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

GB/T 15251-2008

Replacing GB/T 15251-1994; GB/T 3514-1992

Rubber -- Determination of free sulfur

橡胶 游离硫的测定

(English Translation)

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Foreword

This revised standard adopts ISO 7269:1995 "Determination of free sulfur rubber" (in English), including its amendments ISO 7269:1995 /Cor. 1.2002 (in English).

This standard replaces GB/T 3514-1992 "vulcanized rubber Determination of sulfur content of free sulfite method", GB/T 15251-1994 "Determination of free sulfur rubber copper spiral method." this standard is based on ISO 7269:1995 re-drafted.

This standard is the international standard ISO 7269:1995 and its main difference causes changes reg structured as follows.

--- Increasing the standard iodine titration solution and sodium thiosulfate standard titration solution preparation and calibration method (This standard 5.1.7,5.1.8), in order to enhance the standard of operability.

--- In the sulfite process, added porcelain zeolite (this standard 7.1.2) when heated; use ordinary conical flask (7.2.1 of this standard); silicon Diatomaceous earth by the thickness of 5mm to 2mm (ISO 7269:1995 7.3.3; 7.3.3 of this standard); instead of the surface with a small funnel Cover dish conical mouthpiece (ISO 7269:1995 7.3.1; 7.3.1 of this standard). Such provisions enhance the operability of this standard for sex.

For ease of use, this standard made the following editorial changes.

- a) "this International Standard" by "this standard";
- b) Delete the international standard preface;
- c) increase the informative Appendix B.

This standard is GB/T 3514-1992 and GB/T 15251-1994 revision of integration, and technical content GB/T 3514-1992 and GB/T 15251-1994 compared to the main changes are as follows.

- Increased introduction;
- Increased warnings;
- Increase the limitations of standard instructions (edition 4);
- Increased rapid extraction method, copper spiral method B (6 edition);
- Increasing the size measurement method used glassware (GB/T 3514-1992 of 4, GB/T 15251-1994 5;
Excerpts 5.2,6.2,7.2);
- Increasing the standard iodine titration solution and sodium thiosulfate standard titration solution preparation and calibration methods (Excerpts 5.1.7, 5.1.8);
- Modify the processing method of compound (GB/T 3514-1992 of 5, GB/T 15251-1994 6;
Excerpts 5.3,6.3, 7.3);
- In the sulfite method. when heated to prevent foaming, an increase of liquid paraffin (Excerpts 7.1.10,7.3.1); instead of using diatomaceous earth Asbestos as a filter packing (GB/T 3514-1992 3.8 and 5; edition of 7.1.9 and 7.3.3);
- Modified test procedure. The back-titration method instead of direct titration (GB/T 3514-1992 5; edition 7.3.4);
- Increased content requires the test report (edition 8);
- The test replicates tolerance to informative Appendix B (GB/T 3514-1992 7; edition of Appendix B);
- Increase the informative appendix, accelerator on (Excerpts Appendix A) on the test results.

The Standard Appendix A and Appendix B are informative.

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Standards Commission rubber physical and chemical test methods Technical Committee (SAC/TC35/SC2) centralized.

The main drafting standard units. Beijing Rubber Industry Research and Design Institute, Hangzhou Zhongce Rubber Co., Ltd.

Introduction

Uncured rubber compounds content of free sulfur (such as rubber compound unbound sulfur content) should represent all of the added elemental sulfur.

As the sulfur and gradually combine with the rubber vulcanization, the number of free sulfur will be gradually reduced, so the recipe is known and elemental sulfur (package Under

appropriate ratio including other components), the number of free sulfur in any given product in both its degree of cure of a certain relationship.

A high percentage of free sulfur vulcanized rubber easier blooming, so in some specific product specification need to be shown the largest free sulfur content value.

Rubber - Determination of free sulfur Warning --- personnel should use this standard regular laboratory work experience. This standard does not point out all possible security asked question. Users have the responsibility to take appropriate safety and health practices and to ensure compliance with the conditions relevant national regulations.

1 Scope

This standard specifies the vulcanized rubber in the determination of free sulfur three methods, namely, Auger and one of Copper sulfite method.

This standard applies to all kinds of vulcanized rubber two copper spiral method also applies to Chapter 4 describes the unvulcanized rubber material within a confined range.

Appendix A provides more accelerators Goods influence of free sulfur determination, expressed in mass fraction.

2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, subsequent Amendments (not including errata content) or revisions do not apply to this standard, however, encourage the parties to the agreement are based on research Whether the latest versions of these documents. For undated reference documents, the latest versions apply to this standard.

GB/T 3516 Determination of rubber solvent extract (GB/T 3516-2006, ISO 1407:1992, MOD) ISO 383. 1976 Laboratory glassware interchangeable cone Interface Principle 3

3.1.1 Method A

Solvent extraction method according to GB/T 3516 regulations, with acetone extraction of the free sulfur in a sample, and placing the flask in acetone Place a helical copper mesh. The extracted copper and sulfur reaction and produce copper sulfide. Treated with hydrochloric acid does not remain in the flask Dissolved in acetone copper and copper sulfides. With cadmium acetate buffer solution to absorb the generated hydrogen sulfide, the resulting final determination of cadmium by iodometry Sulfides.

3.1.2 Method B

Fast copper and copper helix spiral method B A similar method, except that the use of Method B have a vapor jacket Soxhlet extraction to reduce the time between. Soxhlet

extractor by reducing the volume (volume 6mL) to increase the extraction temperature.

3.2 Sodium sulfite method

Free sulfur in a sample is reacted with sodium sulfite, sodium thiosulfate generated measured by iodometry.

4 Limitations

4.1

Determination of copper spiral method usually only elemental sulfur content, but thiuram disulfide and diamine sulfide sulfur vulcanizing agent will guide carrier Cause high results. In addition, the unvulcanized rubber, elemental sulfur is added if insoluble in acetone, or the so-called insoluble amorphous sulfur, the copper Auger does not apply. In a sample of the total mass of free sulfur should be between 0.2mg ~ 10mg.

4.2

Method of sulfite sulfur vulcanizing agent and antioxidant carrier too sensitive, and does not apply to uncured rubber.

4.3

These methods without sulfur donor curing agent is relatively precise conditions.

Note: In fact, in addition to the determination of elemental sulfur, a number of active sulfur compounds, such as the disulfide and polysulfide sulfur, will produce the same experiment effect.