

Translated English of Chinese Standard: GB/T24153-2009

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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 83.060
G 40

GB/T 24153-2009

Rubber and Elastomer Materials
– Determination of N-nitrosamines

橡胶及弹性体材料 N-亚硝基胺的测定

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Issued on: June 15, 2009

Implemented on: February 1, 2010

**Issued by: General Administration of Quality Supervision, Inspection and Quarantine;
Standardization Administration of PRC.**

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Foreword

This Standard's Appendix A is normative; while Appendix B is informative.

This Standard was proposed by China Petroleum and Chemical Industry Association.

This Standard shall be under the jurisdiction of National Technical Committee for Standardization of Rubber and Rubber Products (SAC/TC 35).

Drafting organizations of this Standard: Fujian Putian Entry-Exit Inspection and Quarantine Bureau, Shenyang Rubber Research and Design Institute, and Fujian Quanzhou Entry-Exit Inspection and Quarantine Bureau.

Chief drafting staffs of this Standard: Tong Yugui, Cheng Qun, Lin Bifen, Li Yijun, Liu Huichun, and Lin Zhong.

Rubber and Elastomer Materials

– Determination of N-nitrosamines

Warning: personnel using this Standard shall have practical working experience in the formal laboratory. This Standard doesn't indicate all possible laboratory work. The users shall take responsibility to take appropriate safety and health measures; and ensure compliance with the conditions set forth in the relevant national regulations.

1 Scope

This Standard specifies adopting gas chromatography – mass selective detector (GC-MSD) to determine 12 types of N-nitrosamines (see Appendix A) contents in the rubber and elastomer.

This Standard is applicable to the rubber, elastomer materials and their products.

2 Principle

The sample was extracted by methanol; after concentrating, the extracting solution passes through C₁₈ solid-phase extraction column to purify; the sample solution enters into the gas chromatography – mass selective detector (GC-MSD) to be measured; qualified by full-scanning detection; select ions to quantify by external standard method.

3 Reagents and Materials

Unless otherwise is specified, all the reagents used are chromatographic pure.

3.1 Methanol, the blank test check there are no interfering substances.

3.2 C₁₈ solid-phase extraction column: 500mg/3mL.

3.3 Standard stock solution: separately and accurately weigh the appropriate amount of each N-nitrosamines standard products; use methanol to prepare the standard stock solution with concentration of 200mg/L; inject into the brown reagent bottle; store in the refrigerator at 5°C below; and valid for 3 months.

NOTE: N-nitrosamines substances are easy to be decomposed by the ultraviolet rays; the extracting solution and standard solution shall avoid exposing to the sunshine or fluorescent lamps or other light sources. The sample or standard solution shall be wrapped by aluminum foil or stored in the dark at 5°C below.

3.4 Standard working solution: take appropriate volume of standard stock solution (3.3) as required to mix; then use methanol to dilute to the standard working solution with suitable concentration; inject into the brown reagent bottle; it shall be prepared for current use.

4 Apparatus

- 4.1 Gas chromatograph: equipped with mass selective detector (MSD).
- 4.2 Ultrasonic generator: operating frequency of 40kHz.
- 4.3 Rotary evaporator: equipped with vacuum gauge (can show the vacuum to 10kPa).
- 4.4 Solid-phase extraction device: equipped with vacuum pump.
- 4.5 Vortex mixer.
- 4.6 Conical flask: brown, with ground stopper, 100mL.
- 4.7 Concentrated bottle: brown, 150mL.
- 4.8 Centrifuge tube: brown, 10mL.

5 Analytical Procedures

5.1 Specimen preparation and extraction

Take the representative sample, use appropriate tool to cut it into particles with edges of 3mm below, mix evenly. Take 5.0g (accurate to 0.01g) of specimen from the above mixed sample; place into conical flask (4.6); add 30mL of methanol (3.1); perform the ultrasonic extraction in the ultrasonic generator (4.2) for 30min. Transfer the extracting solution into the concentrated bottle (4.7); add 20mL of methanol into the conical flask; extract once again; combine the extracting solutions. Place the concentrated bottle onto the rotary evaporator (4.3); control the vacuum degree to be 21.3kPa~16.3kPa; slowly concentrate in the 35°C water bath, so that the volume is slightly less than 4.5mL. Take off the concentrated bottle; rotate and shake fully onto the vortex mixer (4.5) for 1min (so that the substances attached on the bottle wall can dissolve into the extracting solution); the transfer the sample solution in the concentrated bottle into 5mL brown volumetric flask. Take another 0.5mL of methanol to leach the concentrated bottle wall; after rotating and shaking fully on the vortex mixer for 1min, transfer into the volumetric flasks to combine; use methanol to make constant volume of 5mL; shake evenly.

5.2 Purification

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