INTERNATIONAL STANDARD

ISO 2454

Third edition
1995-04-01

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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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2454 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 2454:1982), of which it constitutes a minor revision.

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Rubber products — Determination of zinc content — EDTA titrimetric method

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WARNING — Persons using this International Standard shall be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies a titrimetric method using ethylenedinitrilotetraacetic acid (EDTA) disodium salt for the determination of the zinc content of all rubber products.

The presence of lead, magnesium, iron, titanium, antimony, silica and silicates in the ash does not interfere with the determination. The method is not applicable, however, if cobalt is present.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 247:1990, *Rubber — Determination of ash.*

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 9028:1989, *Rubber — Dissolution by acid digestion.*

3 Principle

A test piece is incinerated and the ash dissolved in hydrochloric acid. Silica is extracted by treatment with hydrofluoric and sulfuric acids. Aluminium chloride and aluminium fluoride are added to precipitate calcium and magnesium as hexafluoroaluminates. Interference from iron, titanium and excess aluminium is removed or reduced by the formation of complexes with fluoride ion (interference from large amounts of iron is further reduced by addition of 2,4-pentanedione). The zinc is titrated with a standard volumetric solution of EDTA disodium salt 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.

4.1 Acetone.

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

4.3 Hydrochloric acid, ρ = 1,18 Mg/m^3.

4.4 Sulfuric acid, ρ= 1,84 Mg/m^3.

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

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4.6 Ammonium hydroxide, ρ = 0,91 Mg/m^3 solution.

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 cm^3 with water.

4.8 Aluminium chloride,
$c(AlCl_3{\cdot}6H_2O)$ = 0,1 mol/dm^3 solution.

Dissolve 2,42 g of aluminium chloride hexahydrate ($AlCl_3{\cdot}6H_2O$) in water and dilute to 100 cm^3 with water.

4.9 Magnesium chloride,
$c(MgCl_2{\cdot}6H_2O)$ = 0,1 mol/dm^3 solution.

Dissolve 2,03 g of magnesium chloride hexahydrate ($MgCl_2{\cdot}6H_2O$) in water and dilute to 100 cm^3 with water.

4.10 Ammonium fluoride,
$c(NH_4F)$ = 3 mol/dm^3 solution.

Dissolve 55,5 g of ammonium fluoride (NH_4F) in water and dilute to 500 cm^3 with water.

Store in a polyethylene or wax-coated bottle.

4.11 Zinc chloride, standard reference solution corresponding to 1 g of ZnO per cubic decimetre.

Calcine zinc oxide in a porcelain crucible for 2 h in the furnace (5.1), maintained at 550 °C ± 25 °C, and cool in a desiccator. Weigh, to the nearest 0,1 mg, about 1 g of the dried reagent and dissolve in a mixture of 50 cm^3 water and 20 cm^3 hydrochloric acid (4.3). Transfer to a 1 000 cm^3 volumetric flask and dilute to the mark with water.

1 cm^3 of this standard reference solution contains 1 mg of ZnO.

4.12 EDTA disodium salt [ethylenedinitrilotetraacetic acid disodium salt] ,dihydrate, standard volumetric solution,
$c(C_{10}H_{14}N_2O_8Na_2{\cdot}2H_2O)$ = 0,01 mol/dm^3.

4.12.1 Preparation

Dissolve 3,72 g of EDTA disodium salt in water and dilute to 1 000 cm^3 with water.

4.12.2 Standardization

Pipette 25 cm^3 of zinc chloride standard reference solution (4.11) into a 250 cm^3 conical flask. Add 5 cm^3 of hydrochloric acid (4.3) and proceed in accordance with 6.3 beginning with "...add 2 cm^3 of aluminium chloride solution". Carry out the titration as described in 6.4 , using the 50 cm^3 burette (5.3).

4.12.3 Standardization factor

The standardization factor T of the EDTA disodium salt solution, expressed as grams of zinc oxide (ZnO) per cubic centimetre, is given by the equation

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

where

m_1 is the mass, in grams, of dried zinc oxide used in the preparation of the zinc chloride standard reference solution (4.11);

V_1 is the volume, in cubic centimetres, of EDTA disodium salt solution used in the titration of the zinc chloride standard reference solution (4.11).

4.13 Dithizone indicator.

Dissolve 0,01 g of dithizone [1,5-diphenylthiocarbazone] in 10 cm^3 of acetone (4.1).

Prepare a fresh solution every 48 h.

4.14 Methyl orange indicator paper.

5 Apparatus

Ordinary laboratory apparatus, plus the following:

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

5.2 Burette, of capacity 10 cm^3, graduated in 0,02 cm^3 divisions, conforming with the requirements of ISO 385-1, class A.

5.3 Burette, of capacity 50 cm^3, graduated in 0,1 cm^3 divisions, conforming with the requirements of ISO 385-1, class A.

5.4 Platinum crucibles, of capacity 50 cm^3.

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6 Procedure

6.1 Weigh, to the nearest 0,1 mg, approximately 1 g of the test sample. Place this test piece in one of the platinum crucibles (5.4) and ash in accordance with method A of ISO 247:1990. If halogenated rubbers are present, use method A of ISO 9028:1989. Cool the crucible and add approximately 50 cm^3 of hydrochloric acid (4.3). Transfer the contents of the crucible to a 250 cm^3 beaker with approximately 50 cm^3 of water. Break up any large cakes of ash with a glass stirring rod. If any insoluble residue is present after cooling, proceed in acordance 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 in a second platinum crucible (5.4), add 2 cm^3 of sulfuric acid (4.4) then heat over a gas burner to volatilize the excess sulfuric acid. Transfer the crucible and its contents to the muffle furnace (5.1), maintained at 550 °C ± 25 °C, and heat until all the carbon is completely oxidized and a clean ash is obtained.

Moisten the residue with 5 to 10 drops of sulfuric acid (4.4) and add 5 cm^3 of hydrofluoric acid solution (4.5) in a fume cupboard. Evaporate the hydrofluoric acid and stop heating as soon as the evolution of white fumes indicates sulfuric acid decomposition. When cool, add an additional 5 to 10 drops of sulfuric acid and 5 cm^3 of hydrofluoric acid solution. Repeat the evaporation of hydrofluoric acid and add 1 cm^3 of sulfuric acid and 5 cm^3 of 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 into the retained filtrate, wash the crucible with distilled water and add the washings to the filtrate.

6.3 If necessary, evaporate the solution or filtrate to a volume of approximately 50 cm^3. Transfer the cooled solution to a 100 cm^3 volumetric flask and make up to the mark with water. Select an aliquot portion from table 1 according to the expected zinc content and transfer to a 250 cm^3 conical flask.

If necessary, dilute the aliquot portion to 25 cm^3 and add 2 cm^3 of aluminium chloride solution (4.8), 5 cm^3 of magnesium chloride solution (4.9) and 10 cm^3 of ammonium fluoride solution (4.10).

Table 1 — Aliquot portions

ZnO content expected % (*m/m*)	Aliquot portion cm^3	Capacity of burette to be used cm^3
⩽ 3	25	10 (5.2)
> 3 but ⩽ 8	10	10 (5.2)
> 8	10	50 (5.3)

Add ammonium hydroxide solution (4.6) until the solution is alkaline to methyl orange indicator paper (4.14). Acidify with approximately 1 cm^3 of sulfuric 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 cm^3. Add 10 cm^3 of buffer solution (4.7), 60 cm^3 of acetone (4.1), 5 cm^3 of 2,4-pentanedione solution (4.2) and 5 drops of dithizone indicator solution (4.13). Cool the solution in an ice-bath.

6.4 Titrate with EDTA disodium salt standard volumetric solution (4.12), using the appropriate burette indicated in table 1. The end-point is reached at a yellowish green colour, which does not change on the addition of a further drop of the EDTA disodium salt standard volumetric solution.

7 Expression of results

Calculate the zinc content, expressed as a percentage by mass of zinc oxide (ZnO), from 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 4.12.3;

V_2 is the volume, in cubic centimetres, of EDTA disodium salt standard volumetric solution (4.12) used in the titration of the aliquot portion of the test solution;

V_3 is the volume, in cubic centimetres, of the aliquot portion;

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

8 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the product tested;

c) the method of ashing used;

d) the results and the units in which they are expressed;

e) any unusual features noted during the determination;

f) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;

g) the date of the test.

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