SRI LANKA STANDARD 899 : PART 3 : 1992

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METHODS OF TEST FOR

RUBBER COMPOUNDING INGREDIENTS

PART 3 - SULFUR

SRI LANKA STANDARDS INSTITUTION

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SLS 899 : 1992

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SRI LANKA STANDARDS INSTITUTION
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SRI LANKA STANDARD

METHODS OF TEST FOR RUBBER COMPOUNDING INGREDIENTS

PART 3 : SULFUR

FOREWORD

This Standard was finalized by the Sectoral Committee on Rubber and Rubber Products and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1992-03-26.

This part is one of the series of standards Methods of Test for Rubber Compounding Ingredients. The first and second parts of this standard which have been issued cover the methods of test for carbon black and zinc oxide respectively.

This part of the standard consists of the following sections.

Section 1 : Determination of total sulfur content.
Section 2 : Determination of sieve residue.
Section 3 : Determination of carbon disulfide insoluble matter.
section 4 : Determination of ash.
Section 5 : Determination of acidity.
Section 6 : Determination of loss in mass.
Section 7 : Determination of copper and manganese.

In reporting of result of a test or an analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with **CS 102**.

In the preparation of this standard the assistance obtained from the Publications of the International Organization for Standardization and the British Standards Institution is gratefully acknowledged.

1 SCOPE

This standard prescribes the methods of test for any commercial sulfur used as rubber compounding ingredient.

2 REFERENCES

CS 102 Presentation of numerical values
CS 124 Test sieves
SLS 428 Random sampling methods

3 SAMPLING

Samples required for the tests specified in the relevant sections of this standard shall be obtained as prescribed in the corresponding clause of the product standard.

SECTION 1 : DETERMINATION OF TOTAL SULFUR

1 PRINCIPLE

The sulfur is first converted to thiosulfate which is then measured by titration with an iodine solution.

2 REAGENTS

Unless otherwise specified, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

2.1 *Sodium sulfite,* 50 g/l solution.

2.2 *Standard iodine solution,* freshly prepared, $c(I_2) = 0.05$ mol/l.

2.3 *Formaldehyde solution,* 40 per cent (V/V).

2.4 *Glacial acetic acid.*

2.5 *A wetting solution,* dissolved 2 g of arylalkylsulfonate in 100 ml water.

2.6 *Starch solution,* 10 g/l.

3 PROCEDURE

Grind the sample to pass through a sieve of 150 u. Weigh to the nearest 0.1 mg about 0.2 g of the ground sample in a watch glass. Add about 1 ml of wetting solution (2.5)) using a pipette. Transfer the mixture to a 250-ml flask through a small glass funnel using about 25 ml of sodium sulfite solution (2.1). Add a few antibumping granules, fix an air-condenser and simmer the contents gently for a period of 2 hours on an electrically heated sand bath. Control the temperature to avoid frothing or formation of large bubbles. Remove the flask and allow it to cool to 15 °C. Wash the walls of the condenser with distilled water and collect the washings in the flask. Remove the condenser. Transfer the contents into a 100-ml volumetric flask. Make up the solution to 100-ml with distilled water.

Pipette 25 ml of the solution to a conical flask and add 10 ml of formaldehyde solution (2.3), 25 ml of the acetic acid solution (2.4) and 1 ml of the starch solution (2.6). Titrate with standard iodine solution (2.2) until a blue colour is obtained which persists for 15 second. Carry out the titration in triplicate and report the average value.

Carry out a blank determination.

4 CALCULATION

$$\text{Total sulfur, per cent by mass} = \frac{(V_1 - V_2)\ c \times 4 \times 64}{10m}$$

where,

V_1 is the volume, in ml, of the iodine solution required for the titration of the sample;
V_2 is the volume, in ml, of the iodine solution required for the blank solution;
c is the concentration, in mol/l, of the iodine solution; and
m is the mass, in g, of the sample.

SECTION 2 : DETERMINATION OF SIEVE RESIDUE

1 . APPARATUS

1.1 *Test sieves,* 200 mm diameter, having a fitting lid and reciever, with aperture sizes of 150 , 75 conforming to **SLS 124**;

1.2 *A mechanical test sieve shaker;*

2 PROCEDURE

Arrange the sieve one above the other with the largest aperture sieve (150 microns) on top, and the smallest aperture (75 microns) at the bottom. Blend the total sample and then weigh, to the nearest milligram, about 50 g of the sample, using a weighing bottle.

Transfer the sample onto the top sieve and fit the lid and shake for 5 minutes in the mechanical shaker (1.2). Inspect the top sieve, if the material remain break up any loose agglomerates by brushing lightly with the camel hair brush and shake for further 5 minutes. Transfer all the material retained on each sieve to a clean dry tared weighing bottle. Weigh, each weighing bottle containing sieve residue to the nearest milligram.

3 CALCULATION

$$\text{Residue on 150 microns sieve, per cent by mass} = \frac{m_1}{m} \times 100$$

$$\text{Residue on 75 microns sieve per cent by mass (including residue on 150 microns sieve)} = \frac{m_2}{m} \times 100$$

where,

m is the mass, in grams, of the test portion;
m_1 is the mass, in grams, retained on 150 microns sieve; and
m_2 is the mass, in grams, retained on 75 microns sieve;

SECTION 3 : DETERMINATION OF CARBON DISULFIDE INSOLUBLE MATTER

1 REAGENTS

Unless otherwise specified, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

1.1 *Carbon disulfide* - Reagent grade special safety precautions should be required when using carbon disulfide since it is a toxic and inflammable solvent.

2 APPARATUS

2.1 *Glass filter crucible,* porosity p 4.

2.2 *Oven,* maintained at 80 ± 5°C.

3 PROCEDURE

Weigh, to the nearest milligram, about 5 g of the sample in a 250-ml beaker. Add 100 ml of carbon disulfide and stir for 30 min using a magnetic stirrer. Filter through a pre-weighed crucible (2.1) applying very light suction. Wash three times with 20 ml portions of carbon disulfide and dry (see note) the crucible in oven (2.2) at 80 ± 5 °C for 1 hour. Cool in a desiccator and weigh.

NOTE

Do not allow the solution to pass through completely before the last washing in order to prevent sulfur deposition.

3 CALCULATION

$$\text{Insoluble sulfur, per cent by mass} = \frac{m_2 - m_1}{m}$$

where,

m_1 is the mass, in grams, of the glass crucible;
m_2 is the mass, in grams, of the glass crucible with the residue; and
m is the mass, in grams , of the test sample

SECTION 4 : DETERMINATION OF ASH

1 APPARATUS

Furnace, maintained at 875 ± 25°C.

2 PROCEDURE

Weigh, to the nearest milligram, about 50 g of the sample into a silica dish, previously heated at 875 ± 25 °C. Place the dish on a hot plate and gently melt the sulfur. When melting is completed, set fire to the sulfur with a small gas flame. Adjust the heating rate so that the sulfur burns slowly. When the burning is completed, place the dish in the furnace (1) at 875 ± 25 °C for 1 hour. Allow to cool in a desiccator and weigh to the nearest milligram. Repeat the process of heating, cooling and weighing until the constant mass is reached.

3 CALCULATION

$$\text{Ash, per cent by mass} = \frac{m_2 - m}{m} \times 100$$

where,

m is the mass, in grams, of the empty dish;
m_1 is the mass, in grams, of sample; and
m_2 is the mass, in grams, of the dish with residue.

SECTION 5 : DETERMINATION OF ACIDITY

1 REAGENTS

Unless otherwise specified, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

1.1 *Propan-2-ol,* previously boiled, cooled and neutralized to phenolphthalein with sodium hydroxide solution.

1.2 *Standard sodium hydroxide,* $c(NaOH) = 0.1$ mol/l solution

1.3 *Phenolphthalein,* 10 g/l ethanolic solution.
Dissolve 1 g of phenolphthalein in 60 ml of 95 per cent (V/V) ethanol and dilute to 100 ml with water.

2 PROCEDURE

Weigh, to the nearest milligram, about 25 g of the sample into a conical flask and add 25 ml of the propan-2-ol (1.1) and shake until the sample is completely wetted. Then, add 50 ml of water, shake and allow to stand for 20 minutes. Titrate with the sodium hydroxide solution (1.2) in the presence of a few drops of the phenolphthalein solution (1.3) to the first permanent pink colour.

3 CALCULATION

$$\text{Acidity, (as sulfuric acid), percent by mass} = \frac{4.9 \times V \times C}{m}$$

where,

c is the concentration, in mol/l of the sodium hydroxide solution;

V is the volume, in millilitres, of the standard sodium hydroxide solution; and

m is the mass, in grams of the sample.

SECTION 6 : DETERMINATION OF LOSS IN MASS AT 80°C

1 APPARATUS

Oven, maintained at 80 ± 5°C.

2 PROCEDURE

Weigh, to the nearest milligram about 50 g of the sample in a dried, tared weighing bottle. Keep the bottle and the stopper in the oven at 80 ± 2°C for 3 hours. Allow to cool in a desiccator and weigh. Repeat the process of heating, cooling and weighing until the constant mass is reached.

3 CALCULATION

$$\text{Loss in mass, per cent by mass} = \frac{m_2 - m}{m_1 - m} \times 100$$

where,

m_0 is the mass, in grams of the test portion before heating; and
m_2 is the loss in mass, in grams after heating.

SECTION 7 : DETERMINATION OF COPPER AND MANGANESE

1 REAGENTS

Unless otherwise specified, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

1.1 *Hydrochloric and nitric acid mixture*
2 volumes of hydrochloric acid, (rel. den = 1.18) 1 volume of nitric acid, (rel. den = 1.42) and 3 volumes of water.

1.2 *Citric acid,* 500 g/1 solution.
Dissolve 50 g of citric acid in 100 ml water.

1.3 *Ammonia solution* (rel. den = 0.880).

1.4 *Stabilized water*
Distill about 1 litre of water containing about 0.1 g of potassium permanganate and few drops of sulfuric acid and collect the distillate discarding the first 50 ml.

1.5 *Standard potassium permanganate,* $c(KMnO_4)$ = 0.005 mol/1.

1.6 *Sulfuric acid,* (rel den = 1.84).

1.7 *Sulfuric acid diluted*

1.8 *Orthophosphoric acid,* 85 to 90 per cent H_3PO_4 (V/V).

1.9 *Phosphoric acid dilute (V/V) with orthophosphoric acid (1.8)*.

1.10 *Potassium periodate*

1.11 *Potassium hydrogen sulfate,* fused and powdered.

1.12 *Standard solution for metals*

Prepare, standard solutions from pure metals, metalic compounds, or manufactured standard solution. Bring these solutions into a linear working range of the instrument by diluting with water.

2 APPARATUS

2.1 *Muffle furnace,* maintained at 550 ± 25°C.

2.2 *Atomic absorption spectrophotometer,* operating in the flame mode. The instrument must be operated according to the manufacturers directions for optimum performances of flame operation.

2.3 *Sintered glass filter,* porosity P 3.

3 PROCEDURE

3.1 Preparation of solutions

3.1.1 *Copper test solution*

Weigh, to the nearest milligram, about 5 g of the sample into a crucible, previously heated to 550 ± 25 °C in the muffle furnace (2.1) for 30 minutes. Allow to cool in a desiccator and weigh. Repeat the process heating, cooling and weighing until the constant mass is reached. Moisten the contents with 1 ml of water, add 10 ml of hydrochloric and nitric acid mixture (1.1), and heat on a boiling water bath for 30 min to 60 min. Wash the contents of the crucible into a small beaker, add 5 ml of citric acid solution (1.2), and then ammonia solution (1.3) dropwise until the solution is just alkaline. Cool the solution and add 2 ml of ammonia solution (1.3).

3.1.2 *Manganese test solution*

Weigh, to the nearest milligram, about 50 g of the sample into a crucible previously heated to 550 ± 25 °C in the muffle furnace (2.1). Then transfer the crucible to the furnace (2.1) and heat until the ash is substantially free of carbon. Add few drops of sulfuric acid (1.6) to moisten the residue. Carefully heat the crucible so that copious evolution of sulfur trioxide occurs, until the acid is driven off and fuming ceases, heat for a few minutes longer. Repeat this process until all organic matter is destroyed. Moisten with a little sulfuric acid and gently ignite. Avoid strong ignition in order to retain the ash in the form of sulfate. Add 5 g of potassium hydrogen sulfate (1.10) and fuse cautiously at first, gradually increasing the temperature to a moderate red heat, until a clear melt is obtained. Cool and add 2 ml of dilute sulfuric acid (1.7) and 15 ml of water Transfer the content to a 100-ml beaker and heat until all soluble matter is dissolved. Cool to 10 °C, filter through a sintered glass filter (2.3), and wash the beaker with a few millilitres of water. Transfer the filtrate and washings to a 100-ml conical flask. Adjust the volume of the filtrate to approximate 40 ml with water add 4 ml of dilute phosphoric acid (1.9) and mix well.

If the solution is yellow in colour add 1 ml portions of phosphoric acid at a time until the solution is colourless. Add 0.3 g of potassium periodate (1.10) raise to the boil for 10 minutes and keep above 90 °C for a further 10 minutes to ensure full development of the permanganate colour. Cool to 20 °C and dilute to 50 ml using stabilized water (1.4) and mix well.

NOTE

The permanganate colour is stable for a period of long time and any sign of fading indicates incomplete removal of either organic matter or chloride.

3.1.3 *Blank solution*

Prepare the blank solutions omitting samples.

3.1.4 *Dilute test solutions and blank solutions*

Dilute, test solutions (3.1.1, 3.1.2) and the blank solution (3.1.3) with water to a concentration within the optimal measuring range of the spectrophotometer.

3.2 **Measurement**

3.2.1 *Copper*

Set the spectrophotometer (2.2) at a wave length 324.4 nm using an oxidizing air-acetylene flame. Aspirate successively in triplicate the copper standard solution (1.11) the dilute test solution (3.1.4) and the blank solution (3.1.3), washing with water between each aspiration. Plot calibration curve using the median absorbances of the standard solution against concentration of copper. Determine the concentration of copper in the test solution and the blank solution with reference to the calibration curve.

3.2.2 *Manganese*

Carry out the measurements as (3.2.1) at a wave length 279.5 nm using an oxidizing air-acetylene flame. Plot calibration curve using the median absorbances of the standard solution against concentration of manganese. Determine the concentration of manganese in the test solution and the blank solution with reference to the calibration curve.

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SRI LANKA STANDARDS INSTITUTION

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