SRI LANKA STANDARD 899 : PART 2 : 1991

UDC 678.021.17 : 661.847.9

METHODS OF TEST FOR

RUBBER COMPOUNDING INGREDIENTS

PART 2 - ZINC OXIDE

SRI LANKA STANDARDS INSTITUTION

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SLS 899:1991

Gr. 7

SRI LANKA STANDARDS INSTITUTION
53, Dharmapala Mawatha,
Colombo 3,
Sri Lanka.

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SRI LANKA STANDARDS INSTITUTION

SRI LANKA STANDARD
METHODS OF TEST FOR RUBBER COMPOUNDING INGREDIENTS
PART 2 - ZINC OXIDE

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1991-11-14, after the draft, finalized by the Drafting Committee on Rubber compounding ingredients, had been approved by the Chemicals Divisional Committee.

This part is one of the series of standards methods of test for rubber compounding ingredients. The first part of this which have been issued covers the methods of test for carbon black.

This part of the standard consists of the following sections:

Section 1 : Determination of matter volatile at 105 °C
Section 2 : Determination of matter soluble in water
Section 3 : Determination of acidity
Section 4 : Determination of residue on sieve
Section 5 : Determination of lead, cadmium, copper, manganese and iron
Section 6 : Determination of loss on ignition
Section 7 : Determination of zinc oxide

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, British Standards Institution and Industrial Research Institute of Malaysia is gratefully acknowledged.

1 SCOPE

This standard prescribes the methods of test for Zinc oxide used as rubber compounding ingredient. The methods covered in this standard are applicable for all commercial zinc oxides.

2 REFERENCES

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

3 SAMPLING

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

SECTION 1 : DETERMINATION OF MATTER VOLATILE AT 105^{O}C

1 APPARATUS

1.1 *Weighing bottle*

1.2 *Oven,* maintained at 105 ± 2 ^{o}C.

2 PROCEDURE

Dry the weighing bottle (1.1) with the stopper in the oven (1.2). Allow to cool and weigh, to the nearest milligram, about 10 g of the sample in the weighing bottle. Spread the sample to form a uniform layer on the bottom of the weighing bottle and keep in the oven, at 105 ± 2 ^{o}C (1.2) without the stopper for 1 h. Allow to cool in a desiccator with the stopper and weigh to the nearest milligram. Repeat the process of heating, cooling and weighing at 30 minute intervals until the difference between two successive weighings does not exceed 0.1 g. Record the lowest mass.

3 CALCULATION

$$\text{Matter volatile at 105 } ^{O}\text{C, per cent by mass} = \frac{m_0 - m_1}{m_0} \times 100$$

where,

m_0 is the mass, in grams of the test sample; and
m_1 is the mass, in grams of the residue.

SECTION 2 : DETERMINATION OF MATTER SOLUBLE IN WATER

1 REAGENTS

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

2 APPARATUS

Oven, maintained at 105 ± 2 °C.

3 PROCEDURE

Weigh to the nearest milligram, about 20 g of the sample, into a beaker. Wet the sample with a few millilitres of water. Add 200 ml of water and stir continuously for 1 h at room temperature. Transfer to 250 ml - volumetric flask and dilute upto the mark with the water. Mix thoroughly and filter through a colloid filter returning the filtrate to the filter until it runs clear. Evaporate 100 ml of the clear filtrate to dryness in the previously weighed evaporating dish on a water bath.

Dry the residue in the evaporating dish in the oven at 105 ± 2 °C (2) cool to the room temperature and weigh. Repeat the process of heating, cooling and weighing at 30 minute intervals until the difference between two successive weighings does not exceed 0.1 g. Record the lowest mass.

4 CALCULATION

$$\text{Water soluble matter, per cent by mass} = 250 \frac{m_1}{m_0}$$

where,

m_0 is the mass, in grams, of the test sample; and
m_1 is the mass, in grams, of residue.

SECTION 3 : DETERMINATION OF ACIDITY

1 REAGENTS

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

1.1 *Methyl red indicator* , 1g/1 solution in 60 per cent (V/V) ethanol.

1.2 *Standard sodium hydroxide,* solution, C(NaOH) = 0.05 mol/1.

2 PROCEDURE

Weigh, to the nearest milligram, about 20 g of the sample into a beaker. Wet the sample with a few millilitres of water. Add 200 ml of water and stir and boil for 5 min. Cool to the room temperature, transfer to 250 ml - volumetric flask and dilute upto the mark. Mix thoroughly and filter through a filter, returning the filtrate to the filter until it runs clear. Pipette 100 ml of the clear filtrate and add 5 drops of the methyl red indicator (1.1).

If the solution is orange, report the sample being neutral.
If the solution is red titrate it with the sodium hydroxide solution (1.2) to an orange end-point.

3 CALCULATION

$$\text{Acidity, (as sulfuric acid), per cent by mass} = \frac{12.25 \times VC}{m}$$

where,

V is the volume, in milliliters, of the sodium hydroxide solution;
c is the concentration in mol/1, of the sodium hydroxide solution; and
m is the mass, in grams, of the sample.

SECTION 4 : DETERMINATION OF RESIDUE ON SIEVE

1 REAGENTS

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

2 APPARATUS

2.1 *Test sieve,* 45 um conforming to CS 124.

2.2 *Sintered glass crucible,* porocity p 40.

2.3 *Oven,* maintained at 105 ± 2 °C.

3 PROCEDURE

Weigh, to the nearest milligram, about 100 g of the sample. Disperse the test portion in 600 ml of water. Pour the dispersion through the sieve (2.1) with the aid of a wash bottle, rinse the beaker and pour all the rinsings through the sieve. Wash with water until the washings passing through the sieve are clear and free of material. Carry out washing process within period of 5 min.

Transfer the residue into the weighed sintered glass crucible (2.2) and dry in the oven at 105 ± 2 °C (2.3). Allow to cool in a desiccator and weigh. Repeat the process of heating, cooling and weighing at 30 minute intervals until the difference between two successive weighings does not exceed 0.1 g. Record the lowest mass.

4 CALCULATION

$$\text{Residue on sieve, per cent by mass} = \frac{m_1}{m_0} \times 100$$

where,

m_0 is the mass, in grammes of the test sample;
m_1 is the mass, in grammes of the residue.

SECTION 5 : DETERMINATION OF LEAD, CADMIUM, COPPER MANGANESE AND IRON

1 REAGENTS

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

1.1 *Hydrochloric acid,* 36 per cent (m/m), (rel. den = 1.19).

1.2 *Nitric acid,* 65 per cent (m/m), (rel. den = 1.42).

1.3 *Standard solution for metals,* prepared standard solutions from pure metals, metalic compounds or manufactured standard solution. The usual concentration of solutions is 1.000 ug of the metal per cubic centimetre of solution. Suitable dilutions with water will bring these solutions into the linear working range of the instrument.

2 APPARATUS

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

3 PROCEDURE

3.1 Preparation of solutions

3.1.1 *Test solution*

Weigh, to the nearest milligram 0.2 to 10 g of the sample into 150 ml-beaker. Add enough water to make thick "slurry" and add hydrochloric acid (1.1) till the sample is completely dissolved. (see note 1). Boil the solution and add carefully 2 ml of nitric acid (1.2) continue heating for 5 min. Cool to the room temperature, transfer the solution to a 250-ml volumetric flask, dilute the solution up to the mark (see Note 2).

NOTE 1

Failure to observe this sequence will result in a hard cake of zinc oxide, which will be difficult to dissolve in the water acid mixture.

NOTE 2

The amount of the test portion to be measured and the final volume of the solution is left to the discretion of the analyst and must be determined by prior knowledge of the approximate amount of lead, copper, cadmium, manganese and iron in the type of ZnO being analysed.

3.1.2 *Blank solution*

Prepare the blank solution omitting the sample.

3.1.3 *Dilute test solution and blank solutions*

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

3.2 Measurement

3.2.1 *Lead*

Set the spectrophotometer (2) at a wave length of 283.3 nm using an oxidizing air-acetylene flame. Aspirate successively in triplicate the lead standard solutions (1.3), the dilute test solution 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 concentrations of lead. Determine the concentration of lead in the test solution and the blank solution with reference to the calibration curve.

3.2.2 *Cadmium*

Carry out the measurements as 3.2.1 at a wave length 228.8 nm using an oxidizing air-acetylene flame. Plot calibration curve using the median absorbances of the standard solution against concentrations of cadmium. Determine the concentration of cadmium in the test solution and the blank solution by reference to the calibration curve.

3.2.3 *Copper*

Carry out the measurements as 3.2.1 at a wave length 324.4 nm using an oxidizing air-acetylene flame. Plot calibration curve using the median absorbances of the standard solution against concentrations of copper. Determine the concentration of copper in the test solution and the blank solution by reference to the calibration curve.

3.2.4 *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 concentrations of manganese. Determine the concentration of manganese in the test solution and the blank solution by reference to the calibration curve.

3.2.5 *Iron*

Carry out the measurements as 3.2.1 at a wave length 248.3 nm using stoichiometry air-acetulene flame. Plot calibration curve using the median absorbances of the standard solution against concentrations of iron. Determine the concentration of iron in the test solution and the blank solution by reference to the calibration curve.

SECTION 6 : DETERMINATION OF LOSS ON IGNITION

1 APPARATUS

Muffle furnace, maintained at 800 °C.

2 PROCEDURE

Weigh, to the nearest milligram, about 3 g of the sample dried at 105 ± 2 °C, into a previously ignited porcelain crucible. Ignite in furnace (1) at temperature 800 ± 5 °C. Cool the crucible to the room temperature in a desiccator and weigh. Repeat the process of heating, cooling and weighing at 30 minute intervals until the difference between two successive weighings does not exceed 0.1 g. Record the lowest mass.

3 CALCULATION

$$\text{Loss on ignition, percent by mass} = \frac{m_0 - m_1}{m_0} \times 100$$

where,

m_0 is the mass, in grammes, of the test sample; and
m_1 is the mass, in grammes, of the test sample after ignition.

SECTION 7 : DETERMINATION OF ZINC OXIDE

1 REAGENTS

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

1.1 *Nitric acid,* 65 percent (m/m), (rel. den = 1.4).

1.2 *Zinc metal,* refined having a purity of 99.995, percent (m/m).

1.3 *Nitric acid,* 30 percent (m/m).

1.4 *Ferric ammonium sulfate,* 86.0 g/l solution.

1.5 *Hydrogen peroxide,* 3 per cent (m/m) solution.

1.6 *Ammonium chloride,* 250 g/l solution.

1.7 *Ammonia solution,* 25 percent (m/m), (rel. den = 0.91).

1.8 *Bromothymolblue solution,* dissolve 0.1 g of bromothymolblue in 100-ml of 95 percent (V/V) ethyl alcohol.

1.9 *Hydrochloric acid,* 20 percent (m/m), (rel. den = 1.1).

1.10 *Masking solution*, dissolve 30 g of ammonium fluoride, 100 g of ammonium thiosulfate and 250 g of ammonium acetate and dilute to 1 litre.

1.11 *Xylenolorange solution*, dissolve 0.2 g of xylenolorange tetra sodium salt in 100 ml of water.

1.12 *EDTA standard solution,* c(EDTA) = 0.1 mol/l

Dissolve 37.225 g of ethylenedinitrilotetra acetic acid disodium salt in water in a 1000-ml volumetric flask and dilute upto the mark.

2 PROCEDURE

2.1 Preparation of test solution

Weigh, to the nearest milligram, about 20 g of the sample, add 100 ml of distilled water and dissolved carefully with approximately 90 ml of nitric acid (1.1). When the zinc oxide is dissolved, boil for a short time, cool, to the room temperature and transfer into 1000-ml volumetric flask and dilute upto the mark. Add four drops of bromothymolblue solution (1.8) and neutralize with hydrochloric acid (1.9). When the colour changes from blue to light yellow add two more drops of hydrochloric acid (1.9), add 20 ml of masking solution (1.10) and seven drops of xylenolorange solution (1.11).

2.2 Standardization of EDTA solution

Weigh, to the nearest milligram 20 g of zinc (1.2) and dissolve with nitric acid (1.3) in a beaker cool to the room temperature transfer into 1000-ml volumetric flask and dilute upto the mark. Pipette 50 ml of this solution and add 10 ml of ferric ammonium sulfate (1.4) solution. Shake the solution and add 5 ml of hydrogen peroxide solution (1.5), 60 ml ammonium chloride solution (1.6) and 30 ml of ammonia solution (1.7) cool the solution shaking. After dilute to 250 ml, filter through a dry folded filter paper. Transfer the solution into a 600-ml beaker and dilute to 300 ml with water. Titrate with EDTA solution (1.12) until the colour changes from purple red to orange yellow.
Titrate two more aliquots and calculate the average volume of EDTA solution required for the titration.

NOTE

Lead and iron are precipitated as hydroxide by adding the ammonia solution. In the presence of ammonium thiosulfate and ammonium fluoride copper and aluminium are not titrated with EDTA. Even though cadmium is titrated with EDTA the amount of cadmium found is usually lower than 0.1 percent in the rubber grade zinc oxide. This error is negligible.

2.3 Determination of zinc oxide

Pipette 50 ml of the test solution prepared in 2.1 and titrate with EDTA solution (1.12) following the procedure given in 2.2.

3 CALCULATION

$$\text{Zinc oxide, per cent by mass} = 1.245 \times \frac{V_1}{V_2} \times \frac{m_1}{m_0} \times 100$$

where,

V_1 is the volume, in millilitres of EDTA solution required for the titration with the test solution ; and
V_2 is the volume, in millilitres of EDTA solution required for the standardization.
m_0 is the mass in grammes of the sample ; and
m_1 is the mass in grammes of the zinc metal (1.2).

SRI LANKA STANDARDS INSTITUTION

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