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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 15252-2014

Replacing GB/T 15252-1994

Rubber, compounded or vulcanized - Determination of sulfide sulfur content - Lodometric method

混炼胶或硫化胶 硫化物型硫含量的测定 碘量法

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Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;

Standardization Administration of the People's Republic of China.

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Foreword

This Standard replaces GB/T 15252-1994 “Rubber - Determination of sulfide sulfur content - Iodometric method.” Compared with the standard GB/T 15252-1994, the main differences are as follows:

- MODIFY the standard name;

- ADD the foreword;

- ADD the warning;

- REVISE “cadmium acetate buffer solution” TO “zinc acetate buffer solution” (see 4.4, 4.2 of 1994 edition);.

- MODIFY the description of the surfactant solution (see 4.5, 4.4 of 1994 edition);

- ADD the description of “all-glass extraction device” (see 5.1);

- ADD the description of “reaction between hydrogen sulphide and absorption device” (see 5.2);

- REVISE “burette” TO “brown burette” (see 5.6, 5.3 of 1994 edition);

- REVISE the reaction heating time from “30min ~ 40min” to “0min ~ 80min” (see 7.1.2, Chapter 6 of 1994 edition);

- MODIFY the description of “Analysis steps” (see Chapter 7, Chapter 6 of 1994 edition);

- ADD the content of the test report (see Chapter 9).

This Standard was proposed by China Petroleum and Chemical Industry Association.

This Standard shall be under the jurisdiction of the General Methods of Test Branch of the National Standardization Technical Committee on Rubber and Rubber Technology (SAC/TC 35/SC 2).

Responsible drafting organization of this Standard: Xuzhou Xugong Tyres Co., Ltd.

Participating drafting organizations of this Standard: Qingdao Yikesi New Material Co., Ltd., Guangzhou Institute of Synthetic Material Co., Ltd., Beijing Center for Physical and Chemical Analysis, Beijing Research and Design Institute of Rubber Industry.

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Rubber, compounded or vulcanized - Determination of sulfide sulfur content - Lodometric method

Warning: Personnel who use this Standard shall have experience of regular laboratory work. This Standard does not point out all possible safety issues; users have the responsibility to take appropriate safety and health measures, and to ensure that it complies with the conditions specified in relevant national regulations.

1 Scope

This Standard specifies the test methods for determination of sulfide sulfur content in compounded and vulcanized rubber by iodometric method.

This Standard applies to compounded and vulcanized rubber of halogenated rubber, nitrile rubber and hydrocarbon rubber (including natural rubber).

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB/T 601-2002 Chemical reagent - Preparations of standard volumetric solutions

GB/T 603-2002 Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 3516-2006 Rubber - Determination of solvent extract

GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods

GB/T 17783-1999 Rubber, vulcanized - Preparation of samples and test pieces - Chemical tests

3 Principle

For rubbers extracted by acetone, use the mixture of hydrochloric acid and acetic acid

5.5 25mL pipette (grade A).

5.6 Brown burette.

6 Sampling and sample preparation

6.1 CUT the needed samples from vulcanized rubber specimen and finished rubber products according to the specifications in GB/T 17783-1999. For compounded rubber, it shall be made into compounded rubber slices by open mills with a roll pitch of not more than 0.5mm for 6 times at room temperature, and directly cut the test samples into small particles with a side length of less than 1mm. Vulcanized rubber is directly cut into small particles with a side length of less than 1mm.

6.2 WEIGH 0.5g ~ 2.0g of sample, accurate to 0.1mg.

6.3 WRAP the sample with filter paper, PLACE the sample in the the extraction unit (5.1) containing acetone (4.2) to extract for 6h ~ 8h.

6.4 TRANSFER the sample from Soxhlet extraction bottle to a weighing bottle; DRY in a 70°C oven for 15 min; PUT the dried sample into the extraction bottle (A).

7 Procedures

7.1 Determination

7.1.1 INSTALL the extraction bottle (A) at the unit shown in Figure 2, all connectors are sealed with glycerin or petrolatum (4.10). ADD 100mL of zinc acetate buffer solution (4.4) and 1mL of surfactant (4.5) into a conical flask (I). ADD zinc acetate buffer solution into the gas washing bottle (B and C), until it fills half cylinder capacity. INLET nitrogen (4.9), then adjust the flow rate of nitrogen in the absorption bottle to about one bubble per second.

7.1.2 ADD 50mL of hydrochloric acid - acetic acid mixture solution (4.3) slowly from the separating funnel (G) to the extraction bottle (A); HEAT slowly to boiling; MAINTAIN at micro-boiling for 60min ~ 80min. After the termination of heating; ADD appropriate nitrogen flow, to remove any residual hydrogen sulfide. At this moment, the solution in the washing bottle (C) shall has no white precipitate. Otherwise, it must be remeasured with a smaller sample or slower gas flow speed.

7.1.3 DISSEMBLE the receiver connector (E), to cool down the solution in the conical flask to about 15°C; under cooling condition, accurately transfer iodine solution (4.6) with a volume of 25.00mL into the conical flask (I) by a pipette, to make the iodine excess. PLACE the Erlenmeyer flasks (I) in the dark for 20min to make the iodide reacts with the precipitate adheres on the receiver; after the precipitate dissolving,

a) Standard number and name of this Standard;

b) Detailed description of test samples;

c) Test results;

d) Any anomalies observed during the determination;

e) All operations that are not included in this Standard;

f) Test date.

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