SAUDI STANDARDS, METROLOGY AND QUALITY ORGANIZATION

SASO 2192:2025

Testing Methods for Synthetic Detergents – Quality Test and Determination of Nonionic 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 2192:2025 "*Testing Methods for Synthetic Detergents – Quality Test and Determination of Nonionic Surface Active Agent*" based on relevant Standards and references.

Testing Methods for Synthetic Detergents – Quality Test and Determination of Nonionic Surface Active Agent

1. Scope and Field Application

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

2. Complementary References

2.1 SASO 2189:2003 “Testing Methods for Synthetic Detergent – Part 3: Determination of Ethanol Soluble”.

3. Quality Test and Determination of Nonionic Surface Active

3.1 Quality of test of nonionic surface active agent

The object of this method is only the nonionic surface-active agent of polyoxyethylene series. This method does not inhibit the coexistence of anionic surface-active agent, but does not apply to the case of coexistence of cationic surface-active agent.

3.2 Reagents

Reagents shall be as follows:

3.2.1 Sodium phosphomolybdate solution (10 % w/v).

Dissolve 10 g commercial sodium phosphomolybdate in water to make 100 ml.

3.2.2 Barium chloride solution (10 %w/v).

3.2.3 Hydrochloric acid (1 + 10).

3.3 Operation

The operation shall be carried out as follows:

3.3.1 Take (1 % w/v or 1 % v/v) solution 5 ml of sample into a test tube, add hydrochloric acid (1+ 10) 10 ml and barium chloride solution (10 % w/v) 10 ml and heat.

3.3.2 When turbidity or precipitate is generated, filter this, and add sodium phosphomolybdate solution (10 % w/v) 1 ml to the filtrate.

3.3.3 When nonionic surface-active agent coexists, slight yellow precipitate is generated.

3.4 Determination of nonionic surface active agent

3.4.1 Ion exchange chromatography

3.4.1.1 Summary

By using anionic exchange resin and cationic exchange resin, the anionic surface active agent and cationic surface active agent are separated and the nonionic surface active agent and the nonionic surface active agent to be eluted is determined.

3.4.1.2 Reagents

Reagents shall be as follows:

3.4.1.2.1 Ethanol (95).

3.4.1.2.2 Strongly acidic cation exchange resin

The strongly acidic low bridge formation degree ion exchange resin (H type). There are Dowex 50 WX2, Dowex 50 WX4. Amberlite XE-100, Dia ion SK 102, SK 104 etc. The grain size is 297 to 149 μm.

3.4.1.2.3 Strongly basic anion exchange resin

The strongly basic low bridge formation degree ion exchange resin (OH type). There are Dowex 1 X 2, Dowex 1 X 4. Amberlite IRA – 401, Dia ion SA 103, etc. The grain size is 297 to 149 μm.

3.4.1.3 Apparatus

Apparatuses shall be as follows:

3.4.1.3.1 Tube for column

The bore is 1 cm and the length is approximately 30 cm. The bottom part is filled with glass wool.

3.4.1.3.2 Beaker 500 ml.

3.4.1.3.3 Separatory funnel 100 ml.

3.4.1.3.4 Oven

The oven capable of maintaining temperature at 105 ± 2 °C.

3.4.1.4 Preparation of column

Flow the ion exchange resin to be used with ethanol (95) respectively into tube for column to fill, and make the height 15 to 20 cm. Assemble the column filled with strongly acidic cation exchange resin on the upper, and the column filled with strongly basic anion exchange resin on the lower.

3.4.1.5 Operation

The operation shall be carried out according to Fig. (1) Systematic Separation Figure (A).

3.4.1.5.1 Transfer 100 ml the ethanol solution obtained in the Saudi standard mentioned in items (2.1), and (2.4.1) to a separatory funnel from the beaker, flow gradually from the separatory funnel to column and receive the eluate in a beaker 500 ml.

3.4.1.5.2 Flow ethanol (95) 200 to 250 ml to wash the inside of column, join the washings with the eluate, heat on water bath to remove the ethanol, put into an oven adjusted at 105 ± 2$_o$C to dry until becoming constant amount, and weight the mass.

3.4.1.6 Calculation

Calculate the nonionic surface-active agent according to the following formula:

$$C = \frac{A \times \frac{250}{100}}{S} \times 100 - D = \frac{A \times 250}{S} - D$$

Where:

C: nonionic surface-active agent (%)

A: amount of eluate (g)

S: mass of sample in SASO (2.1) and (2.4.1) (g)

D: petroleum ether soluble content (%).

3.4.2 Alumina column chromatography

3.4.2.1 Summary

The synthetic detergent is separated to adsorption substance and nonadsorption substance according to a alumina column chromatography, and the chloroform soluble content in non-adsorption substance is taken as the amount of nonionic surface active agent.

3.4.2.2 Reagents

Reagents shall be as follows:

3.4.2.2.1 Mixed solvent

Mix the ethyl acetate and the methanol in volume ratio of 1:1.

3.4.2.2.2 Active alumina

The active alumina for chromatograph shall be (43 to 74 μm).

3.4.2.3 Apparatus

The apparatus shall be as follows:

3.4.2.3.1 Tube for column

A tube of 2.5 cm in bore and 35 cm in length, attached with a glass cock.

3.4.2.3.2 Separatory funnel 300 ml.

3.4.2.3.3 Erlenmeyer flask 100 ml, 500 ml.

3.4.2.3.4 Oven

The oven capable of maintaining temperature at 105 ± 2$_{o}$C.

3.4.2.4 Preparation of column

Fill [(1)] the absorbent cotton or glass wool in the top end of tube for column, so as the filler not to flow out. Take active alumina 80 to 100 g into a beaker, suspend with mixed solvent, flow into a tube for column so as air bubbles not to enter, and fill up to the height of 60 to 70 % of tube for column. After precipitation of active alumina, press its upper part with filter paper or cotton stopper, open the cock to flow out gradually the mixed solvent, and remain a small amount of mixed solvent at the upper part to such degree that air bubbles are not generated.

Note:

(1) After filling absorbent cotton or glass wool, after placing sea sand of 5 mm degree, if flow-in active alumina, the filling becomes easy.

3.4.2.5 Operation

The operation shall be carried out as follows according to Fig. (2). Separation systematic Figure (B).

3.4.2.5.1 Weigh out the sample(1) 2 to 3 g(2) to the nearest 0.1 mg, dissolve in mixed solvent(3) of as small amount as possible and transfer to column.

3.4.2.5.2 Open the cock, and flow out the mixed solvent to the degree not to generate air bubbles at the upper part. Wash the beaker with a small amount of mixed solvent and repeat this operation twice.

3.4.2.5.3 Next, connect the separatory funnel containing the mixed solvent 300 ml with the column, flow out at a speed of 0.3 to 0.5 ml/min, and receive(4) in an Erlenmeyer flask 500 ml.

3.4.2.5.4 Heat on a water bath to remove the mixed solvent, transfer to the Erlenmeyer flask 100 ml of the already known mass, again heat on water bath to remove the mixed solvent, thereafter enter in an oven adjusted at 105 ± 2oC to dry until becoming constant amount and weight the mass.

3.4.2.5.5 Calculation

Calculate the alumina column eluate content according to the following formula:

$$C_E = \frac{A_E}{S} \times 100$$

Where:

C_E: alumina column eluate content (%)

A_E: alumina column eluate amount (g)

S: mass of sample (g)

3.4.2.5.6 Next, add chloroform approximately 40 ml to the column eluate to heat, and in the case where insoluble matter is generated, obtain the mass of chloroform soluble matter as the amount of urea.

3.4.2.5.7 Calculation

Calculate the nonionic surface-active agent according to the following formula:

In the case where no chloroform insoluble matter is generated.

$$C = C_E - D$$

Where:

C: nonionic surface-active agent (%)

C_E: alumina column eluate content (%)

D: petroleum ether soluble content (%)

In the case where chloroform insoluble matter is generated

$$C = \frac{A}{S} \times 100 - D$$

Where:

C: nonionic surface active agent (%)

A: chloroform soluble amount (g)

S: mass of sample (g)

D: petroleum ether soluble content (%).

Notes:

(1 In the case where the sample is in pulverized state or grain state, collapse them in a mortar, and in the case where the sample is liquid, remove the moisture as far as possible according to azeotropic distillation or the like with ethanol on water bath.

(2 In the case where anionic surface-active agent coexists, take the sample so that it becomes not more than 700 mg as anionic surface-active agent.

(3 In the case where nonsoluble matter to mixed solvent exists, after suspending transfer to column.

(4 After transferring the sample to column, collect the eluate over the whole operation.

3.5 Confirmation of nonionic surface-active agent

3.5.1 Summary

The eluate obtained by ion exchange chromatography or alumina column chromatography is separated according to thin layer chromatography, and the existence of nonionic surface-active agent of polyoxyethylene series and nonionic surface-active agent of alkylolamide series is confirmed.

3.5.2 Reagents

Reagents shall be follows:

3.5.2.1 Silica gel for thin layer chromatography

The silica gel containing calcium sulfate ($CaSO_4 \cdot \frac{1}{2} H_2O$) approximately 13%.

3.5.2.2 Coloring reagents (Dragendorff varied method reagent)

3.5.2.2.1 Acetic acid (30 % v/v)

3.5.2.2.2 The matter made by dissolving basic bismuth nitrate 0.85 g in water 20 ml.

3.5.2.2.3 The matter made by dissolving potassium iodide 8 g in water 20 ml.

3.5.2.2.4 Above-mentioned (3.6.2.2.1), (3.6.2.2.2), (3.6.2.2.3) and water are mixed at volumetric ratio of 1: 1: 4: 10 directly before using.

3.5.2.3 Development solvent

Enter the solvent mixed with ethyl acetate, acetone and water at volumetric ratio of 55: 35: 10 in a development tank, and saturate the inside of tank with solvent vapor.

3.5.3 Thin layer plate

Prepare a thin layer plate of 0.3 mm in thickness, after drying in air to hardness, carry out activation in an oven at 120 ◦C for 1.5 h, and preserve in a tightly sealed container containing desiccating agent.

3.5.4 Operation

The operation shall be carried out as follows:

3.5.4.1 Make the eluate obtained in (3.4.1.5) or (3.4.2.5) approximately 5 % (w/v) solution with ethanol (95), and spot this solution approximately 5 µ1 by using micropipette at the position of approximately 1.5 to 2 cm from the lower end of thin layer plate.

3.5.4.2 After air-drying, enter in a development tank, and develop up to the height of approximately 17 cm from the origin. The development temperature should preferably be about 25 ◦C.

3.5.4.3 After air drying or drying in a drier the plate after developing, spray uniformly the coloring reagent. When having detected many spots colored to orange or reddish orange on the yellow substrate, the existence of nonionic surface-active agent of polyoxyethylene series is anticipated [(1)].

3.5.4.4 When orange color spots (usually single spot) are detected at the upper part of plate, the existence of nonionic surface-active agent of alkylolamide series is anticipate [(2)].

3.5.4.5 Otherwise, in the case where coloring is not generated by the coloring reagent, it may be considered that nonionic surface-active agent of Polyoxyethylene series and nonionic surface-active agent of alkylolamide series do not exist [(3)].

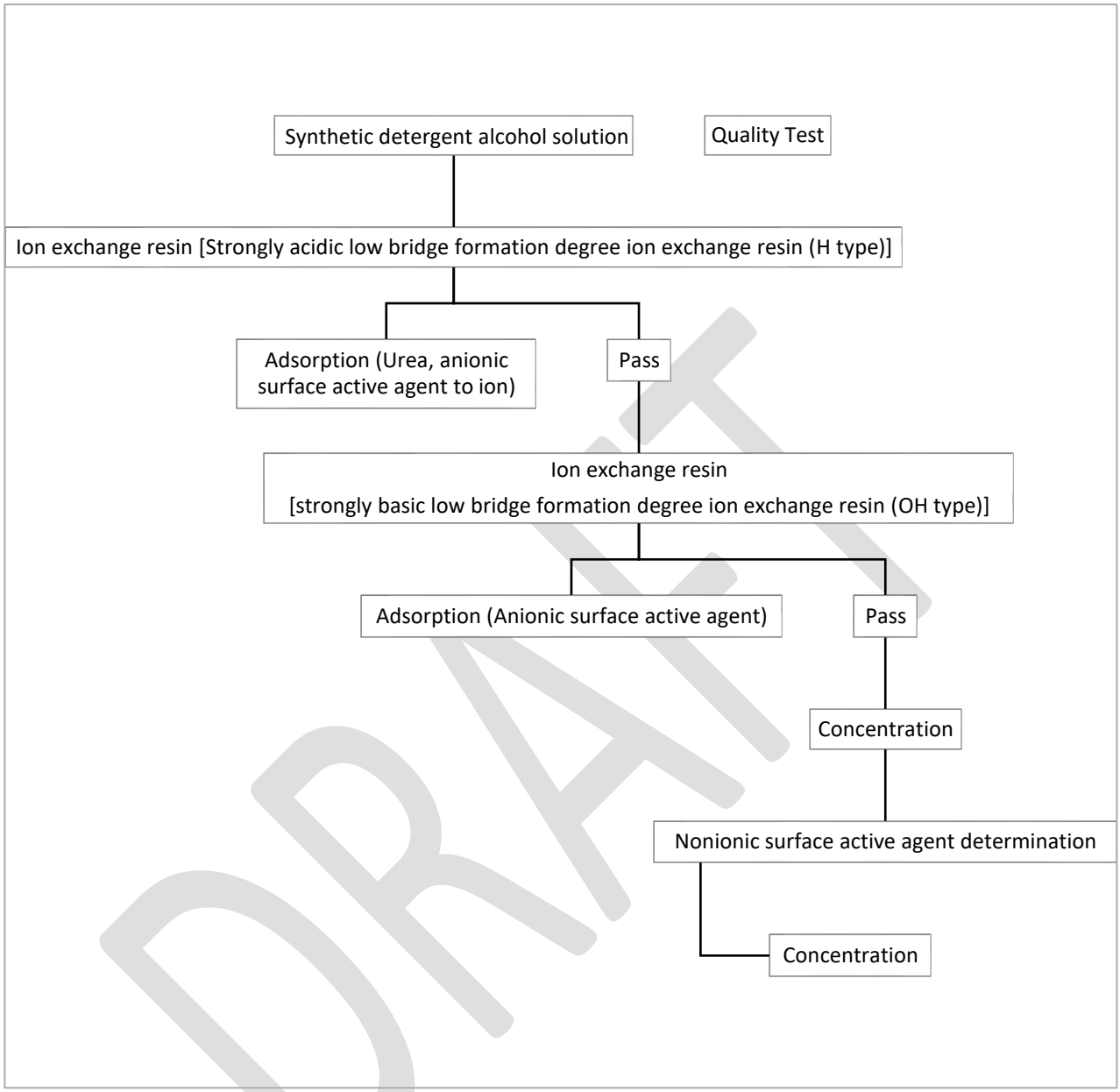
Notes:

1) The matter of large adding number of ethylene oxide is insufficient in separation and the tailings confirmed at near the origin. Further, relating to the matter of 1. 2mol or less in average adding number of ethylene oxide the mutual separating properly is good.

2) The spot of nonionic surface-active agent of alkylolamide series is thinner than the spot of nonionic surface-active agent of polyoxyethylene series. Further, in nonionic surface-active agent of alkylolamide series, several component spot is detected in some cases.

3) Relating to nonionic surface-active gent other than nonionic surface-active agents of polyoxyethylene series and of alkylolamide series and polyhydric alcohol as well as nonreacted substances (high-grade alcohol, alkylbenzene, etc). etc if, further, analyzed by using other developing solvent or by other analysis method, the estimation of component is possible

Fig (1). Systematic separation Figure (A) Ion exchange chromatography



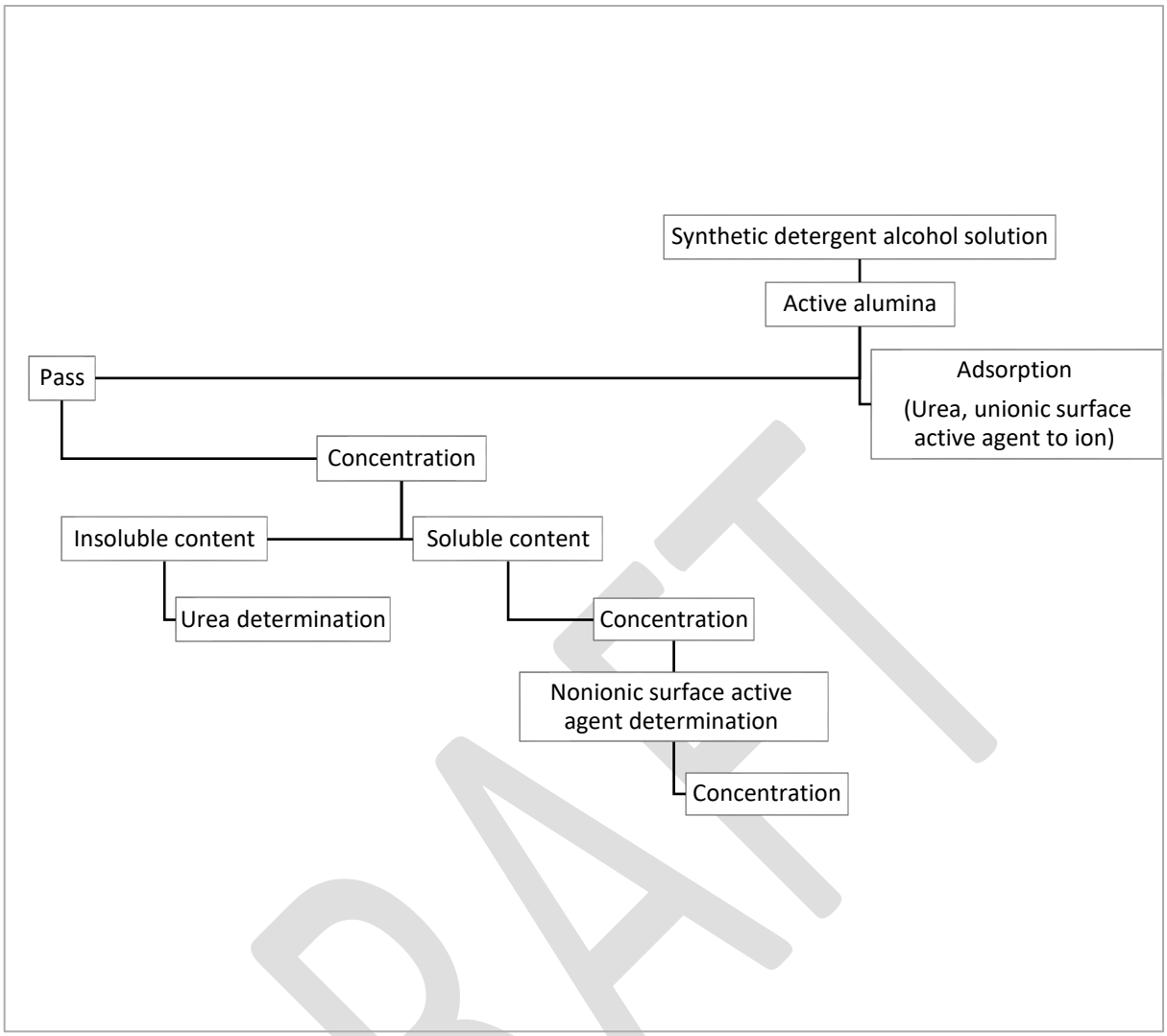


Fig. (2) Systematic Separation Figure (B) Alumina column chromatography

Appendix (A)

Ethanol Solubility Estimation

1- Summary

For ethanol solubility, the sample is dissolved in ethanol and the mass of the material dissolved in ethanol is obtained upon extraction.

2- Reagents

The reagents should be as follows:

2.1 Ethanol (95).

2.2 Ethanol (95.5).

3- Apparatus

The apparatus should be as follows:

3.1 Erlenmeyer Flask

A 300 mL Erlenmeyer flask connected to a glass tube 65 cm or longer in length.

3.2 Glass Filter

Small-hole flat-bottom filter.

3.3 Filter Receiver.

3.4 Dryer with a temperature control of 105 ± 2°C.

4- Operation

The operation should be carried out as follows:

4.1 Weigh approximately 5 grams of sample into a 300 ml Erlenmeyer flask to the nearest 1 gram. Add, connect the glass tube, and mix briefly by shaking at intervals. Place (1) 100 ml of ethanol in a water bath for 30 minutes to dissolve.

4.2 Filter the hot solution as is using a glass filter, and add another 50 ml of ethanol (95) to the remaining solution in the Erlenmeyer flask to dissolve. Filter the hot solution using a glass filter, and thoroughly wash the Erlenmeyer flask and glass filter with hot ethanol. Allow to cool to room temperature. Transfer the filtrate and wash into a 20 ml single-marked flask, and add ethanol (95) up to the upper line. From this, each 100 ml is taken separately into two 200 ml beakers of known weight using a transfer pipette.

4.3 Heat one of the beakers in a water bath. After removing the ethanol, dry it in an oven at 105 ± 2°C for one hour. After cooling in a desiccator, measure the mass.

Note (1):

For powdered or granular samples, ethanol (95) is used, and for liquid or paste samples, ethanol (95.5) is used.

5- Calculations

The content of soluble substances in ethanol is calculated according to the following equation:

$$C = \frac{A}{S \times \frac{100}{250}} \times 100 = \frac{250 \times A}{S}$$

Where:

C = ethanol soluble content (%)

A = residual amount of drying (g)

S = mass of sample (g).

In the case of carrying out the infrared absorption spectral analysis of surface active agent component, the ethanol soluble content used for determination shall be taken as sample.