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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

ICS 71.100.40 CCS Y 43

GB/T 9985-2022

Replacing GB/T 9985-2000

Detergents for Hand Dishwashing

手洗餐具用洗涤剂

Issued on: December 30, 2022 Implemented on: January 1, 2024

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

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Detergents for Hand Dishwashing

1 Scope

This document specifies the requirements, test methods, inspection rules, as well as marking, packaging, transportation and storage of detergents for hand dishwashing.

This document is applicable to detergents for hand dishwashing formulated with surfactants and auxiliaries and detergents for hand dishwashing with the function of fruit and vegetable cleaning.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 1535 Soya Bean Oil

GB/T 6368 Surface Active Agents - Determination of pH of Aqueous Solution - Potentiometric Method

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 8937 Edible Lard

GB/T 13173-2021 Surface Active Agents - Test Methods for Detergents

GB/T 13529 Sodium Ethoxylated Alkyl Sulfate

GB/T 15818 Test Method for Biodegradability of Surfactants

GB/T 21801 Chemicals - Ready Biodegradability - Manometric Respirometry Test

GB/T 21802 Chemicals - Ready Biodegradability - Modified MITI Test (I)

GB/T 21803 Chemicals - Ready Biodegradability - DOC Die-away Test

GB/T 21831 Chemicals - Ready Biodegradability: Closed Bottle Test

GB/T 21856 Chemicals - Ready Biodegradability - CO₂ Evolution Test

GB/T 21857 Chemicals - Ready Biodegradability - Modified OECD Screening Test

Appendix A

(Normative) Determination of Detergency

A.1 Oil Removal Rate Method (arbitration method)

A.1.1 Principle

Evenly apply standard artificial dirt on the glass slide, use a dishwashing detergent solution with a certain concentration to wash it under specified conditions, and determine the percentage of oily dirt removal.

NOTE: the oil removal rate method is applicable to dishwashing detergents of various formulas.

A.1.2 Reagents and materials

The used reagents and materials are as follows:

- a) Hydrochloric acid, $V(HCl): V(H_2O) = 1:6$ solution;
- b) Sodium hydroxide, 50 g/L solution;
- c) Absolute ethanol;
- d) Urea;
- e) Glyceryl monostearate (40%);
- f) Calcium chloride (CaCl₂);
- **NOTE 1:** calcium chloride with a certain hydration number may also be used, but in specific use, it needs to be converted in accordance with the amount of calcium chloride (CaCl₂) stipulated in A.1.2m).
- g) Magnesium chloride (MgCl₂ 6H₂O);
- NOTE 2: anhydrous magnesium chloride or magnesium chloride with a certain hydration number may also be used, but in specific use, it needs to be converted in accordance with the amount of magnesium chloride (MgCl₂ 6H₂O) stipulated in A.1.2m).
- h) Refined butter;
- i) Refined lard: edible lard (GB/T 8937);
- j) Refined vegetable oil: soya bean oil (GB/T 1535);

- k) Sodium alkylbenzene sulfonate;
- 1) Sodium ethoxylated alkyl sulfate ($C_{12} \sim C_{14}$, EO 2 ~ 3, GB/T 13529);
- m) 250 mg/L hard water: weigh-take 16.70 g of calcium chloride and 20.37 g of magnesium chloride to prepare 10 L, about 2,500 mg/L hard water. During use, take 1 L and dilute to 10 L, that is, 250 mg/L hard water.

A.1.3 Instruments, equipment and utensils

The used instruments and equipment are as follows:

- a) Balance: with a division value of 0.2 g;
- b) Balance: with a division value of 0.1 mg;
- c) Electromagnetic heating stirrer;
- d) Vertical-type detergency testing machine and corresponding complete set of equipment;
- e) Thermometer: with a measuring range of 0 °C \sim 100 °C (maximum allowable error \pm 0.2 °C), 0 °C \sim 200 °C (maximum allowable error \pm 2 °C);
- f) Glass slide for microscope: $2 \text{ mm} \times 76 \text{ mm} \times 26 \text{ mm}$;
- g) Tweezers, clips;
- h) Enamel tray: $300 \text{ mm} \times 400 \text{ mm}$;
- i) Tall beaker: 100 mL;
- j) Constant-temperature water bath.

A.1.4 Test

A.1.4.1 Preparation of standard dishwashing detergent

In accordance with the mass ratio, respectively weigh-take 14 portions of sodium alkylbenzene sulfonate (counted by 100%), 1 portion of ethoxylated alkyl sodium sulfate (counted by 100%), 5 portions of absolute ethanol and 5 portions of urea; add water to 100 portions and mix it well; use hydrochloric acid or sodium hydroxide to adjust pH to $7 \sim 8$ and reserve it for later use.

A.1.4.2 Preparation of mixed oil stain

In accordance with the mass ratio, respectively weigh-take 5 portions of butter, 5 portions of lard, 10 portions of vegetable oil and 1 portion of glyceryl monostearate, and place in a beaker. On an electric furnace, heat it to 180 °C to melt, and through electromagnetic stirring, maintain at this temperature for 10 min. After natural cooling, store it in the refrigerator for later use; the

shelf life is 6 months.

A.1.4.3 Preparation of smeared slides

A.1.4.3.1 Environmental conditions

The whole process of smeared slide preparation shall maintain at room temperature of (22 ± 3) °C and a relative humidity of (50 ± 10) % (that is, the selection or adjustment of environmental conditions during the test is based on the principle of obtaining smeared slides that comply with the requirements).

A.1.4.3.2 Preparation of glass slides

Newly purchased glass slides need to be boiled in a detergent solution for 15 min, washed with clean water, until no water droplets hang on them, then, soaked in chromic acid cleansing solution for 1 h, rinsed with clean water and washed with distilled water, then, dried and stored in a drying oven.

Mark the serial No. on each slide and draw a line 10 mm away from the upper edge of the slide to show that that the smear is below this line; draw a line 5 mm from the bottom edge of the slide to show that the excess oil stain below this line shall be wiped off.

Weigh-take the mass of each glass slide (accurate to 1 mg); use the prepared clips to clamp the glass slides of known mass one by one along the line and above, then, hang them on the drying rack and place the drying rack in the enamel tray to prepare for smearing.

A.1.4.3.3 Smearing of glass slides

Take an appropriate amount of the prepared mixed oil stain and place it in a constant-temperature water bath to maintain the oil temperature at (50 ± 2) °C.

Remove the glass slides, together with the clips, from the drying rack one by one; hold the clips and slowly immerse the slides in the oil stain at an oil temperature of (50 ± 2) °C to 10 mm below the upper line, then, slowly take them out, repeat this for $2 \sim 3$ times; after the last smearing, when the dripping of the oil stain slows down, hang the smeared slides back to the original drying rack and successively prepare the smeared slides; after the oil stain on the glass slides becomes solidified, firstly use filter paper or absorbent cotton to wipe off the excess oil stain on the bottom edge and both sides of the lower edge of the smeared slides at 5 mm, then, use tweezers to hold the absorbent cotton dipped in absolute ethanol or petroleum ether to wipe them clean, and let them dry for 3 h under the environmental conditions required by this test; move the air-dried smeared slides to a weighing rack and use an analytical balance to accurately weigh the mass of each smeared glass slide; the amount of smearing for each slide shall be controlled at (0.13 ± 0.02) g, every 4 slides shall be a group, and the amount of smearing for each group of slides shall be controlled at (0.50 ± 0.05) g.

A.1.4.4 Test procedures

 m_2 ---the mass of the smeared slide after washing, expressed in (g).

b) Precision

The relative mean deviation of the two groups of results $\leq 5\%$.

c) Detergency evaluation

Take the arithmetic mean of the ratio of the oil removal rate of the specimen to the oil removal rate of the standard dishwashing detergent as the test result. When the ratio of oil removal rate is greater than 1.05, it shall be determined that the detergency is greater than that of the standard dishwashing detergent; when the ratio of oil removal rate is less than 0.95, it shall be determined that the detergency is less than that of the standard dishwashing detergent; when the ratio of oil removal rate is greater than or equal to 0.95, and less than or equal to 1.05, it shall be determined that the detergency is equal to that of the standard dishwashing detergent.

A.2 Foam Level Method

A.2.1 Principle

Apply a certain amount of artificial dirt on the dishes and wash them in a dishwashing detergent solution of a specified concentration. Since the dirt washed can eliminate the foam of the washing solution, and the number of dishes (i.e. the amount of dirt) that can be cleaned by each detergent solution is related to its detergency, take the surface foam layer disappearing to half as the end point of washing and the number of dishes washed as the evaluation of detergency.

NOTE: the foam level method is not applicable to the determination of the detergency of low-foaming dishwashing detergents.

A.2.2 Reagents and materials

The used reagents and materials are the same as A.1.2, plus the following items:

- a) whole milk powder;
- b) wheat flour;
- c) fresh eggs.

A.2.3 Instruments, equipment and utensils

The used instruments and equipment are as follows.

- a) Desk-type balance: with a division value of 0.1 g.
- White enamel basin: the diameter of the upper opening is 45 cm and the volume is 8
 L.

c) White porcelain dish: the water absorption rate is not more than 1%; under natural light, through visual inspection, the glaze surface shall not manifest obvious color difference, pores, spots or cracks. There are three specifications: large, medium and small. The outer diameter of the large dish is about 250 mm, and the diameter of the smeared part of the bottom of the dish is not less than 160 mm; the outer diameter of the medium dish is about 200 mm, and the diameter of the smeared part of the bottom of the dish is not less than 120 mm; the outer diameter of the small dish is about 160 mm, and the diameter of the smeared part of the bottom of the dish is not less than 100 mm.

d) Bristle paint brush: with a width of 38 mm and 102 mm.

e) Tubulated bottle: 5,000 mL.

f) Beaker: 150 mL.

g) Measuring cylinder: 1,000 mL.

h) Stopwatch.

A.2.4 Preparation of artificial dirt

Formula of artificial dirt (mass ratio): 15% mixed oil, 15% wheat flour, 7.5% whole milk powder, 30% fresh whole egg liquid and 32.5% distilled water.

In accordance with the number of smeared dishes, determine the amount of dirt prepared; in accordance with the above-mentioned formula ratio, weigh each component.

Shell the fresh eggs and put them in a beaker, evenly stir it and reserve for later use; evenly mix the wheat flour and whole milk powder and reserve for later use; put the mixed oil in a beaker and heat to $50\,^{\circ}\text{C} \sim 60\,^{\circ}\text{C}$ to melt, transfer the evenly mixed wheat flour and whole milk powder into the beaker with melted mixed oil and stir. By several times, add water at 35 °C to the beaker and evenly stir it, then, by several times, add the fresh egg liquid to the beaker and stir it, until it manifests fine and smooth artificial dirt, which is ready to be used for smearing (prepare right before use).

A.2.5 Preparation of smeared dishes

Put the prepared dirt and a 38 mm wide bristle paint brush on a desk-type balance to weigh; use the subtraction method to control the amount of dirt and apply it to the dish one by one. The amount of dirt smeared is 4 g on the large dish, 2 g on the medium dish and 0.8 g on the small dish. If the large dish is used as the unit, then, one medium dish is equivalent to 0.5 large dish, and one small dish is equivalent to 0.2 large dish.

During smearing, use the bristle paint brush to dip the artificial dirt and evenly apply it on the concave center surface of the dish. After smearing, place at room temperature 20 °C \sim 25 °C overnight for 12 h \sim 16 h.

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