CS43–32
Oils: Sulphonated (Sulphated), Saponifiable Types

U. S. DEPARTMENT OF COMMERCE
ROY D. CHAPIN, Secretary
BUREAU OF STANDARDS
LYMAN J. BRIGGS, Acting Director

GRADING OF SULPHONATED (SULPHATED) OILS SAPONIFIABLE TYPES

COMMERCIAL STANDARD CS43–32

[Issued January 26, 1933]
Effective date for new production September 1, 1932

A RECORDED STANDARD OF THE INDUSTRY

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PROMULGATION STATEMENT

At the request of a large user of sulphonated oils, a general conference of representative manufacturers, distributors, and users of sulphonated (sulphated) oils, saponifiable types, on June 30, 1932, adopted Commercial Standard CS43-32 for this commodity. The industry has since accepted and approved for promulgation by the Department of Commerce through the Bureau of Standards the commercial standard as shown herein.

The standard became effective for new production on September 1, 1932.

Promulgation recommended.

Promulgated.

Promulgation approved.

I. J. FAIRCHILD,
Chief, Division of Trade Standards.

LYMAN J. BRIGGS,
Acting Director, Bureau of Standards.

ROY D. CHAPIN,
Secretary of Commerce.
GRADING OF SULPHONATED (SULPHATED) OILS
SAPONIFIABLE TYPES

COMMERCIAL STANDARD

PURPOSE

1. This commercial standard is established to provide a basis of understanding for the guidance of producers, distributors, and users, in order to eliminate confusion resulting from a diversity of opinion relative to the grading of sulphonated (sulphated) oil.

SCOPE

2. This specification covers the method of grading sulphonated (sulphated) oils, saponifiable types, which split off their organically combined SO₃ upon boiling with mineral acids, and includes definition, nomenclature, and methods of analysis.

DEFINITION

3. For the purposes of these specifications, the term “sulphonated oil” shall designate the product of interaction between a saponifiable oil or fat or its fatty acids, or a mixture of saponifiable oils or fats or their fatty acids, and sulphuric acid or similar sulphonating agent, the reaction to take place under such conditions that some or all of the oil is converted into a sulphate. With the exception of water and alkali, it shall contain no other admixture.

NOMENCLATURE

4. A sulphonated oil of any particular designation shall contain no other oil or fat except the kind designated. For example, an oil sold as sulphonated castor oil shall be manufactured from no other oil or fat except castor oil.

STRENGTH

5. The strength or concentration of a sulphonated oil shall be expressed as the sum of the percentages by weight of the following active ingredients as determined under “methods of analysis”:

(a) Total fatty matter.
(b) Total alkali bound as soap, calculated as (Na – 1).
(c) Neutralized organically combined SO₃, calculated as (SO₃Na – 1).

METHODS OF ANALYSIS

6. (a) Total fatty matter.—Weigh 5 to 10 g of the oil into a 500 ml Erlenmeyer flask and add 25 ml of water and 30 ml of concentrated hydrochloric acid. Boil while shaking for about 15 minutes, or until
the oil and water layers become clear; cool, extract the fat three times with 50 ml portions of ethyl ether, and wash the combined ether layers three times with 10 ml portions of water, or until the wash water is neutral to methyl orange. Evaporate the ether and dry the residue to constant weight at 105° C. The weight of the dry residue expressed as a percentage of the weight of the oil taken for analysis is the total fatty matter in per cent.1

(b) Total alkali bound as soap calculated as \((Na - 1)\).—Weigh 10 g of the oil into a 250 ml flask, dissolve in 150 ml of water, warming to obtain solution, if necessary. Add 30 g of granulated sodium chloride, 25 ml of ether, and 5 ml of methyl orange indicator (0.1 per cent solution) and titrate with approximately 0.5 \(N\) sulphuric acid. Convert the amount of acid required to its equivalent in milligrams of KOH per gram of sample. Let this value equal \(A\).

Note.—It is to be noted that when the sulphonated oil contains free alkali special methods of analysis are required.

For the value to be added to other active ingredients, calculate as follows:

\[
A = \text{milligrams of KOH per gram of sample} \\
= \frac{\text{ml } H_2SO_4 \times \text{titer of the acid used}}{\text{weight of the sample}}
\]

Note.—The "titer" of a solution in this standard is the normality equivalent of the solution expressed as the number of milligrams of KOH per millilitre of the solution.

Per cent combined sodium = \(A \frac{(Na - 1)}{56.1 \times 10} = A \times \frac{23 - 1}{561} = A \times 0.0392\)

Example.—Let 15.9 = number of millilitres of sulphuric acid solution required and 28.1 = titer of this solution.

Then

Per cent combined sodium = \(\frac{15.9 \times 28.1 \times 0.0392}{10} = 1.75\) per cent

(c) Neutralized organically combined \(SO_3\) calculated as \((SO_3Na - 1)\).—

(1) Combined \(SO_3\): Weigh 8 g of the oil into a 300 ml flask and boil for 1 hour under a reflux condenser with 25 ml of normal sulphuric acid, using glass beads to prevent bumping. Shake frequently. Rinse the condenser, disconnect the flask and cool. Add about 20 ml of ether, 100 ml of water, 30 g of granulated sodium chloride, and 5 ml of methyl orange indicator, 0.1 per cent solution. Titrate with 0.5 \(N\) sodium hydroxide. Frequently stopper and shake the flask during the titration. Deduct the equivalent of the added \(H_2SO_4\) from the quantity of sodium hydroxide required for the titration and calculate the difference to milligrams of KOH per gram of sample. Let this value equal \(F\).

\[
F = \frac{(\text{ml } NaOH \text{ required} \times \text{titer } NaOH) - (25 \times \text{titer } H_2SO_4)}{\text{weight of sample}}
\]

1 The "Total fatty matter" obtained in section 6 (c) is actually too high by 1 hydrogen atom for each "sodium" found in 6(b) and for each "SO_3Na" group found in 6(c)(2). Although the error introduced is small, the atomic weight of hydrogen (1) is introduced into the calculations in 6(b) and 6(c)(2), in order to have the sum of the active ingredients of the oil, section 6, correct.
Then the percentage of combined $SO_3$ equals the sum of $F$ plus $A$, obtained under "total alkali," multiplied by 8 and divided by 56.1, or per cent combined $SO_3 = \frac{(F + A) \times 8}{56.1} = 0.1426 (F + A)$.

Example.—
Let 45.5 = number of ml of NaOH required.
27.6 = the titer of this NaOH solution.
56.3 = the titer of the sulphuric acid solution used.

For $A$ see calculations under "total alkali."

Then

$$\text{Per cent combined } SO_3 = 0.1426 \times \left[ \frac{(45.5 \times 27.6) - (25.0 \times 56.3)}{8} + \frac{15.9 \times 28.1}{10} \right] = 3.67 \text{ per cent}$$

(2) Neutralized organically combined $SO_3$ calculated as $(SO_3Na - 1)$.

The percentage of neutralized combined $SO_3$, calculated as $(SO_3Na - 1)$

$$= \text{Per cent combined } SO_3 \times \frac{SO_3Na - 1}{SO_3}$$

$$= \text{Per cent combined } SO_3 \times \frac{102}{80}$$

$$= \text{Per cent combined } SO_3 \times 1.275$$

Example.—Neutralized combined $SO_3$ equals $1.275 \times 3.67 = 4.68 \text{ per cent.}$

**HISTORY OF PROJECT**

On April 3, 1931, a large user of sulphonated oil requested the cooperation of the Bureau of Standards in the establishment of a commercial standard for that product.

Upon investigation, the bureau found that there was a real demand for such a standard and that the industry would be glad to cooperate in the undertaking. Therefore, Ralph Hart, of the Hart Products Corporation, in collaboration with I. Silverman and R. Wexler prepared a tentative draft of a proposed specification which was discussed at a manufacturers' preliminary conference in New York on November 24, 1931.

This conference was unable to agree on the proposed specification and invited C. P. Gulick, Ralph Hart, I. Silverman, William Seltzer, and E. I. Rice to prepare a specification for consideration at a future meeting.

Accordingly, on April 28, 1932, the manufacturers met and approved their committee's specification with the request that it be considered by a general conference representing the entire industry.

Agreeable to this request a general conference of manufacturers, distributors, and users of sulphonated oils met on June 30, 1932, and adopted the specification with the recommendation that it be circulated to the industry for approval.

The conference approved application of the certification plan, which provides for the listing of those companies who are willing to certify that the grading of their sulphonated oils conforms to the requirements of this commercial standard.
The standard became effective on September 1, 1932, with the understanding that it be considered by the standing committee for revision once yearly.

The conference elected the following standing committee:

Manufacturers:
Ralph Hart (chairman), treasurer, Hart Products Corporation, New York, N. Y.
I. Silverman, L. Sonneborn Sons (Inc.), Belleville, N. J.
George W. Aspey, Jacques-Wolf Co., Passaic, N. J.

Distributors:
C. P. Gulick, National Oil Products Co., Harrison, N. J.
J. L. Schroeder, vice president, A. Klipstein & Co. (Inc.), New York, N. Y.
C. L. Schuttig, sales manager, John Campbell & Co., New York, N. Y.

Users:
Hill Hunter, Proximity Manufacturing Co., Greensboro, N. C.
P. J. Wood, Apex Oriental Corporation, Haledon, N. J.
Hugh Christenson, Arlington Mills, Lawrence, Mass.
ACCEPTANCE OF COMMERCIAL STANDARD

(This sheet properly filled in, signed, and mailed to the address indicated will provide for the recording of your organization as an acceptor of the commercial standard)

Date

DIVISION OF TRADE STANDARDS,
BUREAU OF STANDARDS,
WASHINGTON, D. C.

GENTLEMEN: Having considered the statements on the reverse of this sheet we hereby record our acceptance of the commercial standard as our standard practice in the production, distribution, use of standard grading of sulphonated (sulphated) oils, saponifiable types, for one year beginning (Date), or until the present standard is revised.

Realizing that the value of any commercial standard depends upon the amount of active support it receives, we will use such effort as may be appropriate to secure additional adherence whenever the opportunity offers.

Furthermore, we plan to cooperate with the standing committee in every reasonable way to assist it in the intelligent consideration of constructive revisions to be presented for adoption in accordance with established commercial standards procedure.

Signature

(Above signature should be in ink)

(Kindly typewrite or print the following lines)

Title

Company

Street address

City and State

1 Please designate which group you represent by drawing lines through the other two. In the case of related interests, trade papers, colleges, etc., desiring to record their general approval, the words "In principle" should be added after the signature.
TO THE ACCEPTOR

The following information is given in answer to the usual questions arising in connection with the acceptance form:

1. Commercial standards are commodity specifications voluntarily established by mutual consent of the industry. They present a common basis of understanding between the producer, distributor, and consumer and should not be confused with any plan of governmental regulation or control. The Department of Commerce has no regulatory power in the enforcement of their provisions, but since they represent the will of the industry as a whole, their provisions through usage soon become established as trade customs.

2. The acceptor's responsibility.—The purpose of commercial standards is to establish for specific commodities nationally recognized grades or consumer criteria and the benefits therefrom will be measurable in direct proportion to their general recognition and actual use. Instances will occur when it may be necessary to deviate from the standard and the signing of an acceptance does not preclude such departures; however, such signature indicates an intention to follow the commercial standard where practicable, in the production, distribution, or consumption of the article in question.

3. The department's responsibility.—The function performed by the Department of Commerce in the establishment of a commercial standard is fourfold; first, to act as an unbiased coordinator to bring all branches of the industry together for the mutually satisfactory adjustment of trade standards; second, to supply such assistance and advice as past experience with similar programs may suggest; third, to canvass and record the extent of acceptance and adherence to the standard; and fourth, to add all possible prestige to the enterprise by publication and promulgation when accepted by the industry.

When the standard has been endorsed by companies representing a satisfactory majority of production, the success of the project is announced. If, however, in the opinion of the standing committee of the industry or the Department of Commerce, the support of any standard is inadequate, the right is reserved to withhold promulgation and publication.
ACCEPTORS

ASSOCIATIONS

Better Fabrics Testing Bureau, New York, N. Y.
Laundryowners National Association of the United States and Canada, Joliet, Ill.
National Association Institute of Dyeing and Cleaning (Inc.), Silver Spring, Md.
National Association of Wool Manufacturers, Boston, Mass.

FIRMS

Aberfoyle Manufacturing Co., Chester, Pa.
Acadia Mills, Boston, Mass.
Alabama Polytechnic Institute, School of Textile Engineering, Auburn, Ala. (in principle).
Althouse Chemical Co., Reading, Pa. (in principle).
American Felt Co., New York, N. Y.
American Finishing Co., Memphis, Tenn.
American Mills Co., The, Waterbury, Conn.
American Woolen Co., New York, N. Y.
Anchor Color & Gum Works, Dighton, Mass.
Apex Chemical Co. (Inc.), New York, N. Y.
Apex Oriental Corporation, Paterson, N. J.
Arkansas Co. (Inc.), New York, N. Y.
Arnold, Hoffman & Co. (Inc.), Providence, R. I.
Arabol Manufacturing Co., The, New York, N. Y.
Asheville Cotton Mills, Asheville, N. C.
Associated Dyeing & Printing Co. (Inc.), Paterson, N. J.
Avondale Mills, Birmingham, Ala.
Barre Wool Combing Co. (Ltd.), The, South Barre, Mass.
Belmar Dye Works (Inc.), Astoria, L. I., N. Y.

Bernat & Sons Co., Emile, Jamaica Plain, Mass.
Bigelow-Sanford Carpet Co., Amsterdam, N. Y.
Bigelow-Sanford Carpet Co. (Inc.), Thompsonville, Conn.
Blumenthal & Co. (Inc.), Sidney, Shelton, Conn.
Botany Worsted Mills, Passaic, N. J.
Bradford Cotton Mills, Prattville, Ala.
Bradford Dyeing Association, Bradford, R. I.
Broadalbin Knitting Co. (Ltd.), The, Broadalbin, N. Y.
Burbart-Schier Chemical Co., Chattanooga, Tenn.
Callaway Mills, La Grange, Ga.
Campbell & Co., John, New York, N. Y.
Carbie Color & Chemical Co. (Inc.), New York, N. Y.
Charlton Woolen Co., Chariton City, Mass.
Ciba Co. (Inc.), New York, N. Y.
Clark Thread Co., The, Bloomfield, N. J.
Clemson A. & M. College, textile department, Clemson College, S. C.
Cliffside Mills, Cliffside, N. C.
Columbia Mills (Inc.), The, Minetto, N. Y.
Conestoga Cotton Mills (Inc.), Lancaster, Pa.
Cramerton Mills (Inc.), Cramerton, N. C.
Dacotah Cotton Mills, Lexington, N. C.
Dennis Co., The Martin, Newark, N. J.
Diamond Braiding Mills, Chicago Heights, Ill.
Ducas Co. (Inc.), B. P., New York, N. Y.
DuPont de Nemours & Co., E. I., Chicago, Ill.
Dystuffs Corporation of America, Boston, Mass.
Eccleston Chemical Co., The, Detroit, Mich.
Eno Cotton Mills, Hillsboro, N. C.
Esselen, jr., Gustavus J., Boston, Mass.
Forstmann & Co., Julius, Passaic, N. J.
Franklin Oil & Gas Co., The, Bedford, Ohio.
French Textile School, A., Atlanta, Ga. (in principle).
Fulton Dye & Import Co. (Inc.), New York, N. Y.
Gotham Silk Hosiery Co. (Inc.), Wharton, N. J.
Granite Finishing Works, Haw River, N. C.
Great Lakes Thread & Yarn Co., Detroit, Mich.
Greenville Finishing Co. (Inc.), Greenville, R. I.
Guerin Mills (Inc.), (Rosemont Mill), Woonsneck, R. I.
Hart Products Corporation, The, New York, N. Y.
Haynes Mills (Inc.), Cliffside, N. C.
Herrick-Voight Chemical Corporation, Bayonne, N. J.
Hochstadder Laboratories (Inc.), New York, N. Y. (in principle).
Hubbard Drycleaning & Redyeing School, Silver Spring, Md. (in principle).
Kem Products Co. (Inc.), Newark, N. J.
Klipstein & Co. (Inc.), A., New York, N. Y.
Lavonia Manufacturing Co., Lavonia, Ga.
Lawrason & Co. (Ltd.), S. F., London, Ontario, Canada.
Leomar Processing Corporation, Providence, R. I.
Lowell Bleachery South, Griffin, Ga.
Lowell Textile Institute, Lowell, Mass.
Mansure Co., E. L., Chicago, Ill.
Manufacturers Soap & Chemical Co., Cleveland, Tenn.
Marden-Wild Corporation, Somerville, Mass.
McClellan & Son, Chas. P., Fall River, Mass.
Mellon Institute, Pittsburgh, Pa.
Minneola Manufacturing Co., Gibsonville, N. C.
Montgomery Co., Windsor Locks, Conn.
Monument Mills, Housatonic, Mass.
National Oil Products Co., Harrison, N. J.
Neutrasol Products Corporation, Jersey City, N. J.
North Carolina Finishing Co., Salisbury, N. C.
Nyanza Color & Chemical Co. (Inc.), New York, N. Y.
Onyx Oil & Chemical Co., Jersey City, N. J.
Pacific Mills, Boston, Mass.
Pacific Mills, worsted division, Lawrence, Mass.
Passaic Print Works, Passaic, N. J.
Peppercoll Manufacturing Co., Biddeford, Me.
Piedmont Processing Co., Belmont, N. C.
Powdrell & Alexander (Inc.), Danielson, Conn.
Providence Drysalters Co., Providence, R. I.
Providence Dyeing, Bleaching & Calendering Co., Providence, R. I.
Proximity Cotton Mills, Greensboro, N. C.
Proximity Manufacturing Co., Greensboro, N. C.
Proximity Print Works, Greensboro, N. C.
Quidnick Dyeworks, Quidnick, R. I.
Renfrew Bleachery, Travelers Rest, S. C.
Revolution Cotton Mills, Greensboro, N. C.
Richards Chemical Works (Inc.), The, Jersey City, N. J.
Riverside & Dan River Cotton Mills (Inc.), Danville, Va.
Roanoke Mills Co., Roanoke Rapids, N. C.
Rosemary Manufacturing Co., Roanoke Rapids, N. C.
Royce Chemical Co., Carlton Hill, N. J.
Sagamore Color & Chemical Co., Boston, Mass.
Salem Oil & Grease Co., Salem, Mass.
Salisbury Cotton Mills, Salisbury, N. C.
Seacoast Laboratories (Inc.), New York, N. Y.
Searell, George W., New Bedford, Mass.
Seydel Chemical Co., Jersey City, N. J.
Seydel-Woolley Co., Atlanta, Ga.
Silk Grading & Testing Laboratory (Inc.), New York, N. Y.
Slack Corporation, John T., Springfield, Vt.
Sonneborn Sons (Inc.), L., New York, N. Y.
South Dakota State College, Brookings, S. Dak. (in principle).
Spalding Knitting Mills, Griffin, Ga.
Standard Bleachery & Printing Co., The, Carlton Hill, N. J.
Statesville Cotton Mills, Statesville, N. C.
Stone, Charles H., Charlotte, N. C.
Summerville Cotton Mills, Summerville, Ga.
Sunbury Converting Works, Sunbury, Pa.
Tabardrey Manufacturing Co., Haw River, N. C.
Textile Products Co., Providence, R. I.
Tubize Chatillon Corporation, Rome, Ga.
Union Bleachery, Greenville, S. C.
United Piece Dye Works, The, Lodi, N. J.
U. S. Appraisers' Laboratory, New York, N. Y.
U. S. Oil Co., Providence, R. I.
Waite Carpet Co., Oshkosh, Wis.
Waynesboro Knitting Co., Waynesboro, Pa.
White, Noel D., Signal Mountain, Tenn.
White & Hodges, Everett, Mass.
White Oak Cotton Mills, Greensboro, N. C.
Woburn Degreasing Co. of New Jersey, Harrison, N. J.
Wolf & Co., Jacques, Passaic, N. J.

GOVERNMENT
United States Department of the Interior, Washington, D. C.
Treasury Department, Washington, D. C.
Veterans' Administration, procurement division, Washington, D. C.
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Notice.—Those interested in commercial standards with a view toward accepting them as a basis of everyday practice in their industry, may secure copies of the above standards, while the supply lasts, by addressing the Division of Trade Standards, Bureau of Standards, Washington, D. C.