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

GB/T 29614-2021

**Vulcanized rubber - Determination the content of polycyclic
aromatic hydrocarbons**

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Foreword

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

This document replaces GB/T 29614-2013 "Determination of Polycyclic Aromatic Hydrocarbons in Vulcanized Rubber", compared with GB/T 29614-2013,

In addition to structural adjustments and editorial changes, the main technical changes are as follows.

- Changed the reagents for sample preparation in Method A (see 4.2.2.1, 4.2.3.1 of the.2013 edition);
- Added optional procedures for the use of acetaldehyde (see 4.4.3.2.9);
- Changed the number of PAHs tested in Method B (see Chapter 5, Chapter 5 of the.2013 edition);
- Changed the extraction reagents in Method B (see 5.2.1, 5.2.1 in the.2013 edition);
- Change the sample weight of method B (see 5.5.1, 5.5.1 of the.2013 edition);
- Changed the precision data in Appendix D (see Table D.1, Table 1 in the.2013 edition).

This document uses the redrafting method to modify and adopt ISO 21461.2012 "Determination of Aromatic Oil in Rubber Vulcanizates".

Compared with ISO 21461.2012, this document has many structural adjustments. Appendix A lists the differences between this document and ISO 21461.2012.

A list of chapter number comparisons.

The technical differences between this document and ISO 21461.2012 and the reasons are

as follows.

---Regarding normative reference documents, this document has made adjustments with technical differences to adapt to China's technical conditions and adjustments

It is collectively reflected in Chapter 2 "Normative Citation Documents", and the specific adjustments are as follows.

* Replace ISO 1407 (see 4.3.2) with GB/T 3516 modified to adopt international standards;

---Changed 4.4.3 "NMR analysis", added 2 test steps that are missing in the international standard;

---Chapter 5 "Method B. Gas Chromatography-Mass Spectrometry" was added, because the determination of polycyclic aromatic hydrocarbons content by gas chromatography-mass spectrometry has been

widely used;

---Change the precision data in Appendix D to the comparison data of domestic laboratories, and add the precision data and configuration of 2 samples at the same time.

To make the data more comprehensive;

---According to the requirements of Method B, an informative appendix E "Retention time, molecular formula,

Relative molecular mass, qualitative ion and quantitative selective ion";

---According to the requirements of Method B, an informative Appendix F "Typical Gas Chromatography-Mass Spectrometry of 18 Polycyclic Aromatic Hydrocarbons and 3 Internal Standards has been added

Chromatogram".

The following editorial changes have been made to this document.

--- In order to coordinate with the existing standards, the standard name was changed to "Determination of Polycyclic Aromatic Hydrocarbon Content in Vulcanized Rubber".

1 Scope

This document describes two methods for determining the PAH content in vulcanized rubber. Method A is hydrogen nuclear magnetic resonance spectroscopy, method B is gas Gas chromatography-mass spectrometry.

This document applies to the determination of polycyclic aromatic hydrocarbons content in vulcanized rubber, as well as to the determination of polycyclic aromatic hydrocarbons content in tires.

2 Normative references

The contents of the following documents constitute the indispensable clauses of this document through normative references in the text. Among them, dated quotations

Only the version corresponding to that date is applicable to this document; for undated reference documents, the latest version (including all amendments) is applicable to

This document.

GB/T 3516 Determination of rubber solvent extract (GB/T 3516-2006, ISO 1407.1992, MOD)

3 Terms and definitions

The following terms and definitions apply to this document.

3.1

Polycyclic aromatic hydrocarbon; PAH

A hydrocarbon containing two or more benzene rings in the molecule.

Note. According to the connection mode of the benzene ring, polycyclic aromatic hydrocarbons can be divided into three types. polyphenylene substituted aliphatic hydrocarbons, biphenyl and condensed polycyclic aromatic hydrocarbons.

4.1 Principle

In the nonlinear PAH molecular structure composed of three or more rings, there is a typical three-sided concave area at the molecular edge, and the hydrogen atoms in this area

Known as "Bay Area Hydrogen" (see Figure 1), the ¹HNMR spectrometer can sensitively identify and quantify the hydrogen atoms in this area, which is called polycyclic aromatics.

The characteristic hydrogen atom of hydrocarbons.

This method describes the steps of using ¹HNMR to determine the "Bay Area Hydrogen" content (mass fraction) in the sample solution to characterize vulcanized rubber

Aromatic properties of China Oil. The higher the "Bay Area Hydrogen" content, the higher the aromaticity.