ICS 83.060
CCS G 40

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 46232-2025

Rubber, unvulcanized - Determination of bound-rubber content

未硫化橡胶 结合橡胶含量的测定

Issued on: August 29, 2025 **Implemented on: March 1, 2026**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

Table of Contents

Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents". Drafting.

Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents.

This document was proposed by the China Petroleum and Chemical Industry Federation.

This document is under the jurisdiction of the National Technical Committee on Standardization of Rubber and Rubber Products (SAC/TC35).

This document was drafted by: Shandong Linglong Tire Co., Ltd., Qingdao Double Star Tire Industry Co., Ltd., and Shandong Yanggu Huatai Chemical Co., Ltd.

Co., Ltd., Anhui Giti Passenger Radial Tire Co., Ltd. R&D Branch, Guangzhou Synthetic Materials Research Institute Co., Ltd., Guizhou Tire Joint-stock limited companies, Weihai Junle Tire Co., Ltd., Bayi Rubber Co., Ltd., Suzhou Newmai Analytical Instruments Co., Ltd., Double Coin The group includes Shanghai Tire Research Institute Co., Ltd., Zhuzhou Times New Material Technology Co., Ltd., and Beijing Rubber Industry Research and Design Institute Co., Ltd.

Company. Beijing Rubber & Tire Testing Technology Service Co., Ltd.

The main drafters of this document are: Zhang Lijie, Lu Yuan, Guo Fei, Chu Wendong, Shi Lilong, Fang Xuan, Wu Haibin, Li Bingyu, Xie Yufang, and Ji Jingqi.

Su Juqiao, Song Chengyang, Lun Xin, Ding Wuhui, Song Erhua, Yao Ming, Ding Hao, Chen Luping, Dong Wenwu, Zhang Lingyan, Yang Liu, Ding Xingwu, Ding Xiaoying Wang Na, Lü Jiaping, Yue Min.

Determination of the content of uncured rubber and bonded rubber Warning 1.Personnel using this document should have practical experience working in a formal laboratory. This document does not cover all possible safety hazards.

Problem. Users are responsible for taking appropriate safety and health measures and ensuring compliance with relevant national regulations.

Warning 2---Using the procedures outlined in this document may involve the use or generation of certain substances, potentially resulting in waste. This has It may cause harm to the local environment. After use, it should be handled and disposed of safely in accordance with the relevant documents.

1 Scope

This document describes a method for determining the bound rubber content in uncured rubber using low-field NMR and chemical methods.

This document applies to the determination of the bound rubber content in uncured rubber.

Note. Because these two methods are based on different testing principles, the test results are not comparable.

2 Normative references

This document has no normative references.

3 Terms and Definitions

The following terms and definitions apply to this document.

3.1 Combined with rubber-bound rubber

In rubber compounding, the portion of rubber that is tightly bonded to the filler.

Note. Chemical methods measure the portion of rubber that cannot be dissolved by common solvents; low-field NMR methods measure adsorption through physical and chemical bonding. That part of the rubber.

[Source. GB/T 9881-2008, 2.56, with modifications]

4.1 Low-field nuclear magnetic resonance (NMR)

Uncured rubber containing bound rubber comprises two components. a bound rubber component and an unbound rubber component (containing molecular chain physical entanglement,...

(Two parts of the free chain). The movement of rubber molecular chain segments in the bound rubber component is more restricted, resulting in faster transverse relaxation decay; the unbound rubber component...

These molecules exhibit certain molecular motion characteristics, and their transverse relaxation decay is relatively slow. The low-field nuclear magnetic resonance analyzer uses Hahn Echo sequences for acquisition.

The percentage of the combined rubber signal in the total signal indicates the content of the combined rubber. The schematic diagram of the test principle is shown in Figure 1.

Note. The Hahn Echo sequence is based on a 90°-tau-180°-tau radio frequency pulse sequence. The spin phase is flipped by a 180° pulse, correcting the phase distortion caused by magnetic field inhomogeneity.

The dispersed echoes reconverge at echo time (2tau) to form a spin echo. Indexing symbol explanation.

X --- Lateral relaxation time;

Y --- Nuclear magnetic resonance signal intensity;

A< sub>total< /sub> --- Total signal intensity of uncured rubber;

A. Signal intensity of non-bound rubber components;

A1---Binding rubber signal strength;

A2 --- Signal intensity of physical entanglement of rubber molecular chains;

A3 --- Signal intensity of free chain in rubber molecules;

t1 --- Start time of non-bonded rubber signal.

Figure 1.Schematic diagram of the low-field NMR testing principle.

4.2 Chemical Method

Uncured rubber filled with carbon black and/or silica is immersed in a solvent (usually toluene), because the rubber molecules react with the carbon black and/or silica...

The physical adsorption and chemical bonding forces on the surface of silica prevent the bound rubber in uncured rubber from being dissolved by solvents, while the unbound rubber...

It can be dissolved. The content of the bound rubber is calculated by the change in mass before and after soaking.

5.1 Reagents

Standard viscosity fluid. GB W13609, National Standard Material (NCRM).

5.2 Instruments and Equipment

5.2.1 Low-field nuclear magnetic resonance analyzers shall meet the following technical requirements.

- Magnetic field strength. 0.48T~0.52T;

- Effective spacing between magnets. not less than 35mm;

- Hydrogen probe coil inner diameter. not less than 12mm;

- Overall dead time. less than 15μs;

- Nuclear magnetic resonance analysis application software. includes pulse sequences such as FID and HahnEcho;

- Data processing software. includes Gaussian fitting algorithm;

- Magnetic field uniformity. The relaxation time of the FID signal at 50% of its original value is not less than 1ms.

5.2.2 Connector. Diameter not greater than 12mm, length not less than 180mm, no hydrogen signal.

5.2.3 Sample vial. 11.6 mm in diameter, 32 mm in height, no hydrogen signal.

5.2.4 Dry thermostat. Temperature control range is room temperature to 160°C, accuracy is 0.1°C.

5.3 Sample Preparation

Cut an appropriate amount of sample into two cylindrical specimens with a diameter of