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Rubber products — Determination of zinc content — EDTA titrimetric method

Produits en caoutchouc — Dosage du zinc — Méthode titrimétrique à l'EDTA

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

Australia	Netherlands	Switzerland
Belgium	Poland	Thailand
Bulgaria	Portugal	Turkey
France	Romania	United Kingdom
Germany	South Africa, Rep. of	U.S.A.
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No Member Body expressed disapproval of the document.

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Rubber products — Determination of zinc content — EDTA titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an EDTA titrimetric method for the determination of the zinc content of all rubber products.

Lead, magnesium, iron, titanium, antimony, silica, and silicates in the ash do not interfere. The method is not applicable, however, if cobalt is present.

2 REFERENCE

ISO 247, *Rubber — Determination of ash in raw rubbers, compounded rubbers and vulcanizates.*[1]

3 PRINCIPLE

Incineration of a test portion and dissolution of the ash in hydrochloric acid. Extraction of silica by treatment with hydrofluoric and sulphuric acids. Addition of aluminium chloride and aluminium fluoride to precipitate calcium and magnesium as hexafluoroaluminates. Fluoride complexes iron, titanium and excess aluminium (interference from large amounts of iron is further reduced by addition of 2,4-pentanedione). Titration of the zinc with an EDTA (*di*sodium salt) standard volumetric solution in the presence of dithizone as indicator.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity. All recognized health and safety precautions must be observed in the handling of chemicals listed and in performing the analysis.

4.1 Acetone.

4.2 2,4-Pentanedione, 10 % (*V*/*V*) solution in the acetone (4.1).

4.3 Hydrochloric acid (ρ 1,18 g/ml).

4.4 Sulphuric acid (ρ 1,84 g/ml).

4.5 Hydrofluoric acid, 48 % (*m*/*m*) solution.

4.6 Ammonium hydroxide solution (ρ 0,91 g/ml).

4.7 Buffer solution.

Dissolve 60 g of acetic acid (CH_3COOH) and 77 g of ammonium acetate (CH_3COONH_4) in water and dilute to 1 000 ml[2] with water.

4.8 Aluminium chloride, 0,1 M solution.

Dissolve 2,42 g of aluminium chloride hexahydrate ($AlCl_3.6H_2O$) in water and dilute to 100 ml with water.

4.9 Magnesium chloride, 0,1 M solution.

Dissolve 2,03 g of magnesium chloride hexahydrate ($MgCl_2.6H_2O$) in water and dilute to 100 ml with water.

4.10 Ammonium fluoride, 3 M solution.

Dissolve 55,5 g of ammonium fluoride (NH_4F) in water and dilute to 500 ml with water. Store in a polyethylene or wax-coated bottle.

4.11 EDTA (*di*sodium salt), 0,01 M standard volumetric solution.

4.11.1 *Preparation*

Dissolve 3,72 g of (ethylenedinitrilo)tetraacetic acid *di*sodium salt, dihydrate (EDTA *di*sodium salt) in water and dilute to 1 000 ml with water.

4.11.2 *Standardization*

Pipette 25 ml of the standard zinc chloride solution (4.12) into a 250 ml conical flask. Add 5 ml of the hydrochloric acid (4.3) and proceed according to 6.3 beginning with "add 2 ml of the aluminium chloride solution". Use the burette (5.3) for titration.

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1) At present at the stage of draft. (Revision of ISO/R 247.)

2) The term millilitre (ml) is commonly used for the cubic centimetre (cm^3), particularly to denote the capacity of laboratory glassware. Apparatus with either type of marking is satisfactory to use with this International Standard.

4.11.3 *Standardization factor*

The standardization factor, T, of the EDTA (*di*sodium salt) solution, expressed as grams of zinc oxide (ZnO) per millilitre, is given by the formula

$$T = \frac{m_1}{40\ V_1}$$

where

m_1 is the mass, in grams, of dried zinc oxide used in the preparation of the standard zinc chloride solution (4.12);

V_1 is the volume, in millilitres, of EDTA (*di*sodium salt) solution used in the titration of the standard zinc chloride solution (4.12).

4.12 Zinc chloride, standard solution.

Calcine zinc oxide in a porcelain crucible for 2 h in the furnace (5.1), maintained at 550 ± 25 °C, and cool in a desiccator. Dissolve about 1,0 g of the dried reagent, weighed to the nearest 0,000 1 g, in 50 ml of water and 20 ml of the hydrochloric acid (4.3). Transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

4.13 Dithizone indicator.

Dissolve 0,01 g of dithizone [(phenylazo)thioformic acid, 2-phenylhydrazide] in 10 ml of the acetone (4.1). Prepare a fresh solution every 48 h.

4.14 Universal indicator paper.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Muffle furnace, capable of being controlled at 550 ± 25 °C.

5.2 Burette, of capacity 10 ml, graduated in 0,02 ml divisions.

5.3 Burette, of capacity 50 ml, graduated in 0,1 ml divisions.

5.4 Platinum crucibles, of capacity 50 ml.

6 PROCEDURE

6.1 Weigh, to the nearest 0,000 1 g, approximately 1 g of the sample. Place this test portion in one of the platinum crucibles (5.4) and ash according to method C specified in ISO 247.

Cool the crucible and add approximately 50 ml of the hydrochloric acid (4.3). Transfer the contents of the crucible to a 250 ml beaker with approximately 50 ml of water. Break up any large cakes of ash with a glass stirring rod. If any insoluble residue is present after cooling, proceed in accordance with 6.2. If no insoluble material is present, proceed in accordance with 6.3.

6.2 Filter the residue through an ashless filter paper. Retain the filtrate. Place the insoluble residue and the filter paper into a second platinum crucible (5.4), add 2 ml of the sulphuric acid (4.4) and ash in the muffle furnace (5.1), maintained at 550 ± 25 °C.

Moisten the residue with 5 to 10 drops of the sulphuric acid (4.4) and 5 ml of the hydrofluoric acid solution (4.5). Evaporate the hydrofluoric acid and stop heating as soon as the evolution of white fumes indicates sulphuric acid decomposition. When cool, add an additional 5 to 10 drops of the sulphuric acid and 5 ml of the hydrofluoric acid solution. Repeat the evaporation of the hydrochloric acid and add 1 ml of the sulphuric acid and 5 ml of the hydrofluoric acid solution to the wet residue. Evaporate the hydrofluoric acid and stop heating as soon as white fumes appear.

Pour the contents of the crucible carefully into the retained filtrate, wash the crucible with distilled water and add the washings to the filtrate. Proceed according to 6.3.

6.3 If necessary, evaporate the solution or filtrate to a volume of approximately 50 ml. Transfer the cooled solution to a 100 ml volumetric flask and make up to the mark with distilled water. Select an aliquot portion from the following table according to the expected zinc content and transfer to a 250 ml conical flask.

ZnO expected	Aliquot portion	Capacity of burette to be used
% (*m*/*m*)	ml	ml
0 to 3	25	10 (5.2)
3 to 8	10	10 (5.2)
more than 8	10	50 (5.3)

If necessary, dilute the aliquot portion to 25 ml, add 2 ml of the aluminium chloride solution (4.8), 5 ml of the magnesium chloride solution (4.9), and 10 ml of the ammonium fluoride solution (4.10).

Add ammonium hydroxide solution (4.6) until alkaline to the universal indicator paper (4.14). Acidity with approximately 1 ml of the sulphuric acid (4.4). Bring the solution to the boil, and then cool to room temperature. Add ammonium hydroxide solution (4.6) until just alkaline. Then add an additional 0,5 ml. Add 10 ml of the buffer solution (4.7), 60 ml of the acetone (4.1), 5 ml of the 2,4-pentanedione solution (4.2) and 5 drops of the dithizone indicator solution (4.13). Cool the solution in an ice bath.

6.4 Titrate with the EDTA (*di*sodium salt) standard volumetric solution (4.11), using the appropriate burette indicated in the table. The end-point is reached at a yellow-green colour, which does not change on the addition of a further drop of the EDTA (*di*sodium salt) standard volumetric solution.

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7 EXPRESSION OF RESULTS

The zinc content of the test portion, expressed as a percentage by mass of zinc oxide (ZnO), is given by the formula

$$\frac{T \times V_2 \times 100 \times 100}{V_3 \times m_2}$$

where

T is the standardization factor as calculated in 4.11.3;

V_2 is the volume, in millilitres, of the EDTA (*di*sodium salt) standard volumetric solution (4.11) used in the titration of the aliquot portion of the test solution;

V_3 is the volume, in millilitres, of the aliquot portion;

m_2 is the mass, in grams, of the test portion.

8 TEST REPORT

The test report shall include the following particulars :

a) a reference to this International Standard;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.

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