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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 15818-2018

Replacing GB/T 15818-2006

Test method for the biodegradability of surfactants

表面活性剂生物降解度试验方法

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Table of Contents

1 Scope
2 Normative references
3 Terms and Definitions
4 Principles
5 Reagents
6 Instruments
7 Test procedure
7.1 Preparation of samples
7.2 Preparation of basic nutrient base solution
7.3 Preparation of test solutions
Appendix A
Appendix B
Appendix C

1 Scope

China's national test method for the primary biodegradability of surfactants - how far a surfactant is broken down by the micro-organisms it meets after it goes down the drain. It specifies the test methods for primary biodegradability and applies to anionic surfactants carrying sulfonate and sulfate groups, to polyoxyethylene surfactants with a single chain of 3 to 40 EO units and two-, three- and four-chain types with 6 to 60 EO units in total, to alkyl glycosides, to cationic and amphoteric surfactants, to fatty acid surfactants, to high-foaming surfactants and to surfactants that substantially lower the surface tension of water. It applies equally to the determination of those surfactants in detergents. Surfactants reach surface water in enormous quantity and are the component of a detergent that does the environmental damage if it persists: a surfactant that survives treatment foams in the receiving water, is toxic to fish at low concentration through its action on gill membranes, and accumulates. The test is therefore a condition of sale, and the number it produces has to be reproducible between the manufacturer and the regulator. The method is a die-away test: the surfactant is put into a synthetic sewage inoculated with activated sludge, held under defined aerobic conditions, and the residual surfactant is measured over time by the analytical method appropriate to its class - two-phase titration for the anionic and cationic types, and the specific methods referenced for the others. Primary biodegradation is what is measured: the loss of surface-active character, not complete mineralisation to carbon dioxide. That distinction is the reason the standard has to be read carefully, because a

substance can pass a primary biodegradability test while leaving a persistent fragment behind. Issued on 28 December 2018 and in force since 1 July 2019, it replaces GB/T 15818-2006.

This standard specifies the test methods for the primary biodegradability of surfactants. This standard is applicable to the determination of anionic surfactants with sulfonic acid groups and sulfuric acid groups. The single-chain EO addition number of polyoxyethylene groups is 3~40 and two, three, four chain total EO adduct number 6~60 surfactants, alkyl glycoside surfactants, cationic and zwitterionic table Surfactants, fatty acid surfactants, surfactants with richer foam and surfactants that can effectively reduce the surface tension of water the biodegradability of the agent. This standard also applies to the determination of the degree of biodegradation of the above-mentioned surfactants in detergents.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this article pieces. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 5173 Determination of the content of anionic active substances in surfactant detergents by direct two-phase titration

GB/T 5174 Determination of cationic active content of surfactant detergents by direct two-phase titration

GB/T 6682 Analysis Laboratory Water Specifications and Test Methods

GB/T 13173 Surfactant Detergent Test Method

GB/T 19464 Alkyl glycosides QB/T 2344 Amphoteric surfactant fatty alkyl dimethyl betaine Preparation of test solutions for titration analysis (volumetric analysis) of QB/T 2739 commonly used test methods for washing products

3 Terms and Definitions

The following terms and definitions apply to this document.

3.1 biodegradability Molecular degradation of organic matter that occurs under the complex activities of living organisms.

3.2 The original parent molecular properties disappeared to a certain extent.

3.3 Disappearance time DT-90 disappeartime(DT-90) The time it takes for the biodegradability of the surfactant to reach 90% of the initial concentration.

4 Principles

The activated sludge that has been cultured and acclimated as a surfactant sample is used as a biodegradable source, added to the test sample for shaking culture, and the culture period is determined. The reduction of surfactant during the period was used to obtain the DT-90 of the sample and the degree of biodegradation at the specified time.

5 Reagents

Unless otherwise stated, only the reagents confirmed as analytical grade and the tertiary water specified in GB/T 6682 are used in the analysis.

5.1 Ammonium chloride.

5.2 Dipotassium hydrogen phosphate.

5.3 Potassium dihydrogen phosphate.

5.4 Disodium hydrogen phosphate.

5.5 Magnesium sulfate.

5.6 Potassium chloride.

5.7 Calcium chloride.

5.8 Ferrous sulfate.

5.9 Ferric chloride.

5.10 Yeast extract (biochemical reagent).

5.11 Linear sodium dodecylbenzene sulfonate. the content is more than 95%, and the biodegradability is more than 99%.

5.12 Linear dodecanol polyoxyethylene ether (7EO). the content is more than 98%, and the biodegradability is more than 99%.

5.13 Microbial source. activated sludge is taken from the sewage treatment plant that treats civil wastewater, and the mass concentration of activated sludge suspended matter should be adjusted to 10g/L~20g/L, use within 5h after sampling.

6 Instruments

Common laboratory instruments and the following.

6.1 Shaking culture flask. capacity 1000mL, sterilize in oven at 170°C for 1h~2h. Tighten with a silicone stopper, the silicone stopper is sealed with pressure steam Sterilizer for sterilization.

6.2 Shaking culture machine. the amplitude is more than 20mm, the oscillation frequency is adjustable from 40 times/min to 300 times/min, the constant temperature range is 5°C to

50°C, and the temperature Control accuracy ± 2 °C. The shaking culture machine should be able to record and monitor the temperature and rotation speed during operation in real time, and confirm the temperature of the whole test cycle. The degree and rotation speed meet the requirements of 7.7. RHZJ-I intelligent biodegradable incubator 1) can meet this requirement. 1) RHZJ-I intelligent biodegradable incubator is an example of a suitable commercial product. This information is given for the convenience of users of this standard and is not Indicates approval for this product.

6.3 Pressure steam sterilizer. rated temperature 126°C, rated pressure 0.14MPa.

7.1 Preparation of samples

7.1.1 If the sample exists in the form of detergent or other mixture, the surfactant sample should be prepared according to GB/T 13173.

7.1.2 The test reference substance (5.11, 5.12) is prepared as a neutral 1g/L solution for use.

7.1.3 The separated surfactant sample is made into a neutral 1g/L solution for use.

7.1.4 Samples such as fatty acids are insoluble in water when they are neutral. When preparing the solution, heat and add lye to dissolve them to make a 1g/L solution for later use.

7.2 Preparation of basic nutrient base solution

7.2.1 Composition of the medium solution used for the test Water 1000mL Ammonium chloride 3.0g Dipotassium hydrogen phosphate 1.0g Magnesium sulfate 0.25g Potassium chloride 0.25g Ferrous sulfate 0.002g Yeast extract 0.3g Yeast extract is added just before use. If the nutrient base solution to which yeast extract has been added is stored for more than 8 hours, it should be autoclaved (at 0.11MPa~0.13MPa, 122°C~125°C, sterilization for 20min), the water used in the test should not contain bacteriostatic substances.

7.2.2 Preparation of medium solution without yeast extract (use this solution for alkyl glycosides and sugar ester surfactants) Solution a. Dissolved potassium dihydrogen phosphate 8.5g Dipotassium hydrogen phosphate 21.75g Disodium hydrogen phosphate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$) 67.2g Ammonium chloride 0.5g Make up to 1000mL with water. In order to ensure this buffer solution, it is recommended to measure its pH value, which is around 7.4. If not, it needs to be reconstituted. solution b. Dissolve

22.5 g of magnesium sulfate ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$), and make up to 1000 mL with water. Solution c. Dissolve

36.4 g of calcium chloride ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$) and dilute to 1000 mL with water. Solution d. Dissolve

0.25 g of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and dilute to 1000 mL with water. The solution is