
**Rubber compounding ingredients —
Natural calcium carbonate — Test methods**

*Ingrédients de mélange du caoutchouc — Carbonate de calcium naturel —
Méthodes d'essai*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 5796 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 5796:1990), of which it constitutes a technical revision.

Annexes A, B and C form a normative part of this International Standard. Annex D is for information only.

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Rubber compounding ingredients — Natural calcium carbonate — Test methods

WARNING — Persons using this International Standard should 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

1.1 This International Standard specifies the methods to be used for the evaluation of natural calcium carbonate (chalk or limestone) ground to a dry powder for use in the rubber industry.

NOTE 1 Classification of natural calcium carbonate according to fineness and chemical purity and typical physical and chemical properties for use in the rubber industry are given in informative annex D.

NOTE 2 This International Standard does not cover calcium carbonates prepared by precipitation from solution.

1.2 There are two sets of analytical methods listed in this International Standard. In the body of the text (4.8.2 to 4.8.4), the traditional spectrophotometric methods are given; these are obsolescent, time-consuming and use a chlorinated solvent. It is recommended that these methods be phased out and replaced by the atomic absorption methods listed in annexes A, B and C.

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2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 787-10:1993, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method.*

ISO 3262-1:1997 *Extendors for paints — Specifications and methods of test — Part 1: Introduction and general test methods.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

ISO 15528:2000, *Paints, varnishes and raw materials for paints and varnishes — Sampling.*

3 Sampling

Sampling shall be carried out in accordance with ISO 15528.

4 Methods of test

4.1 General

During the analysis, all reagents used shall be of recognized analytical reagent quality. Distilled water or water of equal purity shall be used throughout the tests.

4.2 Residue on sieve

Determine the sieve residue in accordance with ISO 3262-1, using 45 μm and 125 μm opening test sieves as defined by ISO 565.

4.3 Calcium carbonate (on dry sample)

Determine the calcium carbonate content in accordance with ISO 3262-1.

4.4 Loss on heating at 105 °C

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

4.5 Loss on ignition at 1 000 °C (on dry sample)

Determine the loss on ignition at 1 000 °C in accordance with ISO 3262-1.

4.6 Matter insoluble in hydrochloric acid

4.6.1 Reagent

4.6.1.1 Hydrochloric acid, 73 g/dm³ solution.

Dilute 170 cm³ of concentrated (35 % by mass) hydrochloric acid ($\rho = 1,18 \text{ g/cm}^3$) to 1 dm³ with water and mix.

4.6.2 Apparatus

4.6.2.1 **Beaker**, of capacity 250 cm³, **watch glass**, suitable for covering the beaker, and **glass rod** suitable for stirring.

4.6.2.2 **Analytical balance**, accurate to 0,1 mg.

4.6.2.3 **Sintered-glass crucible**, porosity grade P 40, in conformity with the requirements of ISO 4793.

4.6.2.4 **Oven**, capable of being maintained at a temperature of 105 °C \pm 2 °C.

4.6.2.5 **Desiccator**.

4.6.3 Procedure

4.6.3.1 Weigh, to the nearest 1 mg, approximately 2 g of sample into the beaker (4.6.2.1).

4.6.3.2 Pour 100 cm³ of hydrochloric acid (4.6.1.1) into the beaker and cover with a watch-glass.

4.6.3.3 Swirl the mixture gently at room temperature, avoiding the formation of foam. Boil the mixture gently for 5 min to 10 min, then leave to cool for 30 min to 60 min, stirring from time to time.

4.6.3.4 Filter off the insoluble matter through the crucible (4.6.2.3) which has previously been washed, dried at 105 °C and weighed. Wash with water until the washings are free from chloride. Discard the filtrate and the washings.

4.6.3.5 Dry the crucible containing the insoluble residue in the oven (4.6.2.4), maintained at 105 °C ± 2 °C, until constant mass is achieved, i.e. until further oven-drying, cooling and weighing yields a mass change of less than 1 mg.

4.6.3.6 Cool in the desiccator (4.6.2.5).

4.6.3.7 Weigh to the nearest 1 mg.

4.6.4 Expression of results

The matter insoluble in hydrochloric acid, M_{is} , is given, as a percentage by mass, by the following equation:

$$M_{is} = \frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the empty dried crucible;

m_2 is the mass, in grams, of the crucible containing the insoluble matter.

Express the result to the nearest 0,1 % by mass.

4.7 Alkalinity

4.7.1 Reagents

4.7.1.1 **Distilled water**, boiled to remove carbon dioxide.

4.7.1.2 **Phenolphthalein**, 0,5 % by mass solution in 95 % by volume ethanol.

4.7.1.3 **Hydrochloric acid**, standard volumetric solution, $c(\text{HCl}) = 0,01 \text{ mol/dm}^3$.

4.7.2 Apparatus

4.7.2.1 **Analytical balance**, accurate to 0,1 mg.

4.7.2.2 **Conical flask**, narrow-mouth, of capacity 250 cm³.

4.7.2.3 **Filter paper**, fine grade.

4.7.2.4 **Burette**, accurate to 0,1 cm³.

4.7.3 Procedure

4.7.3.1 Weigh, to the nearest 1 mg, about 10 g of sample and place this test portion in the flask (4.7.2.2). Add 150 cm³ of distilled water (4.7.1.1). Leave the mixture for 1 h, shaking it from time to time.

4.7.3.2 Filter the mixture through the filter paper (4.7.2.3) and keep all the residue on the filter. Wash three times with a minimum amount of distilled water, adding the washings to the filtrate.

4.7.3.3 Add a few drops of phenolphthalein solution (4.7.1.2) as indicator to the filtrate and titrate with hydrochloric acid (4.7.1.3) until colourless. Read the volume used for the titration to the nearest 0,1 cm³.

4.7.4 Expression of results

The alkalinity, w , expressed in grams of sodium carbonate (Na₂CO₃) per 100 g of sample, is given by the following equation:

$$w = \frac{V \times c \times 0,000\ 53}{m} \times 100$$

where

V is the volume of HCl, in cubic centimetres, used for the titration;

c is the actual concentration, expressed in moles of HCl per cubic decimetre of hydrochloric acid (4.7.1.3);

m is the mass, in grams, of the test portion;

0,000 53 is the mass, in grams, of sodium carbonate corresponding to 100 cm³ of hydrochloric acid, $c(\text{HCl}) = 0,010 \text{ mol/dm}^3$

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4.8 Determination of total copper, total manganese and total iron

4.8.1 Preparation of stock test solution

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4.8.1.1 Reagents

4.8.1.1.1 Nitric acid, $\rho = 1,42 \text{ g/cm}^3$.

4.8.1.1.2 Hydrofluoric acid, 40 % by mass solution.

4.8.1.1.3 Sulfuric acid, 50 % by volume solution.

4.8.1.1.4 Octyl alcohol (octan-1-ol).

4.8.1.1.5 Hydrochloric acid, $\rho = 1,18 \text{ g/cm}^3$ (35 % by mass).

4.8.1.2 Apparatus

4.8.1.2.1 Beaker, of capacity 250 cm³.

4.8.1.2.2 Volumetric flask, of capacity 250 cm³.

4.8.1.2.3 Platinum crucible, of capacity 20 cm³.

4.8.1.2.4 Muffle furnace.

4.8.1.2.5 Fume cupboard.

4.8.1.2.6 Quartz triangle.

4.8.1.2.7 Analytical balance, accurate to 0,1 mg.

4.8.1.3 Procedure

4.8.1.3.1 Weigh, to the nearest 1 mg, about 5 g of sample into the beaker (4.8.1.2.1). Dissolve this test portion carefully in 25 cm³ of water and 15 cm³ of nitric acid (4.8.1.1.1) and heat to boiling. If foam tends to rise during the dissolution of the test portion, break the foam by adding a drop of octyl alcohol (4.8.1.1.4).

4.8.1.3.2 Filter through a medium filter paper, wash the paper with hot water and allow the combined filtrate plus washings to cool. Then transfer the solution into the volumetric flask (4.8.1.2.2) and retain for 4.8.1.3.7.

4.8.1.3.3 Lift out the filter paper, fold it carefully to enclose any residue and place in the platinum crucible (4.8.1.2.3). Heat gently until dry, then heat more to char the paper. Then place the crucible in the muffle furnace (4.8.1.2.4) at 1 000 °C ± 50 °C for 30 min to oxidize and to remove all char.

4.8.1.3.4 Cool the crucible and add 2 cm³ of hydrofluoric acid (4.8.1.1.2), drop by drop, to the crucible in such a way that all the contents are wetted. Then add 0,5 cm³ of sulfuric acid (4.8.1.1.3) drop by drop.

Place the crucible on a hotplate in the fume cupboard (4.8.1.2.5) and evaporate to dryness, taking care to avoid overheating and consequent bumping or spitting. Then continue heating until white fumes appear.

4.8.1.3.5 Transfer the crucible, still in the fume cupboard, to a quartz triangle on a stand and heat to a dull red heat until all white fumes have been driven off.

4.8.1.3.6 Allow the crucible to cool. Add 5 cm³ of water and 1 cm³ of nitric acid (4.8.1.1.1) and warm on a hotplate. Stir with a glass rod to dissolve all salts.

4.8.1.3.7 Cool and transfer the solution quantitatively to the flask containing the original acid-soluble portion (see 4.8.1.3.2), rinsing the crucible thoroughly with water into the flask.

4.8.1.3.8 Finally, dilute with water to the 250 cm³ mark.

The flask now contains the stock test solution, aliquots of which will be required for determination of copper, manganese and iron.

4.8.2 Copper (total) — Spectrophotometric method

4.8.2.1 Reagents

4.8.2.1.1 Biquinolyl reagent, solution.

Dissolve 0,03 g of 2,2'-biquinoline in 100 cm³ of *n*-hexanol (4.8.2.1.8) that has been freshly distilled from solid sodium hydroxide.

4.8.2.1.2 Hydroxylammonium chloride, solution.

Dissolve 25 g of hydroxylammonium chloride in about 80 cm³ of water. Filter, if necessary, and dilute to 100 cm³ with water.

If any appreciable amounts of copper are present in the solution, extract with successive 10 cm³ portions of a 0,01 % by mass solution of dithizone in carbon tetrachloride until there is no more violet coloration due to copper, hence no change in the green colour of the dithizone solution. Then extract the solution with carbon tetrachloride until all colour has been removed from the aqueous solution.

4.8.2.1.3 Sodium acetate buffer solution.

Dissolve 136 g of sodium acetate trihydrate in water and dilute to 1 dm³ with water. If the reagent contains more than a trace of copper, purify this solution by extraction with 0,01 % by mass dithizone solution in carbon tetrachloride as for the hydroxylammonium chloride solution (4.8.2.1.2).

4.8.2.1.4 Hydroquinone, solution.

Dissolve 1 g of hydroquinone in 100 cm³ of redistilled ethanol.

4.8.2.1.5 Copper, standard stock solution.

Dissolve exactly 0,1 g of pure copper in 3 cm³ of nitric acid (4.8.1.1.1), add 1 cm³ of sulfuric acid (4.8.1.1.3) and evaporate in the fume cupboard until white fumes appear. Allow to cool, dissolve the residue in water and dilute to 500 cm³.

1 cm³ of this solution contains 200 µg of Cu.

4.8.2.1.6 Copper, 4 µg/cm³ standard working solution.

Dilute exactly 5 cm³ of the stock solution (4.8.2.1.5) to 250 cm³ with water.

1 cm³ of this solution contains 4 µg of Cu. This solution is used for the calibration of the 1 cm spectrometer cells.

4.8.2.1.7 Copper, 1 µg/cm³ standard working solution.

For the calibration of 4 cm cells, dilute exactly 5 cm³ of the stock solution (4.8.2.1.5) to 1 000 cm³ with water, to give a solution containing 1 µg of copper per cubic centimetre.

4.8.2.1.8 *n*-hexyl alcohol (hexan-1-ol).

4.8.2.2 Apparatus

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Ordinary laboratory apparatus, plus the following:

4.8.2.2.1 Analytical balance, accurate to 0,1 mg ISO 5796:2000

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4.8.2.2.2 Spectrometer, capable of being operated at 540 nm, with 1 cm and/or 4 cm cells.

4.8.2.2.3 Separating funnels, of capacity 250 cm³.

4.8.2.2.4 One-mark volumetric flask, of capacity 10 cm³.

4.8.2.3 Procedure

4.8.2.3.1 Transfer a 100 cm³ aliquot portion of the stock test solution (see 4.8.1) to a separating funnel (4.8.2.2.3) and add 2,5 cm³ of hydroxylammonium chloride solution (4.8.2.1.2) and 25 cm³ of sodium acetate buffer solution (4.8.2.1.3).

4.8.2.3.2 Shake with 6 cm³ of biquinolyl reagent (4.8.2.1.1) for 5 min and allow the phases to separate.

4.8.2.3.3 Run the lower, aqueous, layer into another separating funnel, add 2 cm³ of hydroxylammonium chloride solution and extract again with 2,5 cm³ of biquinolyl reagent.

4.8.2.3.4 Separate the phases and again extract the aqueous layer with 2 cm³ of biquinolyl reagent.

4.8.2.3.5 Combine the three organic extracts in the one-mark volumetric flask (4.8.2.2.4) containing 0,5 cm³ of hydroquinone solution (4.8.2.1.4) and dilute the solution to volume with *n*-hexyl alcohol (4.8.2.1.8).

4.8.2.3.6 Measure the absorbance of the solution in 1 cm or 4 cm cells with the spectrometer (4.8.2.2.2) set at a wavelength of 540 nm.

4.8.2.3.7 Measure also the absorbance (at the same wavelength and in the same size cell) of a reagent blank solution prepared in the same way as the sample solution but omitting the stock solution. Subtract the absorbance of this blank solution from the absorbance found in 4.8.2.3.6.

4.8.2.3.8 Obtain the amount of copper in the aliquot portion taken in 4.8.2.3.1 by use of the calibration curve plotted in 4.8.2.4.4.

4.8.2.4 Calibration

4.8.2.4.1 Use the standard copper working solution containing 4 µg of copper per cm³ (4.8.2.1.6) to calibrate the 1 cm cell and the solution containing 1 µg of copper per cm³ (4.8.2.1.7) for the 4 cm cell, as follows:

4.8.2.4.2 Transfer aliquot portions from 0 to 25 cm³ into separating funnels (4.8.2.2.3). Add 1,5 cm³ of concentrated hydrochloric acid and dilute each solution to 100 cm³ with water.

4.8.2.4.3 Add hydroxylammonium chloride solution and buffer solution, extract the copper with biquinolyl reagent and measure the absorbances as described in 4.8.2.3.6.

4.8.2.4.4 Plot the relationship of the absorbance to the copper concentration for the range 0 to 25 µg of copper (4 cm cells) to obtain the calibration curve.

NOTE A solution containing 25 µg of copper in 10 cm³ of organic extract should have an approximate absorbance reading of 0,984 in 4 cm cells or 0,246 in 1 cm cells.

4.8.2.5 Expression of results

The total copper content in the sample (w_{Cu} , expressed in milligrams per kilogram), is given by the following equation:

$$w_{\text{Cu}} = \frac{2,5m_2}{m_1}$$

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where

m_1 is the mass in grams, of the test portion;

m_2 is the mass, in micrograms, of copper found in the aliquot portion.

4.8.3 Manganese (total) — Spectrophotometric method

4.8.3.1 Reagents

4.8.3.1.1 Phosphoric acid, solution.

To 700 cm³ of water add 200 cm³ of orthophosphoric acid ($\rho = 1,75 \text{ g/cm}^3$). Cool and dilute to 1 dm³.

4.8.3.1.2 Manganese, standard solution.

Dissolve 0,1436 g of potassium permanganate in 250 cm³ of water, reduce with a very slight excess of sulfur dioxide gas dissolved in water and dilute to 500 cm³.

1 cm³ of this solution contains 100 µg of Mn.

4.8.3.1.3 Potassium periodate.