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Vulcanizing accelerator TBBS

硫化促进剂 TBBS

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Vulcanizing accelerator TBBS

1 Scope

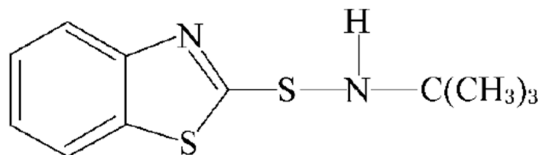
This Standard specifies the requirements, test methods, inspection rules, marks, packaging, transportation and storage for N-tert-butyl-2-benzothiazole sulfenamide (vulcanizing accelerator TBBS).

This Standard is applicable to vulcanizing accelerator TBBS made by oxidation of tert-butylamine and vulcanizing accelerator MBT.

Chemical name: N-tert-butyl-2-benzothiazole sulfenamide

Molecular formula: $C_{11}H_{14}N_2S_2$

Structural formula:



Relative molecular mass: 238.37 (according to the international relative atomic mass in 2005)

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 191, *Packaging and storage marks* (GB/T 191-2008, ISO 780:1997, MOD)

GB/T 601, *Chemical reagent -- Preparations of reference titration solutions*

GB/T 603, *Chemical reagent -- Preparations of reagent solutions for use in test methods* (GB/T 603-2002, ISO 6353-1:1982, NEQ)

The judgment of the test results shall be carried out according to the rounded value comparison method in GB/T 1250.

4.1 Appearance

Use visual inspection method to judge.

4.2 Determination of initial melting point

Carry out according to the provisions of GB/T 11409.1. The inner diameter of the capillary containing the specimen is 1.2 mm ~ 1.4 mm. The thickness of the glass is 0.2 mm ~ 0.3 mm. Fill the sample 3mm ~ 6mm. When the temperature of the heat transfer fluid is 25°C lower than the predicted melting point, heat up at a rate of 3°C/min. When it rises to 10°C lower than the predicted melting point, attach the capillary containing the sample to the thermometer. Make the center of the sample at the same height as the middle of the mercury bulb of the thermometer. Insert into thermal fluid. Heat up at (1±0.2)°C/min.

4.3 Determination of heating loss

Carry out according to the provisions of GB/T 11409.4. The sampling weight is about 2 g (accurate to 0.1 mg). The temperature of the electric constant temperature drying oven is controlled at (70±2)°C. The heating time is 3 h.

4.4 Determination of ash

Carry out according to the provisions of GB/T 11409.7. The sampling weight is about 3 g (accurate to 0.1 mg). The temperature of the high temperature furnace is controlled at (750±25)°C. Burning time is 2 h.

4.5 Determination of sieve residue

Determine according to the drying method in Chapter 2 of GB/T 11409.5-1989. Test sieve (GB/T 6003.1): ϕ 200 mm $\times$ 50 mm / 150 μ m.

4.6 Determination of methanol insoluble

4.6.1 Reagents

Methanol [67-56-1].

4.6.2 Instruments

4.6.2.1 Watch glass: 150 mm in diameter.

4.6.2.2 Beaker: 500 mL.

4.6.2.3 Sand core crucible: G₄.

4.6.2.4 Magnetic stirrer.

4.6.2.5 Oven: capable of maintaining the temperature at $(70 \pm 2)^{\circ}\text{C}$.

4.6.2.6 Suction filter bottle: capacity is 500 mL.

4.6.2.7 Wash bottle: capacity is 500 mL.

4.6.3 Analysis steps

Weigh about 2.5 g (accurate to 0.1 mg) of the finely ground specimen and place it in a beaker. Use a watch glass to cover the mouth of the beaker. Add 125 mL of methanol. Stir at $(25 \pm 5)^{\circ}\text{C}$ for 30 min on a magnetic stirrer. After dissolving, use a sand core crucible with a constant temperature of $(70 \pm 2)^{\circ}\text{C}$ to vacuum filter. Use about 25 mL of methanol to wash the beaker three times. After the filtration, use about 25 mL of methanol to wash the sand core crucible twice. After maintaining 2 min, immediately conduct vacuum suction. At this point, the sand core crucible shall have no visible residue. Place the sand core crucible in an oven at $(70 \pm 2)^{\circ}\text{C}$ for 1 h to dry. Take it out. Cool to room temperature in a desiccator. Weigh (accurate to 0.1 mg).

4.6.4 Calculation of results

Methanol insoluble matter is calculated as mass fraction w_1 . The value is expressed in %. It is calculated according to formula (1):

$$w_1 = \frac{m_2 - m_1}{m_0} \times 100 \quad \dots\dots\dots (1)$$

Where,

m_2 - the mass value of the empty sand core crucible and methanol insoluble matter, in grams (g);

m_1 - the value of the mass of the empty sand core crucible, in grams (g);

m_0 - the numerical value of the mass of the sample, in grams (g).

4.7 Determination of free amine content

4.7.1 Reagents

4.7.1.1 Absolute ethanol [64-17-5].

4.7.1.2 Hydrochloric acid standard titration solution: $c(\text{HCl}) = 0.1 \text{ mol/L}$.

4.7.1.3 Sodium hydroxide standard titration solution: $c(\text{NaOH}) = 0.1 \text{ mol/L}$.

4.7.1.4 Bromophenol blue indicator solution (10 g/L): Dissolve 1 g of bromophenol

ratio of dichloromethane to methanol is 60:40). Use acetonitrile + water as mobile phase. A stainless-steel column with C18 filler and a UV detector (280 nm) are used. TBBS in the specimen is separated and determined by reversed-phase high-performance liquid chromatography.

4.8.2 Reagents and solutions

4.8.2.1 Water: grade two water or ultrapure water filtered through a 0.45 μm pore size filter membrane.

4.8.2.2 Acetonitrile [25-05-8]: chromatographically pure.

4.8.2.3 Glacial acetic acid [64-19-7]: chromatographically pure.

4.8.2.4 Dichloromethane [75-09-2]: chromatographically pure.

4.8.2.5 Methanol [67-56-1]: chromatographically pure.

4.8.3 Instruments

4.8.3.1 High performance liquid chromatography.

4.8.3.2 Recorder: chromatographic data processor or chromatographic workstation.

4.8.3.3 Detector: UV detector.

4.8.3.4 Chromatographic column: C18 (ODS) 4.6 mm $\times$ 150 mm, 5 μm (particle size); can provide 40,000 theoretical plates per meter (at least 10,000 plates are required for analysis).

4.8.3.5 Microinjector: 50 μL or autosampler.

4.8.3.6 Ultrasonic oscillator.

4.8.4 Operating conditions of HPLC

4.8.4.1 Mobile phase: a mixture of acetonitrile and water (the volume ratio of acetonitrile and water is 75:25), filtered through a filter membrane and degassed.

4.8.4.2 Flow rate: 2.0 mL/min.

4.8.4.3 Column temperature: 26°C (the temperature difference shall not exceed 2°C).

4.8.4.4 Detection wavelength: 280 nm.

4.8.4.5 Injection volume: 20 μL .

4.8.4.6 Retention time: about 4.0 min.

The above operating parameters are typical. According to the characteristics of different

This product shall be inspected by the quality supervision and inspection department of the manufacturer according to this Standard before leaving the factory. It is attached with a quality certificate in a certain format, which includes: product name, standard number, manufacturer name, registered trademark, batch number, etc.

5.3 Batching rules

Take a homogeneous product of equal mass as a batch.

5.4 Sampling

Sampling is conducted according to GB/T 6679. The sampling volume shall not be less than 600 g. Divide into two clean, dry ground-mouth bottles (plastic bags). Seal and affix a label, indicating the name of the manufacturer, product name, batch number, sampling date and the name of the sampling person. One bottle (bag) shall be inspected by the inspection department, and the other bottle (bag) shall be kept sealed for 3 months for future reference.

5.5 Reinspection

If one of the indicators in the inspection results of the exit-factory items does not meet the requirements of this Standard, it shall be reinspected by sampling from twice the amount of packaging. If there is still one indicator in the reinspection results that does not meet the requirements of this Standard, then this batch of products shall be rejected.

6 Marks, packaging, transportation and storage

6.1 Marks

6.1.1 There shall be a clear and firm mark on each outer package. Its content includes: product name, standard number, manufacturer name, address, contact number, registered trademark, net content, production date, and batch number.

6.1.2 Each packaged product shall be accompanied by a product exit-factory certificate. Its content includes: product name, standard number, batch number, net content, etc.

6.2 Packaging

6.2.1 This product is packed in wood fiber barrels or woven bags lined with plastic film bags. The net weight of each bag (or barrel) is 25 kg or 20 kg.

6.2.2 If other packaging methods are required, it shall be implemented according to the contract.

6.3 Transpiration

The product transportation shall comply with GB/T 191. Protect from sunlight and

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