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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 29610-2013

Rubber products - Determination of polybrominated biphenyls and polybrominated diphenyl ethers - Gas chromatography and mass spectrometry

橡胶制品 多溴联苯和多溴二苯醚的测定 气相色谱-质谱法

Issued on: July 19, 2013 **Implemented on: December 1, 2013**

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

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Foreword

This standard was drafted in accordance with GB/T 1.1-2009 given rules. The standard proposed by China Petroleum and Chemical Industry Association. This standard by the national Common test methods for rubber and rubber goods Technical Committee of Standardization Technical Committee (SAC/TC35/SC2) Centralized. This standard is drafted by: Beijing Rubber Industry Research and Design Institute, Beijing Physical and Chemical Analysis Center. The main drafters of this standard. Wei Yan, Wei Wei, Ding Xiaoying, Xiejun Fang. Rubber products, polybrominated biphenyls and polybrominated diphenyl ethers By Gas Chromatography - Mass Spectrometry Warning

--- personnel should use this standard regular laboratory work experience. This standard does not point out all possible security asked Questions, the user has the responsibility to take appropriate safety and health practices and to ensure compliance with the conditions relevant national regulations.

1 Scope

China's national method for determining polybrominated biphenyls and polybrominated diphenyl ethers in rubber products by gas chromatography with mass spectrometric detection. It specifies the principle, the reagents, the apparatus, the sample preparation, the procedure and the expression of results for the determination of PBBs and PBDEs in rubber. Polybrominated biphenyls and polybrominated diphenyl ethers were the workhorse brominated flame retardants of the late twentieth century, added to plastics, rubbers, textiles and electronics in large quantities because they work extremely well: a small addition makes a polymer far harder to ignite. They are also persistent, they bioaccumulate, and they are found in human tissue and in wildlife everywhere including places where they were never used. They are restricted under the electrical and electronic equipment substance rules and under the persistent organic pollutant conventions, and a product containing them above the limits cannot be sold into the markets that apply those rules. That is what makes a determination method commercially necessary rather than merely

scientifically interesting: a rubber component in an appliance, a vehicle or a piece of equipment has to be shown not to contain them, and the manufacturer of the component has to be able to demonstrate it to its customer. The determination is by extraction from the rubber followed by gas chromatography with mass spectrometric detection. Rubber is a difficult matrix for exactly this analysis: it is crosslinked so it does not dissolve, it is heavily filled with carbon black which adsorbs the analytes, and it contains oils and other additives that interfere. So the standard fixes the extraction, the clean-up, the chromatographic conditions and the ions monitored, together with the calibration and the precision. Issued on 19 July 2013 and in force since 1 December 2013.

This standard specifies the use of gas chromatography - Determination of rubber products PBBs (polybromobiphenyls mass spectrometry, referred to PBBs, knot Constructions see Figure 1) and polybrominated diphenyl ethers (polybrominateddiphenylethers, called PBDEs, structural formula shown in Figure 2) method. This standard applies to the determination of rubber products PBBs and PBDE content in, but also for rubber tires polybrominated biphenyls and Determination of PBDE content. PBDE Structure Structure Fig.1 PBBs over two

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version suitable for use herein Member. For undated references, the latest edition (including any amendments) applies to this document. Determination of matter

GB/T 3516 rubber solvent extraction

3 Method summary

Sample extraction using toluene as solvent Soxhlet extraction, the extract was concentrated and treated by gas chromatography - mass spectrometer for quantitative analysis.

4 Testing and Materials

4.1 Unless otherwise stated in the analysis using only recognized as analytical grade reagents and distilled water or equivalent purity.

4.2 Toluene. chromatography.

4.3 anhydrous sodium. pure class distinctions.

4.4 silica gel. chromatography, particle size 0.15mm ~ 0.25mm (60 mesh to 100 mesh), activation of 10h ~ 12h at 360 °C, dry cooling Reservoir within the dryer.

4.5 dichloromethane. chromatography.

4.6 quartz wool. wash or bake 4h at 400 °C with dichloromethane (4.5).

4.7 liquid nitrogen. 99.999% purity.