text{C}$  tap water. Drain and replace panel in apparatus in the reverse direction. Rinse sponge in  $25 \pm 3^{\circ}\text{C}$  tap water, squeeze damp-dry, add 50 ml of detergent solution to unused side, replace sponge in box and repeat 100 stroke wash cycle as before. Rinse panel and air dry. Determine the reflectance (see 2.1) of the washed portion of the panel and record.

## 2. EVALUATION OF SAMPLE CLEANER

2.1 Reflectance measurement. Using a photometer (see 3.2) and search unit equipped with tristimulus green filter, reflectance of the surface of the test panel is measured before soiling and after washing using a vinyl panel template of the dimensions given in figure I. Four reflectance measurements shall be made of different areas of the soiled and washed portion of each panel examined. The four readings are measured to the nearest 0.5 percent, and averaged. This procedure shall be repeated for each of the five panels required to test each cleaner. Between panels, the search unit is rested on a standard white plaque of approximately 80 percent reflectance and the instrument checked before each series of readings.

2.2 Cleaning efficiency (C.E.).

$$\text{Percent cleaning efficiency} = \frac{R_2}{R_1} \times 100$$

Where  $R_1$  = average reflectance of unsoiled, precleaned panel.  
 $R_2$  = average reflectance of soiled, test washed panel.

Report the cleaning efficiency results as the average of the cleaning efficiencies obtained from the five test panels. The results shall be reported to the nearest unit percent.

2.3 Cleaning Efficiency Comparison Index (C.I.). If required, the cleaning efficiency of the test detergent can be compared with the cleaning efficiency of the comparison detergent, using the same batch of soiling mixture and vinyl molding from the same batch.

2.3.1 The comparison detergent shall be prepared as described in 1.3.1 and shall have the following composition (see 3):

	<u>Weight percent</u>
Sodium dodecylbenzene sulfonate (85 percent)	23.5
Sodium tripolyphosphate, granular	40.0
Sodium metasilicate pentahydrate (ASTM D537)	7.0
Sodium sulfate, anhydrous (ACS)	29.5

2.3.2 Calculation of cleaning efficiency comparison index. The comparison index (C.I.) shall be calculated as follows:

$$\text{C. I.} = \frac{\text{C. E. of detergent sample}}{\text{C. E. of comparison detergent}}$$

The comparison index shall be reported to the nearest hundredth of a unit.

## 3. SPECIAL APPARATUS AND MATERIALS

3.1 Straightline Washability Apparatus (Henry A. Gardner Laboratory, Inc., Bethesda, MD 20014).

3.2 Photovolt Model 670 or equal with a 610Y search unit and tristimulus green filter (range 520 to 540 millimicrons) manufactured by Photovolt Corp., 1115 Broadway, New York, NY 10010 has been found suitable.

3.3 Cellulose sponge conforming to type II of L-S-626.

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3.4 Gardner adjustable blade ("doctor" blade) Model AG 3820 A (Henry A. Gardner Laboratory, Inc., Bethesda, MD 20014).

A substitute for the "doctor" blade is the Byrd Applicator AR-3800 of 2 inch width and a clearance of 8 mils (Henry A. Gardner Laboratory, Inc., Bethesda, MD 20014).

3.5 Template of vinyl panel, 2 inches by 4 inches with four evenly spaced holes for making reflectance readings. See figure I.

3.6 Standard white plaque (reflectance approximately 80 percent).

3.7 Forced draft oven having  $\pm 2^\circ\text{C}$  tolerance.

3.8 White Vinyl Cove Base.

General Services Administration  
Federal Supply Service  
Chemicals Branch (FMBC)  
Washington, DC 20406

3.9 Hydrogenated Vegetable Shortening

Supplier: Several Sources  
"Crisco" - Procter and Gamble Co.

Product: Hydrogenated vegetable shortening.

Trade Name: Crisco.

3.10 Kerosene, Odorless

Supplier: Miscellaneous.  
Witco Chem. Co., Inc.  
Sonneborn Div.  
277 Park Avenue  
New York, NY 10017

Product: Odorless kerosene

Trade Name: Dispersol - Ottoson Solvents, Inc.  
Deo-Base - Witco Chem., Sonneborn Div.

Specification: VV-K-220.

3.11 Lubricating Oil (SAE 10)

Supplier: Sears Roebuck and Co.

Trade Name: Regular Motor Oil - ML.

Specification: SAE 10-ML.

3.12 Metallic Brown

Supplier: General Services Administration  
Federal Supply Service  
Chemicals Branch (FMBC)  
Washington, DC 20406

Product: Metallic Brown

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3.13 Sulframin 85 (R)Linear dodecylbenzene sodium sulfonate  
(85 percent active)

Supplier: Witco Chemical Corporation

Product: Commercial Linear alkylaryl sodium  
sulfonate, 85 percent active ingredient.3.14 Sodium Metasilicate, PentahydrateSuppliers: Philadelphia Quartz Co.  
1123 Public Ledger Bldg.  
Philadelphia, PA 19106Diamond Shamrock Chemical Co.  
300 Union Commerce Bldg.  
Cleveland, OH 44115Product: Technical  $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ .

Specification: ASTM D537.

3.15 Sodium Sulfate, Anhydrous

Supplier: Fisher Scientific Co.

Product: Anhydrous  $\text{Na}_2\text{SO}_4$   
Catalog No. S-420 or Catalog No. S-421  
for ACS grade.3.16 Sodium Tripolyphosphate, GranularSuppliers: Monsanto Company  
800 N. Lindbergh Blvd.  
St. Louis, MO 63166FMC Corporation  
Inorganic Chemicals Division  
633 3rd Avenue  
New York, NY 10017Product:  $\text{Na}_5\text{P}_3\text{O}_{10}$ 3.17 Stoddard Solvent

Supplier: Miscellaneous

Product: Drycleaning solvent

Specification: P-D-680, type I.

3.18 White Mineral Oil (USP)

Supplier: Miscellaneous

Specification: USP.

FED. STD. TEST METHOD 536/6701

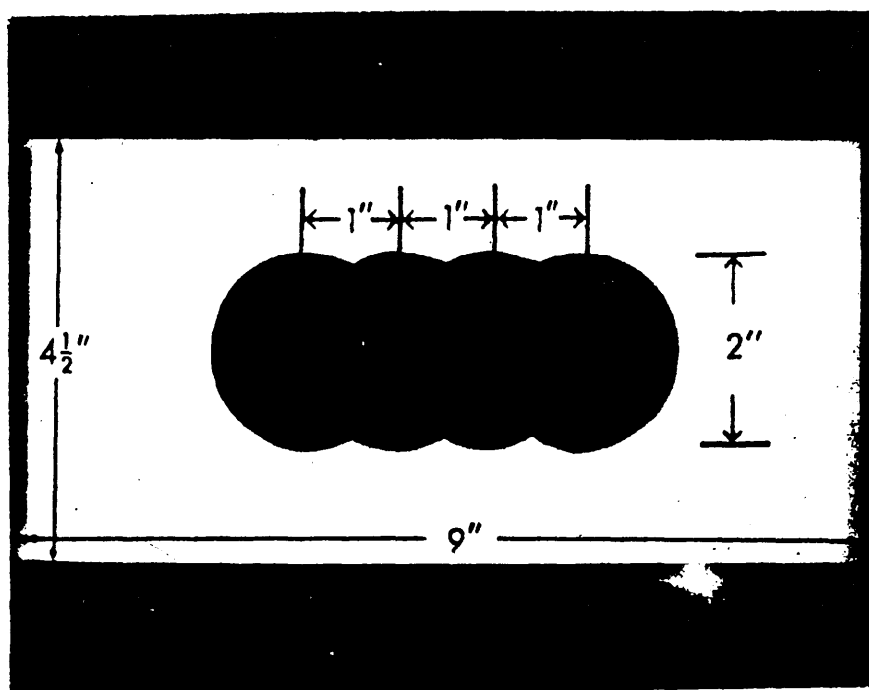


Figure I. Template Dimensions.