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Surface active agents - Test methods for detergents

表面活性剂 洗涤剂试验方法

(ISO 607:1980, ISO 697:1981, ISO 4313:1976, ISO 4321:1977, ISO 4325:1990, ISO 21264:2019, MOD)

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Surface active agents - Test methods for detergents

1 Scope

This standard describes the test methods for indexes of surface active agents and detergents, such as sample reduction, particle size, total phosphorus oxide content, total active substance content, non-ionic surface active agent content, various phosphate content, toluenesulfonate content, foaming power, chelating agent (EDTA) content, apparent density, whiteness, moisture and volatile matter content, active oxygen content, 4A zeolite content, alkylphenol ethoxylates content, effective chlorine content, free alkali content, dry sodium soap content, stability at high and low temperature, odour, colour and appearance, etc.

This standard is applicable to the index determination of surface active agents and detergent products.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6003.1	*Test sieves -- Technical requirements and testing -- Part 1: Test sieves of metal wire cloth* (GB/T 6003.1-2012, ISO 3310-1: 2000, MOD)
GB/T 6682	*Water for analytical laboratory use -- Specification and test methods* (GB/T 6682-2008, ISO 3696: 1987, MOD)
JJG 512	*Whiteness meters*
QB/T 2739-2005	*Preparations of standard volumetric solutions of general test methods for washing products*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

surface active agents

The surface-active compound that dissolves in liquids, especially in water, and has a open the valve fully so as to allow the contents of the hopper to run over the cone, thus dividing the bulk sample into two portions one of which is deposited in each receiver.

Retain one of these portions and discard the other. Pass a fresh quantity of bulk sample through the conical divider and repeat the operation until all the bulk sample has been divided.

Clean the apparatus and again pass the retained portions, corresponding to half the bulk sample, through the apparatus as described above and repeat this operation until a reduced sample of the required mass has been obtained.

4.2.1.3.2 Preparation of several equivalent samples

If more than one sample is required, prepare sufficient reduced sample to obtain 2*n* equivalent samples where 2*n* equals or exceeds the number of samples required.

Subsequently, divide the reduced sample into 2*n* equal portions by means of the conical divider. Immediately place the whole of each portion in an airtight bottle or flask.

4.2.1.3.3 Preparation of test samples

If test samples are required from the laboratory samples, treat the latter as specified in 4.2.1.3.1 and 4.2.1.3.2.

The minimum mass of the test samples shall in no case be less than 10g, otherwise there is a risk that the test samples may not be truly representative of the bulk sample and, hence, will be unsuitable for test purposes.

4.2.2 Products in paste form

4.2.2.1 Apparatus

4.2.2.1.1 Scoop or spatula, for sampling.

4.2.2.1.2 Suitable mixer, provided with a beater, for blending. In general, it shall be sufficiently powerful so that, when used with a beater of suitable design, the whole of the bulk sample is mixed and a creamy mass attained within 5 min.

4.2.2.2 Preparation of a reduced sample

Warm the product (bulk sample or laboratory sample) in its original container to 35°C to 40°C, and mix immediately using the domestic mixer (4.2.2.1.2) for 2 to 3 min until a homogeneous mass is obtained.

The paste shall not be removed from the original container before mixing as this may result in the production of a non-representative sample. It is therefore essential that the bulk sample is in a container which will allow mixing without transfer.

The heating and mixing time shall be as short as possible so as to reduce to a minimum any change in the product. Using the spatula or scoop, remove immediately the required quantity of sample and transfer it into an appropriate, previously tared container, fitted with a stopper.

Allow the contents of the container to cool to room temperature, and reweigh to obtain the mass of the reduced sample.

Note: Contact of the paste with glass vessels readily causes separation of a lye; therefore, once the sample is placed in the vessel, no part shall be withdrawn to adjust the mass.

There is a slight loss of moisture during mixing and weighing but experience has shown that, in practice, this is at an acceptable level.

4.2.3 Products in liquid form

4.2.3.1 Apparatus

4.2.3.1.1 Glass flasks or weighing pipettes, for sampling.

4.2.3.1.2 Manual stirrer (for example, a glass rod).

4.2.3.1.3 Mechanical stirrer.

4.2.3.2 Preparation of a reduced sample

If the product (bulk sample or laboratory sample) is clear and apparently homogeneous, mix it with the manual stirrer (4.2.3.1.2); then, using the flask or weighing pipette (4.2.3.1.1), remove immediately the quantity required for the reduced sample. As little foam as possible shall be produced in the sample during mixing, and any loss of sample by evaporation shall be kept to a minimum.

If the product (bulk sample or laboratory sample) is cloudy or contains a sediment, mix it using the mechanical stirrer (4.2.3.1.3); then remove immediately the required quantity of sample.

If the product (bulk sample or laboratory sample) contains a solid deposit, carefully warm the original container to about 30°C until the sediment can be completely dispersed by stirring or until any crystals disappear, and remove immediately the required quantity of sample.

4.2.4 Products in block form

4.2.4.1 Apparatus

4.2.4.1.1 Knife or cutting wire, for sampling.

4.2.4.1.2 Mechanical pulverizer.

6 Determination of total phosphorus oxide content in detergents

6.1 Quinoline phosphomolybdate gravimetric method

6.1.1 Principle

Hydrolysis of polyphosphates by nitric acid. Precipitation of phosphates in the form of quinoline phosphomolybdate in acetone solution. Drying and weighing of the precipitate.

6.1.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only grade III water or above specified in GB/T 6682.

Note: it is applicable to all tests in this standard.

6.1.2.1 Nitric acid, with density approximately 1.4 g/mL, about 68% (mass fraction) solution.

6.1.2.2 Sodium molybdate citrate reagent (i.e. quinamoxycitrone precipitant): Dissolve 70g of sodium molybdate dihydrate ($Na_2MoO_4 \cdot 2H_2O$) in 150 mL of water (Solution A). Dissolve 60g of citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in 150 mL of water and add 85 mL of the nitric acid solution (6.1.2.1). Pour Solution A into Solution B (Solution C), while stirring. Dissolve 5 mL of pure quinoline (without reducing agent) in a mixture of 100 mL of water and 35 mL of nitric acid (6.1.2.1) (Solution D). Slowly pour Solution D into Solution C and mix well. Place in a polyethylene bottle, stand in the dark for 24h, and filter all the liquid through the filter crucible (6.1.3.2). Add 280 mL of acetone into the filtrate; dilute to 1,000 mL with water; mix well; and store in another clean polyethylene bottle. The solution shall be stored for no more than 7d in the dark.

6.1.3 Apparatus

6.1.3.1 Medium-speed qualitative filter paper, of ø11 cm.

6.1.3.2 Glass crucible: with sintered glass disks having a pore size of 5 μm to 15 μm.

6.1.3.3 Oven, capable of being controlled at 180°C ± 2°C.

6.1.3.4 Volumetric flask, of capacity 500 mL.

6.1.3.5 Pipette, of capacity 20 mL.

6.1.3.6 Beaker, of capacity 400 mL.

6.1.3.7 Measuring cylinder, of capacity 100 mL and 10 mL.

6.1.3.8 Surface dishes.

6.1.3.9 Glass rods.

6.1.4 Procedure

6.1.4.1 Specimen

Weigh the detergent test sample containing 125 mg ~ 500 mg phosphorus oxide (accurate to 2 mg) into a 250 mL beaker.

6.1.4.2 Determination

Add 95% ethanol to 80 mL (for liquid and paste samples, add ethanol absolute to 95% ethanol concentration) to the beaker containing the specimen. After gently stirring with a glass rod, cover the surface dish; boil it on an electric heating plate for 10 min; take it off and cool it; and filter it with a medium-speed qualitative filter paper (6.1.3.1) to keep the solid matter in the beaker as much as possible. Then wash the filter paper twice with water; and the filtrate is collected into the original beaker with solid matter.

Note 1: If it is difficult to filter when washed with water, a small hole can be punched at the bottom of filter paper to speed up washing.

Add water to 80 mL to the original beaker; gently stir with a glass rod; cover the surface dish, and heat it on an electric heating plate to dissolve the solid. Take it off and cool it; transfer it to a 500 mL volumetric flask; dilute it with water to the scale, and shake well. Filter the liquid in the volumetric flask with medium-speed qualitative filter paper (6.1.3.1) (discard the first 10 mL of filtrate). Pipette 20.0 mL of filtrate into a 400 mL beaker; add water to make the total volume 100 mL; add 8 mL of nitric acid (6.1.2.1), put into a glass rod; cover the surface dish; place it on an electric heating plate and boil slowly; and keep it slightly boiling for 40 min. Remove the beaker; carefully add 50 mL of quinamoxycitrone precipitant (6.1.2.2) while hot; do not stir; then cover the surface dish and boil for another 1 min. Take it down to allow it to stand and cool; and stir it for 3 to 4 times during the period to make the generated precipitation settle.

Note 2: When quin molybdenum ketone precipitant is added, it is easy to explode and boil, which makes the contents splash out. Therefore add reagent along the breaker wall as much as possible, slowly at first, then quickly.

Vacuum-filter with a glass crucible (6.1.3.2) dried at (180 ± 2)°C. Wash the precipitate 6 times by decanting it, using 30 mL of water each time. Then transfer the precipitate quantitatively to the glass crucible with the aid of wash-bottle; then wash it for 4 times, with 20 mL to 30 mL of water each time. During washing, add the next part of water after the previous washing water is completely filtered. Remove the glass crucible; place it in an oven (6.1.3.3) controlled at (180 ± 2)°C, and time for 45 min after the

The absolute difference between the two independent test results obtained under the reproducibility conditions shall not exceed 1.1%, under the premise that the proportion exceeding 1.1% does not exceed 5%.

6.2 Phosphorus molybdenum blue colorimetry

6.2.1 Principle

After the specimen solution is filtered out of water-insoluble substances such as pyrophyllite, the remaining polyphosphate is hydrolyzed into orthophosphate in inorganic acid, and phosphorus molybdenum blue is formed with ammonium molybdate in the presence of reducing agent. The absorbance A is measured with a spectrophotometer at the wavelength of 650nm. The mass of phosphorus oxide (P_2O_5) of the corresponding absorbance is calculated from the standard curve, and the phosphorus content of the relative sample is calculated.

Note: This method is suitable for the determination of total inorganic phosphorus. When the formulation of detergent product contains organic matter combined with phosphorus element, there may be interference.

6.2.2 Reagents

6.2.2.1 Sulfuric acid: $c(H_2SO_4)$ = 5 mol/L solution.

6.2.2.2 Ammonium molybdate-sulfuric acid solution: Dissolve 7.2g of ammonium molybdate tetrahydrate [$(NH_4)_6MO_7O_{24}$•$4H_2O$] in water; add 400 mL of sulfuric acid solution (6.2.2.1); and dilute to 1,000 mL with water. The concentration of sulfuric acid in this solution is $c(H_2SO_4)$=2 mol/L, and the content of molybdenum trioxide (MoO_3) is about 6g/L.

Note: Ammonium molybdate-sulfuric acid solution will fail when it is prepared for too long, which can be found by comparing the absorbance change of blank test solution in display reaction.

6.2.2.3 Ascorbic acid solution (25 g/L): Dissolve 2.5g of ascorbic acid in 100 mL water. The solution should be reprepared after 2 to 3d.

6.2.2.4 Phosphorus oxide standard solution (1.00 mg/mL): Dry potassium dihydrogen phosphate (KH_2PO_4) in an oven at 110°C for 2h; cool it in a desiccator; weigh 1.917g (accurate to 0.000 5g); dissolve it with water; transfer it into a 1,000 mL volumetric flask; dilute to the scale with water, and mix well.

6.2.2.5 Phosphorus oxide standard solution (10 μg/mL): accurately pipette 10.0 mL of phosphorus oxide standard solution (6.2.2.4) into a 1,000 mL volumetric flask; dilute to the scale with water, and mix well.

6.2.2.6 Hydrochloric acid, about 1 mol/L solution.

6.2.2.7 Petroleum ether.

6.2.3 Apparatus

6.2.3.1 Spectrophotometer, with the wavelength from 350 nm to 800 nm.

6.2.3.2 Beaker, of capacity 150 mL.

6.2.3.3 Volumetric flasks: of capacity 100 mL, 500 mL and 1,000 mL.

6.2.3.4 Pipette, of capacity 10 mL, 15 mL, 20 mL and 25 mL.

6.2.3.5 Graduated pipette: of capacity 10mL.

6.2.3.6 Hard glass test tube: ø25 mm × 200 mm.

6.2.3.7 Medium-speed qualitative filter paper, ø110 mm.

6.2.3.8 Surface dishes.

6.2.3.9 Glass rods.

6.2.4 Procedure

6.2.4.1 Plotting of standard curve

Respectively pipette 0 mL, 2.0 mL, 4.0 mL, 6.0 mL, 8.0 mL, 10.0 mL, 15.0 mL and 20.0 mL of phosphorus oxide standard service solution (6.2.2.5) into hard glass tubes (6.2.3.6); add water to 25 mL; then add 10 mL of ammonium molybdate-sulfuric acid solution (6.2.2.2) and 2 mL of ascorbic acid solution (6.2.2.3) in turn; heat it for 45 min in a boiling water bath; cool down; then transfer it to 100 mL volumetric flasks (6.2.3.3); dilute it with water to the scale; and mix well. Determine the absorbance of the series of solution at the wavelength of 650 nm with a spectrophotometer (6.2.3.1) and a 10 mm or 20 mm cuvette, using water as reference. Plot the standard curve with net absorbance as Y-coordinate and phosphorus oxide (μg) as X-coordinate.

Note: Net absorbance refers to the absorbance of each standard service test solution containing phosphorus oxide minus the absorbance of 0 mL phosphorus oxide standard service test solution, respectively.

6.2.4.2 Specimen treatment

Without soap detergent: weigh 1g of specimen, accurate to 0.001g; put it into a 150 mL beaker (6.2.3.2) and dissolve it with water; then transfer it to a 500 mL volumetric flask (6.2.3.3); dilute it with water to the scale; and shake well for further use.

With soap detergent: weigh 1g of specimen, accurate to 0.001g; put it into a 150 mL beaker (6.2.3.2); add 95% ethanol to 80 mL (for liquid and paste samples, add ethanol absolute to 95% ethanol concentration) to the beaker. After gently stirring with a glass

7.2.4 Potassium chromate, 50 g/L solution.

7.2.5 Phenolphthalein, 10 g/L solution.

7.2.6 Nitric acid, 0.5 mol/L solution.

7.2.7 Sodium hydroxide: 0.5 mol/L solution.

7.2.8 Chloroform.

7.2.9 Calcium nitrate: 10% aqueous solution.

7.3 Apparatus

7.3.1 Suction flask, of capacity 250 mL, 500 mL or 1L.

7.3.2 Gooch's crucible, of capacity 25 mL ~ 30 mL, paved with medium speed filter paper or asbestos filter layer.

When laying filter paper discs, first lay double-layer filter paper discs between the bottom of the crucible and the porous porcelain plate, then lay double-layer filter paper discs on the porous porcelain plate. The diameter of the filter paper discs shall be consistent with the diameter of the bottom of the crucible as far as possible.

When laying the asbestos filter layer, first lay a layer of quick qualitative filter paper disc between the bottom of the crucible and the porous porcelain plate, then fill up the coarse acid washing asbestos slurry that has been soaked in water for 24h and separated by flotation, and filter it dry after sedimentation. In this way, lay two layers of fine acid washing asbestos, and use it after drying in an oven at (105 ± 2)°C.

7.3.3 Boiling water bath.

7.3.4 Oven, which may control the temperature at (105±2)°C.

7.3.5 Beaker, of capacity 150 mL and 300 mL.

7.3.6 Desiccator, contained with allochroic silica gel or other desiccants.

7.3.7 Measuring cylinders, of capacity 25 mL and 100 mL.

7.3.8 Triangular flask, of capacity 250mL.

7.3.9 Glass crucible, with the pore size of 5 μm ~ 15 μm, about 30 mL.

7.3.10 Burette, of capacity 25 mL.

7.3.11 Drop bottle, of capacity 100 mL.

7.3.12 Surface dish.

7.3.13 Glass rod.

7.4 Procedure

7.4.1 Determination of total active substance content by quantitative ethanol solute and sodium chloride content (including water cosolvent) (Method A)

7.4.1.1 Extraction of ethanol solute

Weigh and accurate the specimen (about 2g for powder and granular samples and about 5g for liquid and paste samples) to 0.001g; place it in a 150 mL beaker (7.3.5); add 5 mL of distilled water, and stir continuously with a glass rod to disperse the solid particles and break the clumps until there are no obvious particles (see note for liquid and paste samples). Add 5 mL of ethanol absolute (7.2.2); continue stirring with a glass rod until the sample dissolves into a paste; then slowly add 90 mL of ethanol absolute (7.2.2) while stirring, and continue stirring for a while to promote dissolution. Leave it for a short time until the solution is clear; filter it by dumping through Goochcrucible (7.3.2) or glass crucible (7.3.9) [filter by suction flask (7.3.1)]. Drain the clear liquid as dry as possible; leave the insoluble substance in the beaker as much as possible; then repeat the extraction with 25 mL of 95% hot ethanol (7.2.1) in the same way and filter for four times. Carefully transfer the ethanol extract from the suction flask to a weighed 300 mL beaker (7.3.5); rinse the suction flask for three times with 95% hot ethanol (7.2.1); combine the filtrate and lotion in a 300 mL beaker (i.e. the ethanol extract).

Note: For liquid or paste sample, directly add 100 mL of ethanol absolute (7.2.2); heat; dissolve and left it; filter it by dumping through Goochcrucible or glass crucible (for some samples that are difficult to filter; the samples can be dried in an oven or electric heating plate to be viscous in advance after weighing; then dissolve and extract it with ethanol).

Place a beaker (7.3.5) containing ethyl extract in a boiling water bath; evaporate ethanol to the end; dry the outer wall of the beaker; place it in (105±2)°C. Dry it in oven (7.3.4) for 1h; move into desiccator (7.3.6); cool for 30 min and weigh (m_1).

7.4.1.2 Determination of sodium chloride content in ethanol solute

Dissolve and wash the ethanol extract in a weighed beaker with 100 mL of water and 20 mL of 95% ethanol (7.2.1) respectively into a 250 mL triangular flask (7.3.8); add 20 mL of calcium nitrate solution (7.2.9); then add 3 drops of phenolphthalein solution (7.2.5). If it is red, neutralize it with 0.5 mol/L nitric acid solution (7.2.6) until the red just fades. If it is not red, neutralize it with 0.5 mol/L sodium hydroxide solution (7.2.7) to reddish; then drop it back with 0.5 mol/L nitric acid solution (7.2.6) until the reddish just fades. Then add 1 mL of potassium chromate indicator (7.2.4) and titrate it with 0.1 mol/L silver nitrate standard solution (7.2.3) until the solution changes from yellow to orange.

7.4.1.3 Calculation of results

the separating funnel (8.3.2). Add water to the separating funnel to wash the resin to neutral [determined by pH test paper (8.2.12) with wide range, pH ≈ 7]. During washing, water shall be added to the separating funnel in time to avoid drying up of the resin.

Note: The column specifications are not limited. When the column is large, more resin can be treated at one time, but taking a long time.

8.4.1.2 Processing of anion exchange resins

Soak the anion exchange resin (8.2.2) overnight in 3 times the volume of 0.5 mol/L ethanol hydrochloric acid solution (8.2.6); wash it with water for 3 times by dumping. Then soak in 3 times the volume of 1 mol/L sodium hydroxide solution (8.2.9); stir it for 10 min, and wash with water for 3 times by dumping. Pour the resin into the column, then pour into the ion exchange column (8.3.1); pad the bottom of the column with glass wool; the top is connected with the separating funnel; add 1 mol/L sodium hydroxide solution (8.2.9) to the separating funnel to wash the resin until there are no chloride ions [1 drop of effluent. Add 3 drops of 6 mol/L nitric acid solution (8.2.8); then add 1 drop of 0.1 mol/L silver nitrate solution (8.2.10), which shall not be cloudy compared with the blank]. During washing, water shall be added to the separating funnel in time to avoid drying up of the resin. Then, wash with water to neutral [determined by a pH test paper with wide range (8.2.12), pH ≈ 7].

Note: The column specifications are not limited. When the column is large, more resin can be treated at one time. Different from the processing of cation exchange resin, the process of washing with 1 mol/L sodium hydroxide solution until there is no chloride ion will take more time.

8.4.1.3 Processing of mixed bed resin

Wash the mixed bed resin (8.2.13) consisting of 1:1 strong acidic cation exchange resin and strong caustic anion exchange resin with distilled water to neutral [determined by a pH test paper (8.2.12) with wide range), pH ≈ 7]; wash it with 95% ethanol (8.2.3) with the volume equal to that of resin for 5 times; then soak it in 95% ethanol (8.2.3) for half an hour.

8.4.2 Specimen

Weigh 5g of test sample into a 150 mL beaker (8.3.4) and accurate it to 0.001g.

8.4.3 Determination

Separate ethanol solute according to 7.4.1.1. Pour the ethanol solute into a 500 mL beaker (8.3.4) and concentrate it in a steam bath to about 100 mL to obtain Solution A. The test is continued in either of the following two ways:

a) Keep the resin for recycling and reusing.

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