
International Standard



2454

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber products — Determination of zinc content — EDTA titrimetric method

Produits en caoutchouc — Détermination de la teneur en zinc — Méthode titrimétrique à l'EDTA

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Descriptors : rubber, vulcanized rubber, chemical analysis, determination of content, zinc, volumetric analysis.

Rubber products — Determination of zinc content — EDTA titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method using ethylenedinitrilotetraacetic (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 References

ISO 247, *Rubber — Determination of ash.*

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*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. Removal or reduction of interference from iron, titanium and excess aluminium by the formation of complexes with fluoride ion (interference from large amounts of iron is further reduced by addition of 2,4-pentanedione). Titration of the zinc with a standard volumetric solution of EDTA disodium salt in the presence of dithizone as indicator.

4 Reagents

All recognized health and safety precautions shall be observed in the handling of chemicals listed and in performing the analysis.

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, $\rho = 1,18 \text{ Mg/m}^3$.

4.4 Sulphuric acid, $\rho = 1,84 \text{ Mg/m}^3$.

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

4.6 Ammonium hydroxide, $\rho = 0,91 \text{ Mg/m}^3$ solution.

4.7 Buffer solution.

Dissolve 60 g of acetic acid (CH_3COOH) and 77 g of ammonium acetate ($\text{CH}_3\text{COONH}_4$) in water and dilute to 1 000 cm^3 ²⁾ with water.

4.8 Aluminium chloride, $c(\text{AlCl}_3 \cdot 6\text{H}_2\text{O}) = 0,1 \text{ mol/dm}^3$ solution.

Dissolve 2,42 g of aluminium chloride hexahydrate ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) in water and dilute to 100 cm^3 with water.

4.9 Magnesium chloride, $c(\text{MgCl}_2 \cdot 6\text{H}_2\text{O}) = 0,1 \text{ mol/dm}^3$ solution.

Dissolve 2,03 g of magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in water and dilute to 100 cm^3 with water.

4.10 Ammonium fluoride, $c(\text{NH}_4\text{F}) = 3 \text{ mol/dm}^3$ 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, standard reference solution corresponding to 1 g of ZnO per cubic decimetre.

1) At present at the stage of draft. (Partial revision of ISO/R 385-1964.)

2) The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the 12th Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes. Glassware with either marking is satisfactory for use with the procedure described in this International Standard.

If necessary, dilute the aliquot portion to 25 cm³, add 2 cm³ of aluminium chloride solution (4.8), 5 cm³ of magnesium chloride solution (4.9), and 10 cm³ of ammonium fluoride solution (4.10).

Add ammonium hydroxide solution (4.6) until alkaline to universal indicator paper (4.14). Acidify with approximately 1 cm³ of 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 cm³. Add 10 cm³ of buffer solution (4.7), 60 cm³ acetone (4.1), 5 cm³ 2,4-pentanedione solution (4.2) and 5 drops dithizone indicator solution (4.13). cool the solution on an ice-bath.

6.4 Titrate with the EDTA disodium salt standard volumetric solution (4.12), using the appropriate burette indicated in the table. 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

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 4.12.3;

V_2 is the volume, in cubic centimetres, of the 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 portion.

8 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) the type and identification of the product tested,
- c) the results and the method of expression used;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

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