

Rubber compounding ingredients — Natural calcium carbonate — Test methods

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The UK participation in its preparation was entrusted to Technical Committee PRI/50, Rubber raw, natural and synthetic, which has the responsibility to:

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Summary of pages

This document comprises a front cover, an inside front cover, the ISO title page, pages ii to iv, pages 1 to 20, an inside back cover and a back cover.

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INTERNATIONAL STANDARD

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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.

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.

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 *Extenders 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.