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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:

$$A = \text{milligrams of KOH per gram of sample} \\ = \frac{\text{ml H}_2\text{SO}_4 \text{ required} \times \text{titer of the acid used}}{\text{weight of the sample}}$$

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 milliliter of the solution.

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

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

Then

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

(c) *Neutralized organically combined SO₂ calculated as (SO₂Na-1).*—
(1) Combined SO₂: 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 H₂SO₄ 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*.

$$F = \frac{(\text{ml NaOH required} \times \text{titer NaOH}) - (25 \times \text{titer H}_2\text{SO}_4)}{\text{weight of sample}}$$

¹ The "Total fatty matter" obtained in section 6 (a) is actually too high by 1 hydrogen atom for each "sodium" found in 6(b) and for each "SO₂Na" 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 8, correct.

Then the percentage of combined SO_2 equals the sum of F plus A , obtained under "total alkali," multiplied by 8 and divided by 56.1, or per cent combined $\text{SO}_2 = \frac{(F+A) 8}{56.1} = 0.1426 (F+A)$.

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

$$\text{Per cent combined SO}_2 = 0.1426 \times \left[\frac{(45.5 \times 27.6) - (25.0 \times 56.3)}{8} + \frac{15.9 \times 28.1}{10} \right] = 3.67 \text{ per cent}$$

(2) *Neutralized organically combined SO_2 calculated as $(\text{SO}_3\text{Na} - 1)$.*—
The percentage of neutralized combined SO_2 , calculated as $(\text{SO}_3\text{Na} - 1)$

$$= \text{Per cent combined SO}_2 \times \frac{\text{SO}_3\text{Na} - 1}{\text{SO}_2}$$

$$= \text{Per cent combined SO}_2 \times \frac{102}{80}$$

$$= \text{Per cent combined SO}_2 \times 1.275$$

Example.—Neutralized combined SO_2 equals $1.275 \times 3.67 = 4.68$ 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:

O. 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.