NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 17783-2019

Rubber, vulcanized or thermoplastic - Chemical tests - Preparation of samples and test piece

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1 Scope

This Standard specifies the preparation methods for vulcanized rubber or thermoplastic rubber sample and test piece for chemical tests.

This Standard is applicable to the preparation of vulcanized rubber or thermoplastic rubber sample and test piece for chemical tests.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 18866, Rubber - Dissolution by acid digestion (GB/T 18866-2017, ISO 9028:2006, MOD)

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 cryogenic size reduction; cryo-grinding

Use liquid nitrogen or other refrigerants to brittle rubber and process into small particle sizes.

[GB/T 9881-2008, definition 2.114]

3.2 thermoplastic rubber

A polymer or polymer blend that has properties similar to vulcanized rubber at its use temperature but it can be processed or re-processed after temperature is elevated like thermoplastic plastic.

[GB/T 9881-2008, definition 2.431] rubber product, it shall separate the vulcanized rubber and other possible ingredients from the product first, for example, metals, cords, cable ties, fibers, and other coated films or coverings. When separating, it shall use mechanical methods whenever possible -- blades, wheels, files and avoid heating.

6.1.2 The preparation of sample and test piece is mainly divided into two categories: one is carried out according to the nature of rubber and the other is carried out according to the use of chemical test.

6.2 Soft vulcanized rubber Use scissors, files, grinders, freeze mashers or liquid nitrogen prototypes to crush the sample. Through a test sieve of which the aperture is about 1.7mm, or through a laboratory mill, under small roll distance, coldly roll and press the sample into sheets of which the thickness is not greater than 0.5mm. When using a grinder or mill to prepare a sample, be careful not to contaminate the sample or overheat it.

6.3 Hard vulcanized rubber Use a file to file the sample into powder or use a grinder or frozen mashing or use liquid nitrogen prototype to crush the sample. Through a test sieve of which the aperture is about 0.4mm and use a magnet to remove iron filings from powder.

6.4 Rubber composite material

6.4.1 When rubber cannot be separated from other materials by mechanical methods, it shall be treated according to 6.4.2~6.4.4 methods.

6.4.2 Expose a sample to a suitable solvent vapor to make rubber swell. Then separate rubber from other materials. In a fume hood, the solvent in the swelling rubber is completely evaporated. Proceed according to the method described in 6.2. For NR-based, SBR-based and BR-based composites, acetone (4.1) or ETA (4.3) are more suitable solvents. For other types of rubbers, see Annex C for the selection of solvent.

The time that the sample is exposed to steam is limited to the separation of rubber from other materials and it shall be as short as possible, lest the plasticizer in rubber be extracted.

Swelled rubber can be placed in a fume hood. Remove all solvents by evaporation at room temperature. Then according to the method described in 6.2, conduct subsequent processing of rubber.

6.4.3 When rubber is chemically bonded to other substrates, it shall note the rubber

composition of the bonding area, which may be very different from the

6.7.1.1 Dry ashing Use dry ashing for halogen-free vulcanized or thermoplastic rubber test piece.

Weigh about 0.2g of test piece (to the nearest of 0.1mg).

According to the provisions of 6.1 and 6.2, prepare the test piece into granular of which the particle size is not more than 1mm. Put into a porcelain crucible.

Conduct a complete low-temperature carbonization on a temperature-controlled electric furnace. Move the crucible into a muffle furnace of which the furnace temperature is (550+/-25)°C to continue heating till the ashing is complete. Keep the ashes in the crucible.

6.7.1.2 Wet ashing Use wet ashing for halogen-containing or unknown test piece. Weigh about 0.2g of test piece (to the nearest of 0.1mg). According to the provisions of 6.1 and 6.2, cut the sample into granular of which the particle size is not more than 1mm.

Put into a porcelain crucible. Add about 3mLof sulfuric acid (4.2) to make test piece digested (the amount of sulfuric acid can be slowly increased according to the degree of swelling of test piece and the severity of the reaction); or use a digestion instrument to digest the sample. Place the crucible that contains test piece in the hole of the asbestos board. In the fume hood, use an electric stove to slowly heat. For example, at the starting state of reaction, when the test piece swells severely, it shall remove the heat source so as to avoid possible sample loss. When the reaction is slow, raise the temperature until excess sulfuric acid evaporates, leaving a dry carbonized residue. Move the crucible that is filled with residues to a muffle furnace of which the furnace temperature is (950+/-25)°C to continue heating till ashing is complete. Keep the ashes in the crucible.

6.7.2 Ash dissolution After test piece ashing, cool to room temperature. Move ashes into a beaker.

Use a measuring cylinder to measure 15mL of nitric acid solution (4.4). Pour into the beaker. Heat carefully on the electric furnace until they are completely dissolved. Cool to room temperature. Move to a 250mL volumetric flask (if ashes cannot be completely dissolved, the solution needs to be filtered). Use water to dilute to the scale, shake well for testing.

6.7.3 Acid digestion For rubber materials that are not applicable for ashing, conduct according to the provisions in GB/T 18866.