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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

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GB/T 15045-2013

Replacing GB/T 15045-1994

Fatty Alkyl Dimethyl Tertiary Amines

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Table of Contents

Foreword.....	3
1 Scope.....	5
2 Normative references	5
3 Product category	5
4 Technical requirements	6
5 Test methods.....	8
6 Inspection rules	15
7 Marks, packaging, transport, storage and guarantee period	17

Foreword

This Standard is drafted in accordance with the rules specified in GB/T 1.1-2009.

This Standard replaces GB/T 15045-1994 *Fatty Alkyl Dimethyl Tertiary Amines*. Compared with GB/T 15045-1994, this Standard has the following main changes:

- Product categories have been increased from 6 to 12 (see 3.2, 3.2 of edition-1994);
- Product grades have been reduced from 3 to 1 (see Chapter 4 of this Standard; Chapter 4 of edition-1994);
- Color indexes of the qualified products have been altered (see Chapter 4 of this Standard; Chapter 4 of edition-1994);
- The content indexes of tertiary amine, primary and secondary amine, and main constituents have been altered (see Chapter 4 of this Standard; Chapter 4 of edition-1994);
- Free alcohols indexes have been increased (see Chapter 4 of this Standard);
- The tertiary amine value required in the original standard has been changed into the total amine value (see Chapter 4 of this Standard; Chapter 4 of edition-1994);
- The analysis methods of primary and secondary amine have been altered (see 5.4, 5.5 of edition-1994);
- The requirements for hetero-carbon not on the main carbon chain have been deleted;
- The chromatographic column requirements in the test methods of main constituents have been altered (see 5.5, 5.4 of edition-1994).

This Standard was proposed by China Light Industry Council.

This Standard shall be under the jurisdiction of China National Technical Committee for Standardization of Surfactant and Cleaning Products (SAC/TC 272).

Drafting organizations of this Standard: Rhodia Feixiang Fine Chemicals Co., Ltd., Productivity Promotion Center of Surfactants & Detergents and China Research Institute of Daily Chemical Industry, and National Cleaning Products Quality Supervision and Inspection Center (Taiyuan).

Main drafters of this Standard: Ma Lin, Yao Chenzhi, Wang Zhijun.

Previous version replace by this Standard is as follows:

-- GB/T 15045-1994.

Fatty Alkyl Dimethyl Tertiary Amines

1 Scope

This Standard specifies the product category, technical requirements, test methods, inspection rules, marks, packaging, transport, storage and guarantee period of fatty alkyl dimethyl tertiary amines.

This Standard applies to fatty alkyl dimethyl tertiary amines (N,N-dimethyl alkyl tertiary amine) produced by fatty alcohols or fatty acid, which are mainly used to produce cationic surfactants, zwitterionic surfactants, non-ionic surfactants, antistatic agents, etc.

This Standard does not apply to fatty alkyl dimethyl tertiary amines produced by oxo-synthesis alcohols.

2 Normative references

The following documents are indispensable for application of this document. For the dated documents so quoted, only dated versions apply to this document. For the undated documents so quoted, the latest versions (including all modification sheets) apply to this document.

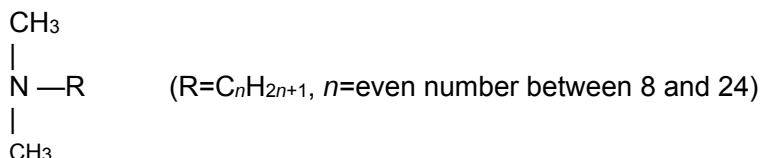
GB/T 3143 Color determination method of liquid chemicals (Hazen unit - Platinum-cobalt scale)

GB/T 6682 Water for laboratory use; Specifications

QB/T 2739 Preparations of standard volumetric solutions of general test methods for washing products

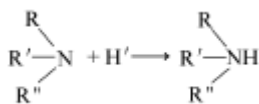
3 Product category

3.1 Structural formula



3.2 Categories and codes

Fatty alkyl dimethyl tertiary amines can be divided into 12 categories according to the fatty alkyl carbon atom numbers and main constituents. Categories and codes are as follows:



Tertiary amine

5.3.2 Reagents

5.3.2.1 HCl

5.3.2.2 Isopropyl alcohol

5.3.2.3 Ethylene glycol

5.3.2.4 Standard solution of HCl- isopropyl alcohol-ethylene glycol, $c = 0.15 \text{ mol/L}$.

Add prepared HCl into the mixed solution of ethylene glycol and isopropyl alcohol (1:1, volume fraction); shake well; then conduct calibration in accordance with the provisions of QB/T 2739. The standard solution shall be sealed and stored in the refrigerator at about 10°C to avoid volatilization of the solvents due to high temperature. Before use, the standard solution shall be placed in room temperature to let it recover to the room temperature; the validity is 1 month.

5.3.2.5 Acetic anhydride

5.3.3 Instruments

5.3.3.1 Titration vessel or beaker, 100 mL.

5.3.3.2 Graduated cylinder, 50mL.

5.3.3.3 Acidometer.

5.3.4 Test procedures

Weigh 0.2 g~0.3 g (less low-molecular-weight sample and more high-molecular-weight sample) of evenly melted and mixed sample (precision: 0.0001g); add into a 100 mL titration vessel or beaker; slowly add in 10 mL of acetic anhydride (if there is any solid in the sample, it shall be heated in hot water till melted); shake well; let the solution stay in room temperature for 15 min; then add in 50 mL of isopropyl alcohol solution; put it on the acidometer; conduct potentiometric titration by using standard solution of HCl- isopropyl alcohol-ethylene glycol (5.3.2.4) until the potential jumps to the maximum value; then write down the volume of standard solution of HCl- isopropyl alcohol-ethylene glycol that has been used in the titration (V_1).

Weigh another sample with an equivalent mass; without adding acetic anhydride, repeat the above test steps; write down the volume of standard solution of HCl- isopropyl alcohol-ethylene glycol that has been used in the titration (V_2).

titration, unit: mL;

c — The molar concentration of standard solution of NaOH-ethyl alcohol, unit: mol/L;

M — The molar mass of fatty alkyl dimethyl tertiary amine, unit: g/mol;

m_3 — The mass of the sample, unit: g.

The arithmetic mean value of two parallel determination results shall be used to express the determination results, correcting to one decimal place.

5.4.6 Degree of precision

The absolute difference of two independent tests' results obtained under the same conditions shall not be more than 0.03%, which is on the premise that the probability with absolute difference more than 0.03% does not exceed 5%.

5.5 Main constituents, free alcohols and average relative molecular mass

5.5.1 Principle

Separate the sample with chromatographic column; calculate the average relative molecular mass, main constituent content and free alcohol content through chromatogram peak area.

5.5.2 Reagents

5.5.2.1 Absolute ethyl alcohol

5.5.2.2 Reference sample: fatty alkyl dimethyl tertiary amine and fatty alcohol with known chain length, taking the sample with known constituents as the reference sample to determine the performance of the chromatographic instrument.

5.5.2.3 Carrier gas, nitrogen, fuel gas, hydrogen, oxidant gas, air.

5.5.3 Instruments

5.5.3.1 Chromatographic instrument, with flame ionization detector and programmed temperature.

5.5.3.2 Chromatographic column: HP-5 can be used for analyzing C_8 - C_{18} , specification: 30 m×0.32 mm×0.5μm or equivalence, Rtx-200 can be used for analyzing $C_{18/22}$ and C_{22} , specification: 30 m×0.53 mm×0.5μm or equivalence.

5.5.3.3 Chromatographic processor or chromatographic work station.

5.5.4 Chromatographic conditions

The typical reference conditions for chromatographic analysis are as follows; necessary

5.5.7 Degree of precision

5.5.7.1 The average relative molecular mass of fatty alkyl dimethyl tertiary amine

The absolute difference of two independent tests' results obtained under the same conditions shall not be more than 1, which is on the premise that the probability with absolute difference more than 1 does not exceed 5%.

5.5.7.2 Free alcohols

The absolute difference of two independent tests' results obtained under the same conditions shall not be more than 0.05%, which is on the premise that the probability with absolute difference more than 0.05% does not exceed 5%.

5.5.7.3 Main constituents

The absolute difference of two independent tests' results obtained under the same conditions shall not be more than 0.7%, which is on the premise that the probability with absolute difference more than 0.7% does not exceed 5%.

6 Inspection rules

6.1 Inspection classification

6.1.1 Delivery inspection

The items that shall take delivery inspection include appearance, tertiary amine content, total amine value, primary and secondary amine and free alcohols as specified in Chapter 4.

6.1.2 Type inspection

The items that shall take type inspection include all the items specified in Chapter 4. Type inspection shall also be conducted under the following conditions:

- a) In formal production, there are great changes in raw materials, process, equipment, management, etc. (including personnel qualities) which may cause influence on products' quality;
- b) In regular production, type inspection shall be conducted regularly;
- c) When resuming production after long-time shutdown;
- d) There are significant differences between the delivery inspection results and the previous type inspection results;
- e) When the type inspection is proposed by national industry administrating

any item of the physicochemical indexes is unqualified, the inspector can get double-size of the collected samples to conduct a re-inspection for the unqualified item. If it is still unqualified after the re-inspection, this batch of products shall be deemed as unqualified.

If both the delivering party and the receiving party can not reach an agreement due to different inspection results, they can apply for an arbitration inspection; the arbitration results shall be deemed as the final basis.

7 Marks, packaging, transport, storage and guarantee period

The marks (pictures and words) printed on the surface of the packages shall be clear and non-discoloring, and shall indicate the following items:

- a) Product name, trademark, standard number adopted and grade;
- b) Production date and guarantee period or product batch number and limited time of use;
- c) Gross weight and net weight;
- d) Waterproof and damp proof marks (words or identifications);
- e) Producer's name, address and contact number.

7.2 Packaging

The products shall be packaged with metal buckets, and the net mass of the package shall comply with the nominal quality.

7.3 Transport

During the transportation, the vessel mouth shall be kept upward to avoid sunshine, rain and damp; the products shall be tenderly loaded and unloaded to prevent packages being damaged.

7.4 Storage

Products shall be stored in dry, clean and ventilated storeroom; when products have to be stored out of doors, necessary damp-proof measures shall be taken; the height of stacks shall ensure not exceeding the maximum load of the supports; the stacks shall have coverings to avoid sun, rain and damp.

7.5 Guarantee period

The guarantee period of the products shall be not less than one year as from the

production date, on the premise that the products comply with the specified transportation conditions and have not been unsealed.

_____ **END** _____

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