

ICS 83.060

CCS G 40



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 46325-2025

Vulcanized rubber - Determination of free sulfur content - Gas chromatography (GC) method and high performance liquid chromatography(HPLC)method

硫化橡胶 游离硫含量的测定 气相色谱法 (GC) 和高效液相色谱法 (HPLC)
(English Translation)

Issued on: October 5, 2025

Implemented on: May 1, 2026

**Issued by: State Administration for Market Regulation and
Standardization Administration of China**

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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.

This document is modified to adopt ISO 2016 3.2019 "Gas chromatography (GC) and high performance liquid chromatography (HPLC) for the determination of free free corrosive organic compounds in vulcanized rubber". Sulfur content.

This document has undergone significant structural changes compared to ISO 2016 3.2019. A table comparing the structural numbering changes between the two documents is provided. See Appendix A.

The technical differences between this document and ISO 2016 3.2019, and the reasons therefor, are as follows.

---Added Method C (Liquid-phase non-derivative method) and its corresponding testing principle, instruments and reagents used, testing procedures, and result description.

The method for preparing standard solutions is described above because it is simpler to operate and has lower toxicity than methods A and B (see Chapters 4-10, Appendix). Record D);

---The purity type of ammonium acetate in section 6.10 has been changed from "analytical grade" to "chromatographic grade" to obtain better test results;

---The sample preparation steps for Methods A and B in Chapter 8 have been modified, adding requirements for sample particle size and recommending extraction solvents.

The extraction time was adjusted, the amount of diluent and the volumetric flask volume were changed, and a blank test was added to facilitate operation and test results. precise;

---The split ratio for gas chromatography-MS in section 9.1 has been changed from "42" to "10.1" to facilitate the testing of target compounds;

---The calculation formulas for Method A and Method B in section 10.2 have been modified

to include the blank test concentration in the formulas.

The following editorial changes have also been made to this document.

---The units of measurement for free sulfur content in Method A and Method B have been modified (see Chapters 10 and 12).

---The errors in the international standard formula have been improved and corrected.

1 Scope

This document describes a method for determining the content of unconsumed inorganic free sulfur (S_x) during rubber vulcanization using chromatography. This method primarily... Used to compare the free sulfur content in rubbers with different degrees of vulcanization.

Note 1: In this document, "inorganic free sulfur" is replaced with "free sulfur".

Note 2: The free sulfur content determined by this method does not include sulfur in the vulcanizing agent.

2 Normative references

The contents of the following documents, through normative references within the text, constitute essential provisions of this document. Dated citations are not included.

For references to documents, only the version corresponding to that date applies to this document; for undated references, the latest version (including all amendments) applies. This document.

GB/T 3516-2024 Determination of solvent extractables in rubber (ISO 1407:2023, IDT)

3 Terms and Definitions

This document does not contain any terms or definitions that need to be defined.

4 Principles

Free sulfur can be quantitatively analyzed using the following three methods. gas chromatography-mass spectrometry (GC/MS) or gas chromatography-flame ionization.

Chemical detection method (GC/FID) (Method A), high performance liquid chromatography derivatization method (HPLC/UV) (Method B), and high performance liquid chromatography non-derivatization method Method (HPLC/UV) (Method C).

For vulcanized rubber samples from methods A and B, extraction was first performed according to method B in GB/T 3516-2024. Pre-extracted solvent was then added to the resulting solution.

Known amounts of triphenylphosphine (TPP). TPP reacts with free sulfur to form the stable compound triphenylphosphine sulfide (TPPS), derivatization reaction equation. The formula is shown in Figure 1. TPPS compounds contain aromatic rings and have the following advantages.

First, it has more active chromophores than free sulfur, and is therefore easier to detect in HPLC/UV;

---Secondly, it has sufficient thermal stability to be injected into the GC.

The following quantitative methods are applicable to both GC and HPLC chromatography. The sample used in GC (Method A) and HPLC derivatization methods (Method B) is... The preparation methods are the same.

---When the free sulfur content in the sample is unknown, the standard addition method is preferred, as this method can correct for matrix effects.