SAUDI STANDARDS, METROLOGY AND QUALITY ORGANIZATION

SASO 2190:2025

Testing Methods for Synthetic Detergents – Part: Quality Test and Determination of Anionic Surface Active Agent

ICS 71.100.40

Foreword

The technical team (Detergents) of Saudi Standards, Metrology and Quality Organization (SASO) has update the Saudi Standard No. SASO 2190:2025 " *Testing Methods for Synthetic Detergents – Part: Quality Test and Determination of Anionic Surface Active Agent*" based on relevant Standards and references.

Testing Methods for Synthetic Detergents – Part: Quality Test and Determination of Anionic Surface Active Agent

1. SCOPE

This Saudi standard specifies the quality test and determination of anionic surface-active agent

2. QUALITY TEST AND DETERMINATION

2.1 Quality test of anionic surface active agent

2.1.1 Summary

According to operation (I), the anionic surface-active agent other than soap generates the complex of methylene blue and blue, and the soap is detected by liberating the fatty acid by acidic solution according to operation (II).

2.1.2 Reagents

Reagents shall be as follows:

2.1.2.1 Methylene blue solution

Add gradually the sulfuric acid 12 g to water of 500 ml and cool. Dissolve the methylene blue 0.03 g and sodium sulfate (anhydride) 50 g into this, and add water to make 1 L.

2.1.2.2 Chloroform

Wash the chloroform with methylene blue solution (1% w/v), add calcium oxide or calcium chloride to distill and by using sodium sulfate (an-hydride) Dehydrate. Use it within three days after distillation.

2.1.2.3 Sulfuric acid (50% v/v)

The sulfuric acid (50 %v/v) prepared by using sulfuric acid.

2.1.3 Operation (I)

The operation shall be carried out as follows:

2.1.3.1 Put methylene blue solution approximately 5 ml and chloroform approximately 5 ml into a test tube, stopper to shake vigorously and stand still to separate layer[(1)].

2.1.3.2 Add to this one drop of approximately 1% (w/v) or 1 % (v/v) solution of sample, after shaking up and down vigorously, stand still to separate layer, and if the chloroform layer becomes blue, it indicates the existence of anionic surface active agent[(2)].

2.1.3.3 Further, add sample solution, and when operated similarly, the chloroform layer becomes more dense blue.

Note [(1)]: The chloroform layer is ordinarily colorless but according to existence of impurities of methylene blue or of ethanol may become slight blue in some cases.

Note (2): The coexistence of nonionic surface-active agent, according to more or less emulsification phenomenon may require more time for layer separation, but it does not become interference to quality test.

2.1.4 Operation (II)

The operation II shall be carried out as follows:

2.1.4.1 Put approximately 1% (w/v) or 1 % (v/v) solution of sample of approximately 5 ml into a test tube.

2.1.4.2 Add two to three drops of sulfuric acid (50 % v/v) to this, mix by shaking make acidic at 4 or less pH value and stand still.

2.1.4.3 At this time, when the sample solution becomes white turbid according to liberation of fatty acid and separate gradually the oil state, it indicates the existence of soap. When the amount of soap is small, the detection by separating quantitatively according to (2.4) shall be carried out.

2.2 Determination of anionic surface-active agent

2.2.1 Summary

The long chain sulfonate such as alkylbenzene sulfonate, alkyl sulfate, alkyl ethoxy sulfate, alkenyl sulfonate, alkyl sulfonate or sulfate forms complex compound with Methylene blue and is dissolved in chloroform to become blue. When adding to this a sufficient amount of cationic surface active agent, the complex salt of anionic surface active agent and cationic surface active agent is generated, the methylene blue is liberated and so the anionic surface active agent can be determined.

2.2.2 Reagents

Reagents shall be as follows

2.2.2.1 Anionic surface active agent standard solution (0.004 mol/L) Weigh out sodium lauryl sulfate on the market of approximately 1.2 g in pure content conversion to the nearest 0.1 mg, after dissolving in water, transfer into a one mark volumetric flask 1000 ml, and add water up to the marked line. Calculate the factor according to the following formula.

$$f_a = \frac{S \times \frac{P}{100}}{M \times \frac{1}{250}} = \frac{2.5\,SP}{M}$$

Where

f_a = factor.

S = mass of sodium lauryl sulfate (g).

P = purity (%).

M = chemical formula weight of sodium lauryl sulfate.

Remark: Method of Purity (P) and Chemical Formula Weight (M) of Sodium Lauryl Sulfate

The value of (P) shall be obtained according to the following:

Weight out the refined sodium lauryl sulfate approximately 5 g according to recrystallization into an Erlenmeyer flask of 300 ml to the nearest 0.1 mg, add to this 0.5 mol/L sulfuric acid 25 ml by using a transfer pipet, attach a condenser and reflux by hot plate or on sand bath. Taking cares to framing, with shaking slightly the Erlenmeyer flask at times reflux it, and after the solution has become transparent and not to foam, further reflux for 2 h. After cooling, wash the inner wall of condenser by using ethanol (99.5) of 30 ml, and then after washing with a suitable amount of water, detach the condenser. After making approximately 100 ml the liquid amount by adding water, add several drops, of phenolphthalein solution (1% w/v), and titrate by 1 mol/l sodium hydroxide solution prepared by sodium hydroxide.

At the same time carry out the blank test, and calculate the purity of sodium lauryl sulfate according in the following formula:

$$P = \frac{(A - B) \times f \times M}{S \times 1000} \times 100$$

Where:

P = purity (%)

A = amount of 1 mol/L sodium hydroxide solution used for titration of sample (ml)

B = amount of 1 mol/l sodium hydroxide solution used for titration of blank test (ml)

f = factor of 1 mol/L sodium hydroxide solution

M = chemical formula weight of sodium lauryl sulfate

S = mass of sample (g).

The value of (M) shall be obtained according to the following:

Take the phenolphthalein solution (1 % w/v) of remark to obtain the above mentioned (P) as the indicator, transfer 50 ml of the titration solution after titrating with 1 mol/L sodium hydroxide solution to a 300 ml separatory funnel, add 100 ml of ethanol (99.5) (2 + 1) and extract twice petroleum ether layer, wash with water of each 50 ml twice, and after dehydrating with sodium sulfate (anhydride) concentrate to a suitable concentration to measure the distribution of number of carbons according to gas chromatograph analysis. Carry out the gas chromatograph analysis under the most suitable measuring conditions, obtain percentage of each component of sample alcohol, obtain the average molecular weight of sample alcohol, and from this obtain the chemical formula weight (M) of sodium lauryl sulfate.

Informative reference: For stationary phase liquid, use chromosolve (W) with 10 % of silicone SE–30, carry out with column tank temperature of 180oC, for carrier gas using nitrogen or helium, for detector the hydrogen flame ionization detector at thermostatic temperature.

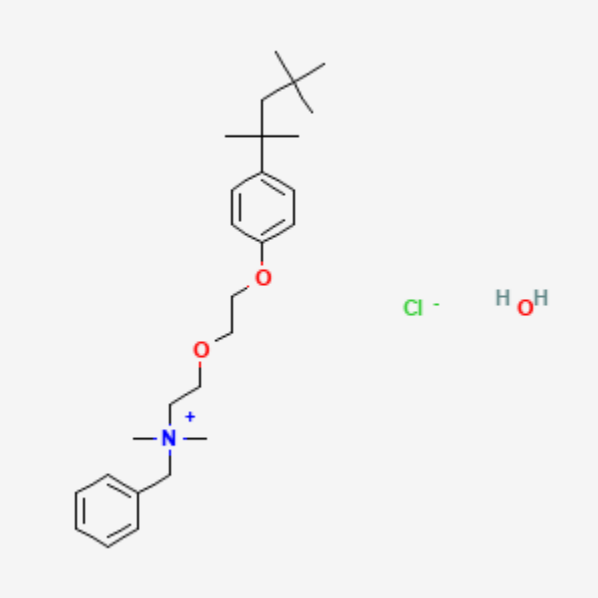
2.2.2.2 Cationic surface-active agent standard solution (0.004 mol/L benzethonium chloride solution).

The preparation and standardization shall be carried out as follows.

Preparation dissolve approximately 1.9 g of benzethonium chloride (monohydrate) on the market in water to make 1L, and take it as the cationic surface-active agent standard solution.

This standard solution 20 ml consumes anionic surface-active agent standard solution 20 ml in the blank test of following (2.4).

Informative reference: PubChem URL https://pubchem.ncbi.nlm.nih.gov



Standardization: Take anionic surface active agent standard solution (0.004 mol/l) 10 ml into a cylinder for titration, add Methylene Blue solution 25 ml and chloroform 15 ml, and then after adding water of 20 ml, titrate with cationic surface active agent standard solution. For titration at first each 2 ml, at each time stopper, and after shaking vigorously stand still. When the separation of two layers is quick, reduce sequentially the titration amount, and at near the end point carry out at each one drop (0.02 to 0.03 ml), let a white plate be background and take the time when the blue of both layers has become the same as the end point. Calculate the factor of cationic surface-active agent standard solution according to the following formula:

$$f_c = \frac{10 \times f_a}{A}$$

Where:

f_c: factor of cationic surface-active agent standard solution

A: amount of cationic surface-active agent standard solution used for titration (ml).

f_a: factor of anionic surface-active agent standard solution.

In the case of applying the back titration, method (A method) of (2.2.4.1.1) the standardization of factor of cationic surface-active agent standard solution is not required.

2.2.2.3 Methylene blue solution.

2.2.2.4 Chloroform.

2.2.2.5 Phenolphthalein solution.

2.2.2.6 0.5 mol/l sulfuric acid.

2.2.2.7 2 mol/l sodium hydroxide solution.

2.2.2.8 Add water to sodium hydroxide approximately 83 g dissolve and make 1 L.

2.2.3 Apparatus

Cylinder for titration, the measuring cylinder having stopper of 100 ml.

2.2.4 Operation

The operation shall be carried out as follows:

2.4.2.1 Determination of bond sulfuric acid of total anionic surface active agent

2.2.4.1.1 Back-titration method (method A)

a) Weight out a suitable amount (including approximately 1.4 g as pure content) of sample to the nearest 0.1 mg, add water 200 ml to dissolve by heating, after cooling, transfer to one mark volumetric flask 1000 ml, add water to make 1 L and take it as sample solution.

b) From this, take 10 ml into a cylinder for titration, add methylene blue solution 25 ml and chloroform 15 ml, and add cationic surface active agent standard solution [0.004 mol/L] 20 ml by means of a transfer pipet.

c) At first add each 2 ml of anionic surface-active agent standard solution, stopper at each time and after shaking vigorously, stand still. When the separation of two layers becomes quick, reduce sequentially the titration amount, at near the end point carry out at each one drop, take a white plate as the background and take the time when the blue of both layers has become the same as the end point.

d) At the same time, carry out the blank test.

e) Calculation

Calculate the bond sulfuric acid of total anionic surface-active agent according to the following formula.

$$C = \frac{(B - A) \times \frac{1}{250} \times f_a \times 80.06}{S \times 1000 \times \frac{10}{1000}} \times 100 = \frac{3.20\,(B - A) \times f_a}{S}$$

Where:

C: bond sulfuric acid of total anionic surface active agent (as SO3) (%)

B: amount of anionic surface-active agent standard solution used for titration of blank test (ml).

A: amount of anionic surface-active agent standard solution used for titration of sample solution (ml).

f_a: factor of anionic surface-active agent standard solution.

S: mass of sample (g).

80.06: chemical formula weight of SO_3.

2.4.2.2 Direct titration method (method B)

a) Take the sample solution 10 ml of (2.2.4.1.1) (a) into a cylinder for titration, and add methylene blue solution 25 ml, chloroform 15 ml and water 20 ml.

b) At first add each 2 ml of cationic surface-active agent standard solution, stopper at its each time and after shaking vigorously, standard still. When the separation of two layers becomes quick, reduce sequentially the titration amount, at near the end point carry out at each drop, and take the time when the blue of both layers has become the same with taking the white plate as the background as the end point.

c) Calculation

Calculate the bond sulfuric acid of total anionic surface-active agent according to the following formula.

$$C = \frac{D \times \frac{1}{250} \times f_c \times 80.06}{S \times 1000 \times \frac{10}{1000}} \times 100 = \frac{3.20 \times D \times f_c}{S}$$

Where:

C: bond sulfuric acid of total anionic surface active agent (as SO3) (%)

D: amount of anionic surface active agent standard solution used for titration of sample solution (ml)

f_c: factor of anionic surface-active agent standard solution

S: mass of sample (g)

80.06: chemical formula weight of SO_3.

2.3 Determination of bond sulfuric acid of alkyl sulfate, alkylethoxy sulfate, etc.

2.3.1 Weight out the suitable amount of sample (including approximately 1.4 g as pure content) to the nearest 0.1 mg, add water 200 ml, dissolve by heating, after cooling, transfer to one mark volumetric flask 250 ml and add water up to the marked line.

2.3.2 From this take 50 ml into an Erlenmeyer flask of 300 ml, add 0.5 mol/L sulfuric acid 50 ml by using a transfer pipet, attach a glass tube of 65 cm or more in length and heat by hot plate or on sand bath for 3 h to dissolve.

2.3.3 After cooling, wash the inner wall by pouring a small amount of water from upper part of glass tube, thereafter with using phenolphthalein solution (1% w/v) as the indicator, add 2 mol/L sodium hydroxide solution to neutralize, thereafter add water to make 200 ml, take this as the sample solution and operate according to (2.2.4.1).

2.3.4 Calculation

Obtain the bond sulfuric acid of alkyl sulfate, alkylethoxy sulfate, etc. according to the following formula.

$$C_A = C - C_u$$

Where,

C_A: bond sulfuric acid of alkyl sulfate (as SO_3) (%).

C: bond sulfuric acid of total anionic surface-active agent (as SO_3) (%).

C_u: bond sulfuric acid of non-decomposed anionic surface-active agent (as SO_3) (%).

In the case where the chemical formula weight of anionic surface-active agent in sample is already known [(1)], calculate the pure content according to the following formula:

$$P = C \times \frac{M}{80.06}$$

Where:

P: pure content (%).

C: bond sulfuric acid (as SO_3) (%).

M: chemical formula weight of anionic surface-active agent.

80.06: chemical formula weight of SO_3.

Note (1): The chemical formula weight of anionic surface-active agent to the compounded in household synthetic detergent on the market is shown in Table (1).

Table (1) Chemical formula weight of anionic surface-active agent

Anionic surface active agent	Range of chemical formula weight	Representative value
Sodium alkylbenzene sulfonate	330 to 370	348 (number of carbons 12)
Sodium alkyl sulfate	270 to 380	288 (number of carbons 12)
Sodium alkyl ethoxy sulfate	400 to 520	420 (number of carbons 12, EO 3 mol)
Sodium alkenyl sulfonate	300 to 370	325 (number of carbons 16)
Sodium alkyl sulfonate	300 to 380	328 (number of carbons 16)

2.4 Determination of soap content

2.4.1 Summary

As to the soap content, the water layer part obtained in (2.1) is acid decomposed, the generated fatty acid is extracted by petroleum ether so as the amount to be obtained, and then the neutral value of the fatty acid is measured to be obtained.

2.4.2 Reagents

Reagents shall be as follows:

2.4.2.1 0.25 mol/L sulfuric acid.

2.4.2.2 Methyl orange solution.

2.4.2.3 Petroleum ether.

2.4.2.4 Sodium sulfate (Anhydride).

2.4.2.5 0.5 mol/L potassium hydroxide solution

In this case, use potassium hydroxide 35 g and amide sulfate of 1.20 to 1.45 g.

2.4.2.6 Neutral ethanol

2.4.2.7 Phenolphthalein solution (1% w/v)

Using the phenolphthalein solution (1% w/v) s the indicator, by 0.5 mol/L potassium hydroxide solution. Prepare this solution just before using.

2.4.3 Operation

The operation shall be carried out as follows:

2.4.3.1 Collect the petroleum ether extraction residual solution of (2.1) and washings, and, using methyl orange solution as indicator; add 0.25 mol/L sulfuric acid until it becomes acidic.

2.4.3.2 Transfer to a separatory funnel, add petroleum ether 100 ml, shake, thereafter stand still to separate into two layers, transfer the lower layer to other separatory funnel, and further extract this by petroleum ether of each 25 ml three times.

2.4.3.3 Separate the petroleum ether layers, use methyl orange solution as indicator, wash with each 100 ml water until it becomes neutral, after dehydrating with sodium sulfate (anhydride), filter into an Erlenmeyer flask 300 ml of already known mass by using dried filter paper, and wash with petroleum ether.

2.4.3.4 After removing by distillation the most part of petroleum ether on water bath, after expelling the remaining petroleum ether by feeding dry air into its inside, leave the Erlenmeyer flask to cool in a desiccator and measure the mass.

2.4.3.5 Calculation

Calculate the amount of fatty acid according to the following formula:

$$F = \frac{A}{S} \times 100$$

Where:

F: amount of fatty acid (%).

A: petroleum ether extraction amount (g).

S: mass of sample of (2.1) (g).

2.4.3.6 Add natural ethanol 50 ml to Erlenmeyer flask, heat to dissolve, add phenolphthalein solution (1 % w/v) 0.5 ml as indicator, and titrate by 0.5 mol/L potassium hydroxide solution.

Take the time when maintaining extremely faint pink color for 30 s as the end point.

2.4.3.7 Calculation

Calculate the neutralization value of fatty acid according to the following formula.

$$N.V. = \frac{28.05 \times B \times f}{A}$$

Where:

N.V.: neutralization value of fatty acid.

B: amount of 0.5 mol/l potassium hydroxide solution used for titration (ml).

F: factor of 0.5 mol/l potassium hydroxide solution.

A: petroleum ether extraction amount (g).

28.05: (chemical formula weight of potassium hydroxide) x ½.

Calculate the soap content as the sodium salt according to the following formula:

$$C = F\left[1 + \frac{N.V.}{2552}\right]$$

Where:

C: soap content (%).

F: amount of fatty acid (%).

N.V.: Neutralization value of fatty acid.

$$2552 = \frac{56106}{22.990 - 1.008}$$

Where:

56106: chemical formula weight of potassium hydroxide 56.106 x 1000

22.990 – 1.008: (atomic weight of sodium) – (atomic weight of hydrogen).

Remark:

Herein the soap content in sample is calculated as fatty acid sodium salt, but in the case where it is clear that it is potassium salt or triethanol amine salt, instead of 2552 in the above formula, in the case of potassium slat use 1473, and in the case of triethanol amine salt use 376.1.