

الهيئة السعودية للمواصفات والمقاييس والجودة

Saudi Standards, Metrology and Quality Org (SASO)



SASO 509:2020

**Synthetic Detergents –Method of test for Household
Synthetic Detergents Powder**

ICS: 71.100.00

Synthetic Detergents –Method of test for Household Synthetic Detergents Powder

1. SCOPE AND FIELD OF APPLICATION

This Saudi standard is concerned with the methods of test for Household Synthetic Detergents Powder.

2. COMPLEMENTARY REFERENCES

- 2.1 **SASO 510:2019** “Household Synthetic Detergent Powders”.
- 2.2 **SASO ISO 607:2011** “Surface active agents and detergents - Methods of sample division”.
- 2.3 **SASO ISO 4318:1998** "Surface active agents and soaps - Determination of water content - Azeotropic distillation method”.
- 2.4 **SASO ISO 2271:1998** "Surface active agents - Detergents - Determination of anionic-active matter by manual or mechanical direct two-phase titration procedure”.
- 2.5 **SASO ISO 4313:1994** “Washing powders - Determination of total phosphorus (V) oxide content - Quinoline phosphomolybdate gravimetric method”.
- 2.6 **SASO ISO 4321:1994**” Washing powders - Determination of active oxygen content - Titrimetric method”.
- 2.7 **SASO IS 4316:1994** “Surface active agents - Determination of pH of aqueous solutions - Potentiometric method”.
- 2.8 **SASO IS 8215:2008** "Surface active agents - Washing powders - Determination of total silica content - Gravimetric method”.

3. REQUIREMENTS

On carrying out tests the following shall be observed:

Reagents used shall be of the analytical purity grade.

Distilled water or other water of equivalent purity shall be used.

4. VISUAL INSPECTION

Samples drawn according to SASO mentioned in (2.1) shall be inspected to ensure the following:

- 4.1 That containers are suitable tightly sealed, with an adequate opening.
- 4.2 That marking information is according to SASO mentioned in (2.1).
- 4.3 That net weight conforms to mean net weight mentioned on the container with a tolerance of $\pm 2\%$.

- 4.4 Presence or absence of objectionable odor.
- 4.5 Easy flow of powder and absence of caking.
- 4.6 Homogeneity of powder.

5. PREPARATION OF SAMPLE

Test sample shall be prepared from the total sample sent to the laboratory according to SASO mentioned in (2.2).

6. DETERMINATION OF MOISTURE AND VOLATILE MATTER

Determination of moisture and volatile matter shall be according to SASO mentioned in (2.3).

7. DETERMINATION OF SURFACE ACTIVE AGENTS

Determination of surface-active agents shall be according to SASO mentioned in (2.4).

8. DETERMINATION OF PHOSPHATES PERCENTAGE

Determination of total content of phosphates shall be according to SASO mentioned (2.5).

9. DETERMINATION OF ACTIVE OXYGEN CONTENT

Determination of active oxygen content shall be according to SASO mentioned in (2.6).

10. DETERMINATION OF pH VALUE

Determination of pH value shall be according to SASO mentioned in (2.7).

11. DETERMINATION OF TOTAL SILICATES PERCENTAGE

Determination of total silicate shall be according to SASO mentioned in (2.8).

12. DETERMINATION OF RINSING PROPERTIES

12.1 Reagents

Hard water: containing (300 ppm hardness).

Prepare by placing 0.246 g. $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.295 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in 1 liter volumetric flask.

Dissolve in distilled water and dilute to the mark.

12.2 Apparatus

12.2.1 1 liter volumetric flask one mark.

12.2.2 Ordinary laboratory equipment.

12.3 Procedure

12.3.1 Dissolve 2g of test samples in 98 ml of hard water (12.1) at $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in a clean 250ml Erlenmeyer flask. Stopper and shake vigorously for one minute, then empty contents.

12.3.2 Rinse the flask 3 times using each time 75 ml of hard water (12.1). Turn the flask upside-down and allow to dry. Examine the flask for presence or absence of any precipitate.

12.3.3 Run a blank determination using a similar Erlenmeyer flask previously rinsed with the hard water (12.1) only and then dried.

12.4 Result

Compare deposit material in both flasks.

The sample flask shall be considered to have passed the test if it contained less deposit than the blank test flask.

13. DETERMINATION OF HEAVY (POISONOUS) METALS (LEAD)

13.1 All reagents used shall be of the analytical purity grade, and the water used shall be de-ionized. Prepare 1000 ml stock solutions containing 1000 (μg) of each of the elements to be analyzed, as follows:-

13.1.1 Lead, stock solution: Dissolve 1.5985 g lead nitrate in 500 ml of 1N nitric acid in 1 liter volumetric flask. Dilute to the mark with water.

13.1.2 Mercury, stock solution: Dissolve 1.080 g mercuric oxide in dilute (1:1) hydrochloric acid solution and dilute to 1 liter with water.

13.1.3 Arsenic, stock solution: Dissolve 1.320 g of arsenic oxide (pre-dried at 110°C for 1 hour and cooled in a desiccator) in 25 ml of 2% (w/v) sodium hydroxide solution,

13.1.4 Return to end point for phenolphthalein by using 20% sulphuric acid, then dilute to 1 liter with the a dilute solution (1%) sulphuric acid.

13.1.5 Cadmium, stock solution: Dissolve 1 g cadmium metal in the least possible volume of dilute hydrochloric acid (1:1), then dilute to 1 liter with the dilute (1%) acid.

13.1.6 Antimony, stock solution: Dissolve 2.743 g. antimony potassium tartrate $\frac{1}{2}$ H₂O ((Sbo)C₄H₄O₆. $\frac{1}{2}$ H₂O) in 1 liter of water.

13.1.7 Aluminum, stock solution: Dissolve 1 g aluminum wire in the least possible volume of 2 mol/l sulphuric acid, then dilute to 1 liter with water.

13.1.8 Copper, stock solution: Dissolve 1 g copper metal in the least possible volume of nitric acid. Add 5 ml hydrochloric acid, evaporate resultant to near dryness, then dilute to 1 liter with 0.1mol/l hydrochloric acid.

13.1.9 Iron, stock solution: Dissolve 1 g iron wire in dilute (1:1) nitric acid, then dilute to 1 liter with water.

13.1.10 Magnesium, stock solution: Dissolve 1 g magnesium tape in the least possible volume of dilute (1.1) hydrochloric acid, then dilute to 1 liter with the dilute (1%) acid.

13.1.11 Concentrated hydrochloric acid sp.gr. 1.18 g/cm.

13.2 Preparation of working standard solutions

These solutions are prepared by diluting specific volumes of the stock metal solutions with the acid solutions corresponding in composition to those used in the test sample solution.

13.2.1 Place, using a pipette, 10 ml from each of the stock metal solutions (13.1) into 100 ml volumetric flask, then dilute to volume with water.

13.2.2 With a pipette draw 0.5, 1, 2, 5, 10 or 20 ml (if necessary) from solution (13.2.1) into 100 ml volumetric flasks. Add 10 ml conc hydrochloric acid and dilute to 1 liter with water. Express these concentrations by $\mu\text{g/ml}$ or mg/l .

13.3 Apparatus

13.3.1 Atomic absorption spectrophotometer equipped with air/acetylene and nitrous oxide/acetylene flames.

13.3.2 Hot plate with a magnetic stirrer.

13.3.3 Lei big condenser, water – cooled, effective length 400 mm.

13.3.4 Ordinary laboratory equipment.

13.4 Preparation of sample solution

13.4.1 Weigh to the nearest, 0.001 g, approximately 2 g of the representative sample and place into 100 ml Erlenmeyer flask. Add 5 ml concentrated hydrochloric acid and 20 ml water, using a graduated cylinder.

13.4.2 Place the flask (13.2.2) with a 2.5 cm magnetic rod inside it, on the hot plate attach the condenser and run the reverse condensation for 1 hour, then allow the flask to cool.

13.4.3 Wash the condenser sides with about 10 ml water, then detach the flask and empty contents quantitatively into a 50 ml volumetric flask and make to volume with water.

13.4.4 Filter the solution using a watman No. 18 filter paper. Reserve the filtrate for carrying out the require determinations.

13.5 Procedure

13.5.1 Adjust the atomic absorption spectrophotometer according to manufacture's instructions, using the appropriate standard solution with the highest concentration.

13.5.2 Calibrate the photometer using 10 % hydrochloric acid as blank solution with any of the suitable standard solution and record absorption for each working sample. Then draw the standard curve relating concentration ($\mu\text{g/ml}$) to absorption.

13.5.3 Draw a portion of the sample solution (13.4.4), and determine absorption for each element in the sample solution.

13.6 Calculations

The concentration of any metal in the sample shall be calculated from the standard curve or by using the following relationship:

$$\text{Conc. Of metal mg/liter} = \frac{\text{Conc of metal in sample solution} \times 50}{\text{Mass of original sample (g)}}$$

14. TEST FOR PRESENCE OF A BRIGHTENING AGENT

14.1 Apparatus

14.1.1 Watman No. 4 filter paper or equivalent.

14.1.2 A source of ultra-violet radiation.

14.2 Procedure

14.2.1 Immerse about 25 mm of a filter paper strip in about 100 ml of 2% aqueous solution of the test sample at 40°C for 15 minutes, then rinse with about 300 ml of distilled water.

14.2.2 Run a blank test on a similar filter paper strip, immersed only in distilled water.

14.2.3 Place the two strips in a dark room under the source of ultra violet radiation.

14.2.4 Compare brightness in both the test sample strip and the blank test strip.

14.3 Result

Presence of a brightening agent can be confirmed if the strip containing the test sample appears brighter than the strip of blank test.

15. DETERMINATION OF SODIUM CARBONATE

15.1 Reagents

The reagents shall be high analytical grade and shall be used distilled water.

15.1.1 Hydrochloric acid solution 0.1 M.

15.1.2 Sodium hydroxide solution 0.1 N.

15.1.3 Methyl orange solution indicator.

15.1.4 Phenol phthalin solution indicator.

15.2 Apparatus

The ordinary laboratory apparatus shall be used and:

15.2.1 Calibration flask 250 ml

15.2.2 A graduate pipette 25, 50 ml

15.3 Procedure

15.3.1 Prepare the test sample accordance SASO mentioned in item (2.2).

15.3.2 Part of test sample

Weight 1 gm from the test sample to the nearest 0.001 gm and dissolve in 100 ml distilled water in calibration flask.

15.3.3 Determination

- 15.3.3.1 The solution calibrated with HCl 0.1 M and used phenol phthalin indicator, the volume to determined the active alkalinity is (m_1).
- 15.3.3.2 Continue the calibration with methyl orange indicator and HCl 0.1 M and the volume of HCl is (m_2). For carrying out the two calibration which need to determined the total alkalinity.
- 15.3.3.3 Heat the solution until boiling, where the CO_2 out.
- 15.3.3.4 After the solution reach to the boiling, remove it from the heater and cooled.
- 15.3.3.5 Calibration again after cooling with HCl 0.1 M and used methyl orange indicator.
- 15.3.3.6 Added drops from phenol phthalin indicator and calibrated with NaOH 0.1 M until the color change to the pink and the volume consume is (m_3).

Note: If the consume volumes are large, may be take 25 ml from the solution and carried out the previously calibration, the consideration weight of the sample is (W) shall be taken.

15.4 Calculation

$$\text{Sodium carbonate (m \%)} = \frac{(m_1 \times m_2 \times m_3)}{W} \times 1.06$$

Where:

m_1 = HCl volume to determined the active alkalinity (mL)

m_2 = HCl volume to determined the total alkalinity (two calibration) ml

m_3 = NaOH volume (ml)

W = mass of sample (gm)

16. DETERMINATION OF SOAP CONTENT

16.1 Principle

The cleaning solution extract by the light petroleum and concentrates the extract with the vaporization, then it titration with the solution of sodium hydroxide standard.

16.2 Reagents

The reagents shall be high analytical grade and shall be used distilled water.

16.2.1 Ethanol

The ethanol shall be pure.

16.2.2 Light petroleum (40-60°C)

16.2.3 H_2SO_4 5 M.

16.2.4 NaOH 0.1 M.

16.2.5 Bromophenol blue indicator

16.2.6 phenol phthalin indicator

Phenol phthalin solution 5 gm/L in ethanol solution 50 %.

16.2.7 Solution chloride

16.3 Apparatus

The ordinary laboratory apparatus shall be used and:

16.3.1 Separate funnel 250 ml.

16.3.2 Flask 250 ml provided with emery glass (24/29).

16.3.3 A suitable distillation apparatus for flask (16.3.2).

16.4 Producer

16.4.1 Part of sample evaporation

Weigh to the nearest 0.1 gm about 5 gm from the synthetic detergent or 3 gm approximately from the base powder of soap in beaker 150 ml.

16.4.2 Determination

16.4.2.1 Added 50 ml water to the part of test sample (16.4.1) and heat to between 40-50 °C and stir and added 50 ml ethanol with few drops from bromo phenol blue indicator solution (16.2.5).

16.4.2.2 Added H₂SO₄ solution (16.2.3) drop by drop until the solution become acidity and transfer the solution to separate turned (16.3.1) and wash. The beaker with 50 ml light petroleum (16.2.2) and added the produce washing to separate funnel.

16.4.2.3 Close the separate funnel and shaking vigorous and turned from time to time and opened the separate funnel for getting ride of the pressure, then leave the layer to separated.

16.4.2.4 Note: If there is difficulty in getting a complete separation of the layer, at the time a little amount of the sodium chloride is added or heating the mixture can help in the separation of layers.

16.4.2.5 Pull the stopper and empties the lower layer to another separate funnel and added to the second separate funnel 50 ml light petroleum.

16.4.2.6 Repeat the procedure in item (16.4.2.3) and pull the stopper and empties the lower layer in an original beaker and transfer the petroleum layer to the separate funnel which contain the first extract.

16.4.2.7 Extract the aqueous layer with 50 ml additional light petroleum by used second separate and transfer the petroleum layer to the separate funnel which contain the two extraction, wash the produce one time with water and drop the aqueous layer.

16.4.2.8 Transfer the extraction collected quantitative to the flask (16.3.2) and evaporated until the volume became 5 ml by used distillation apparatus.

16.4.2.9 Added to the flask (16.3.2) 100 ml ethanol (16.2.1) which moderate for phenol phathalin indicator and titrated with NaOH solution with used phenol phathalin indicator.

16.5 Calculation

$$\text{Soap content (m \%)} = \frac{m \times 100}{1000 \times m_1} = \frac{m \times V \times 0.1}{m_1}$$

Where:

V = NaOH consume volume (ml).

m = relative molecular weight average to the soap

m_1 = mass of sample to be test (gm).