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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 83.040.20

G 71

GB/T 8826-2019

Replacing GB/T 8826-2011

Rubber Antioxidant - Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ)

橡胶防老剂 2,2,4-三甲基-1,2-二氢化喹啉聚合物（TMQ）

Issued on: August 30, 2019 **Implemented on: July 1, 2020**

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

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Warning---personnel using this Standard shall have practical experience of formal laboratory work. This Standard does not point out all the possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

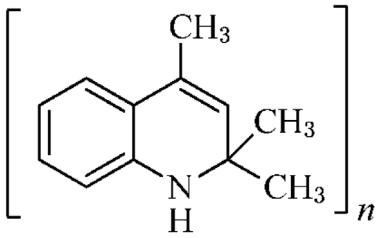
This Standard specifies the classification, technical requirements, test methods, inspection rules, as well as marking, packaging, transportation and storage of rubber antioxidant - polymerized 2,2,4-trimethyl-1,2-dihydroquinoline (hereinafter referred to as rubber antioxidant TMQ).

This Standard is applicable to rubber antioxidant TMQ formed by polycondensation of aniline and acetone in the presence of a catalyst.

Chemical name: 2,2,4-trimethyl-1,2-dihydroquinoline

Molecular formula: $(C_{12}H_{15}N)_n$

Structural formula:



CAS RN: 26780-96-1

2 Normative References

The following documents are indispensable to the application of this document. 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 191-2008 Packaging - Pictorial Marking for Handling of Goods

5 Test Methods

5.1 General Stipulations

Unless it is otherwise specified, only reagents that are confirmed to be analytically pure and Grade-3 water that complies with the stipulations of GB/T 6682-2008 are used in the analysis.

The expression method and rounding-off rules of test data in this Standard shall comply with the relevant stipulations of 4.3.3 on rounding-off value comparison method in GB/T 8170-2008.

5.2 Determination of Appearance

Under natural light, conduct visual inspection.

5.3 Determination of Softening Point

In accordance with the stipulations of 3.3 in GB/T 11409-2008, conduct the determination.

The absolute difference between two parallel determination values shall be not greater than 2 °C. Take the arithmetic mean value as the determination result.

5.4 Determination of Heating Loss

In accordance with the stipulations of 3.4 in GB/T 11409-2008, conduct the determination. The temperature of the electric thermostatic drying oven shall be controlled at 55 °C ± 2 °C; the heating time is 3 h.

The absolute difference between two parallel determination values shall be not greater than 0.04%. Take the arithmetic mean value as the determination result.

5.5 Determination of Ash Content

In accordance with the stipulations of 3.7 in GB/T 11409-2008, conduct the determination. The temperature of the high-temperature furnace shall be controlled at 750 °C ± 25 °C; the sample size is about 2 g, accurate to 0.0001 g; the burning time is 2 h.

5.6 Determination of Ethanol Insoluble Matter Content

5.6.1 Reagent

Absolute ethanol [64-17-5].

5.6.2 Instruments and equipment

5.6.2.1 Sand core crucible: G4.

5.6.2.2 Electric thermostatic blast drying oven.

5.6.2.3 Vacuum pump.

5.6.3 Analytical procedures

Weigh-take about 5 g of finely ground sample, accurate to 0.0001 g; place it in a beaker; add 25 mL of absolute ethanol. At room temperature, slowly stir to thoroughly dissolve the sample. Then, use the sand core crucible, which reaches a constant mass at 70 °C ± 2 °C in advance, to vacuum and filter. Use about 60 mL of absolute ethanol; divide it into three parts to wash the beaker and the sand core crucible. Place the washed sand core crucible (at this moment, there shall be no visible residue on the wall of the sand core crucible) in the electric thermostatic blast drying oven at 70 °C ± 2 °C to dry for 1 h. Take it out; cool it down in a desiccator for 30 min, then, weigh it, accurate to 0.0001 g.

5.6.4 Result calculation

The content of ethanol insoluble matter shall be expressed in mass fraction (w_1) and calculated in accordance with Formula (1):

$$w_1 = \frac{m_1 - m_2}{m_3} \times 100\% \qquad \cdots\cdots(1)$$

Where,

m_1---the numerical value of the sand core crucible and the ethanol insoluble matter, expressed in (g);

m_2---the numerical value of the mass of the sand core crucible, expressed in (g);

m_3---the numerical value of the mass of the sample, expressed in (g).

5.6.5 Permissible error

The absolute difference between two parallel determination values shall be not greater than 0.04%. Take the arithmetic mean value as the determination result.

5.7 Determination of Isopropyl Diphenylamine Content and Total Content of Dimer, Trimer and Tetramer

5.7.1 Reagents

5.7.1.1 Acetonitrile [75-05-8]: chromatographically pure.

5.7.1.2 Tetrabutylammonium bromide [1643-19-2]: with a mass fraction greater than

fraction (w_3), expressed in (%) and calculated in accordance with Formula (4):

$$w_3 = w_{dimer} + w_{trimer} + w_{tetramer} \quad \cdots\cdots (4)$$

Where,

w_{dimer}---the numerical value of dimer content in the rubber antioxidant (TMQ), expressed in (%);

w_{trimer}---the numerical value of trimer content in the rubber antioxidant (TMQ), expressed in (%);

$w_{tetramer}$---the numerical value of tetramer content in the rubber antioxidant (TMQ), expressed in (%).

5.7.7 Permissible error

The absolute difference between two parallel determination values of isopropyl diphenylamine content shall be not greater than 0.10%. Take the arithmetic mean value as the determination result.

The absolute difference between two parallel determination values of dimer, trimer and tetramer content shall be not greater than 1.0%. Take the arithmetic mean value as the determination result.

5.7.8 Typical chromatogram

5.7.8.1 Typical chromatogram of ordinary type sample analysis of rubber antioxidant TMQ

The typical chromatogram of ordinary type sample analysis of rubber antioxidant TMQ is shown in Figure 1.

5.8.2.5 Hydrochloric acid [7647-01-0].

5.8.2.6 Hydrochloric acid solution (2 mol/L): measure-take 167 mL of hydrochloric acid; use water to dilute to 1,000 mL, mix it well.

5.8.2.7 DMAB solution (10 g/L): weigh-take 5 g of DMAB, accurate to 0.0001 g. Use 250 mL of hydrochloric acid solution to dissolve it, then, transfer it to a 500 mL brown volumetric flask. Use methanol to dilute to the scale.

5.8.2.8 Aniline standard stock solution: weigh-take 0.1 g of aniline, accurate to 0.0001 g; place it in a 100 mL volumetric flask. Add 30 mL of hydrochloric acid solution to dissolve it, then, use methanol to dilute to the scale.

5.8.2.9 Filter paper.

5.8.3 Instruments and equipment

5.8.3.1 UV-visible spectrophotometer; wavelength range: 200 nm ~ 900 nm, absorbance range: maximum 2.0. The instrument shall comply with the stipulations of GB/T 26813.

5.8.3.2 Cuvette: 10 mm.

5.8.3.3 Separating funnel: 250 mL, equipped with a Teflon stopper.

5.8.3.4 Brown volumetric flask: 100 mL and 500 mL.

5.8.4 Preparation of aniline standard colorimetric solution

In accordance with the stipulations in Table 4, accurately transfer-take the aniline standard stock solution and place it in a 100 mL volumetric flask. In addition, in accordance with the stipulations, accurately add corresponding hydrochloric acid and DMAB solution. Then, add methanol to approach the scale; gently shake the volumetric flask. After the solution cools down to room temperature, use methanol to dilute to the scale. Let it stand for 30 min. This solution shall be used as aniline standard colorimetric solution.

exit-factory inspection items of the high-content type of the rubber antioxidant TMQ.

6.2 Batch Rules

Uniform products produced in the same production cycle shall constitute one batch.

6.3 Sampling

Sampling shall be carried out in batches and in accordance with the stipulations of GB/T 6679. The total sampling size shall be not less than 500 g, which shall be respectively packed in two clean and dry containers, and labeled, indicating: manufacturer name, product name, model, batch No., sampling date and sampler, etc. One shall be used for the exit-factory inspection, and the other one shall be reserved for the subsequent re-inspection.

6.4 Determination of Qualification

When all the exit-factory inspection results of the ordinary type of the rubber antioxidant TMQ comply with the requirements of Item (1) ~ Item (5) in Table 1, then, this batch of products shall be determined as qualified. When one of the indicators in the inspection results does not comply with the requirements of Item (1) ~ Item (5) in Table 1, then, samples shall be taken from twice the size of the same batch of products for a re-inspection of all the items. Even if only one indicator fails to comply with the requirements of Item (1) ~ Item (5) in Table 1 in the re-inspection results, this batch of products shall also be determined as disqualified.

When all the exit-factory inspection results of the high-content type of the rubber antioxidant TMQ comply with the requirements of Table 1, then, this batch of products shall be determined as qualified. When one of the indicators in the inspection results of the batch of products does not comply with the requirements of Table 1, then, samples shall be taken from twice the size of the same batch of products for a re-inspection of all the items. Even if only one indicator fails to comply with the requirements of Table 1 in the re-inspection results, this batch of products shall also be determined as disqualified.

7 Marking, Packaging, Transportation and Storage

7.1 Marking

On the outer package of the product, there shall be clear and firm marking, which includes: manufacturer name, address, product name, model, batch No., net content, production date and serial No. of this Standard. In addition, in accordance with the stipulations of GB/T 191-2008, indicate “HAND HOOK PROHIBITED” and “KEEP AWAY FROM RAIN”.

7.2 Packaging

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