INTERNATIONAL STANDARD

ISO 9298

Second edition
2017-03

Rubber compounding ingredients — Zinc oxide — Test methods

Ingrédients de mélange du caoutchouc — Oxyde de zinc — Méthodes d'essai

Reference number
ISO 9298:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 9298:1995), which has been technically revised. The main change is the reference to ISO 18852 as the method for the nitrogen adsorption to determine the surface area.

Rubber compounding ingredients — Zinc oxide — Test methods

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 document specifies the methods to be used for the evaluation of zinc oxide for use in the rubber industry.

The analytical methods are applicable to all commercial zinc oxides, for example:

— direct type (American process);

— indirect type (French process);

— other types produced by different chemical methods, i.e. precipitation and calcination.

Zinc oxide can also be coated with organic materials, such as fatty acids, oil, wetting agents, etc., in order to improve the dispersion in rubber.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C*

ISO 787-4, *General methods of test for pigments and extenders — Part 4: Determination of acidity or alkalinity of the aqueous extract*

ISO 787-7, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure*

ISO 787-8, *General methods of test for pigments and extenders — Part 8: Determination of matter soluble in water — Cold extraction method*

ISO 1124, *Rubber compounding ingredients — Carbon black shipment sampling procedures*

ISO 18852, *Rubber compounding ingredients — Determination of multipoint nitrogen surface area (NSA) and statistical thickness surface area (STSA)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Sampling

Sampling shall be carried out in accordance with ISO 1124 for dry powders.

5 Methods of test for the determination of physical and chemical properties

5.1 General

Surface-coated zinc oxides shall be evaluated by the methods specified in Table 1.

This evaluation shall be done without prior calcination or extraction, since there is little purpose in determining volatile-matter content, content of water-soluble matter or acidity if the coating is removed.

Table 1 — Methods for the evaluation of surface-coated zinc oxides

Property	Units	Test method
Matter volatile at 105 °C	% *(m/m)*	ISO 787-2
Water-soluble matter	% *(m/m)*	ISO 787-8
Acidity/alkalinity (H_2SO_4 equiv.)	g H_2SO_4/100 g	ISO 787-4
Residue on sieve	% *(m/m)*	ISO 787-7
Nitrogen-adsorption surface area	m^2/g	ISO 18852
Zinc oxide	% *(m/m)*	Annex A
Lead	% *(m/m)*	Annex B
Cadmium	% *(m/m)*	Annex B
Copper	% *(m/m)*	Annex B
Manganese	% *(m/m)*	Annex B
Acid-insoluble matter	% *(m/m)*	Annex C

5.2 Matter volatile at 105 °C

Determine the loss on heating at 105 °C in accordance with ISO 787-2.

5.3 Water-soluble matter

Determine the percentage of water-soluble matter in accordance with ISO 787-8.

5.4 Acidity/alkalinity

Determine the acidity/alkalinity, in cm^3 of 0,1 mol/dm^3 standard volumetric solution per 100 g of sample, in accordance with ISO 787-4. The result shall be expressed in grams of sulfuric acid per 100 g (g H_2SO_4/100 g) by multiplying the result by $4{,}9 \times 10^{-3}$.

5.5 Residue on sieve

Determine the sieve residue in accordance with ISO 787-7.

5.6 Nitrogen-adsorption surface area

Determine the surface area in accordance with ISO 18852. The test portion shall be 0,7 g to 1,0 g, or more if indicated by the initial test or past experience.

6 Classification and typical values of zinc oxides

Three types of zinc oxide are used in the rubber industry and are described in Annex D.

Typical values for some of the various types of zinc oxide are given in Annex D.

7 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 9298;

b) all details necessary for the identification of the sample;

c) the zinc oxide content of the sample;

d) the lead, cadmium, copper and manganese contents of the sample;

e) the water-soluble matter content of the sample;

f) the acidity/alkalinity of the sample;

g) the residue on sieve;

h) the surface area by nitrogen adsorption;

i) the acid-insoluble matter;

j) the dates of the tests;

k) details of any deviation from the procedures specified in this document.

Annex A
(normative)

Determination of zinc oxide content

A.1 Reagents

Use only reagents of recognized analytical grade and distilled, deionized or distilled/deionized water for sample preparation and required dilutions.

A.1.1 Nitric acid, 65 % (*m/m*), $d \approx 1{,}4$ Mg/m^3.

A.1.2 Hydrochloric acid, 20 % (*m/m*), $d \approx 1{,}1$ Mg/m^3.

A.1.3 Ammonia solution, 25 % (*m/m*), $d \approx 0{,}91$ Mg/m^3.

A.1.4 Hydrogen peroxide solution, 3 % (*m/m*).

A.1.5 Iron(III) solution.

Dissolve 86 g of iron(III) ammonium sulfate in water and dilute to 1 000 cm^3.

A.1.6 Ammonium chloride solution.

Dissolve 250 g of ammonium chloride in water and dilute to 1 000 cm^3.

A.1.7 Masking solution.

Dissolve 30 g of ammonium fluoride, 100 g of ammonium thiosulfate and 250 g of ammonium acetate in water and dilute to 1 000 cm^3.

A.1.8 Bromothymol blue solution.

Dissolve 0,1 g of bromothymol blue in 100 cm^3 of ethanol.

A.1.9 Xylenol orange solution.

Dissolve 0,2 g of xylenol orange, tetrasodium salt, in 100 cm^3 of water.

A.1.10 EDTA, standard volumetric solution, c(EDTA) = 0,1 mol/dm^3.

Dissolve 37,225 g of ethylenedinitrilotetraacetic acid, disodium salt (Na_2EDTA), in water in a 1 000 cm^3 one-mark volumetric flask, dilute to the mark and mix well. Alternatively, commercially available standard solutions may be used.

A.1.11 Zinc metal, of minimum purity 99,995 % (*m/m*).

A.2 Apparatus

The usual laboratory apparatus and, in particular, the following.

A.2.1 **Volumetric flasks**, class A, of capacity 250 cm^3, 500 cm^3 and 1 000 cm^3.

A.2.2 **Pipettes**, class A, of capacity 50 cm^3 and 100 cm^3.

A.2.3 **Burette**, class A, of capacity 50 cm^3.

A.2.4 **Balance**, of capacity 250 g, weighing to an accuracy of at least ±1 mg.

A.2.5 **Heating device**, e.g. a hotplate.

A.2.6 **Filter paper**, acid-washed and fluted.

A.2.7 **Beakers**, of capacity 600 cm^3 and 1 000 cm^3.

A.2.8 **Conical flasks**, of capacity 500 cm^3 and 1 000 cm^3.

A.3 Sampling

Take a representative sample in accordance with ISO 1124.

A.4 Procedure

A.4.1 Suspend 20 g of the zinc oxide sample, weighed to ±0,01 g, in 100 cm^3 of water in a 1 000 cm^3 beaker (A.2.7) and dissolve carefully with approximately 90 cm^3 of nitric acid (A.1.1). When the zinc oxide has dissolved, boil for a short time, cool down the solution and transfer it to a 500 cm^3 volumetric flask (A.2.1). Carefully dilute the solution to the mark with water and shake.

A.4.2 Pipette 50 cm^3 of this solution into a 250 cm^3 volumetric flask (A.2.1). Add 10 cm^3 of iron(III) solution (A.1.5). Shake and then add successively 5 cm^3 of hydrogen peroxide solution (A.1.4), 60 cm^3 of ammonium chloride solution (A.1.6) and 30 cm^3 of ammonia solution (A.1.3).

A.4.3 Shake briefly and cool down. Make up to the mark and filter through a dry folded filter paper (A.2.6) into a dry 500 cm^3 conical flask (A.2.8). Pipette 50 cm^3 of this solution into a 600 cm^3 beaker and dilute with water to about 300 cm^3.

A.4.4 Add four drops of bromothymol blue solution (A.1.8) and neutralize with hydrochloric acid (A.1.2). The colour changes from blue to light yellow. Add two drops of hydrochloric acid in excess. After addition of 20 ml of masking solution (A.1.7) and seven drops of xylenol orange solution (A.1.9), titrate with EDTA solution (A.1.10) until the colour changes from purple-red to orange-yellow.

A.4.5 After further dropwise addition of 0,5 cm^3 to 1 cm^3 of EDTA solution, the colour changes sharply to pale yellowish-green. Let the total volume of EDTA solution added be V_1.

A.5 Standardization procedure

Dilute concentrated nitric acid to a concentration of approximately 30 % (*m/m*), $d \approx 1{,}2$ Mg/m^3.

WARNING — Acid should be added carefully to water.

Then dissolve 20 g of refined zinc (A.1.11), weighed to ±0,01 g, by heating in a beaker with 40 cm^3 of the diluted nitric acid. Allow the solution to cool, transfer to a 1 000 cm^3 volumetric flask and dilute to the mark. Proceed according to A.4.2 to A.4.5 to obtain the titration volume V_2.