INTERNATIONAL STANDARD

ISO 5794-1

Fourth edition
2010-03-15

Rubber compounding ingredients — Silica, precipitated, hydrated —

Part 1:
Non-rubber tests

Ingrédients de mélange du caoutchouc — Silices hydratées précipitées —

Partie 1: Essais sur le produit brut

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ISO 5794-1:2010(E)

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5794-1 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*.

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This fourth edition cancels and replaces the third edition (ISO 5794-1:2005), of which it constitutes a minor revision comprising the following changes:

- the distilled or deionized water used for the tests has been replaced throughout the document by grade 3 water as defined in ISO 3696;

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- the way in which the silica content is calculated (see Table 1) has been simplified;
- for the sieve specified in F.3.2, an alternative sieve height (45 mm) has been included in addition to the sieve height of 25 mm originally specified.

This fourth edition also incorporates the Technical Corrigenda ISO 5794-1:2005/Cor.1:2006 and ISO 5794-1:2005/Cor.2:2007.

ISO 5794 consists of the following parts, under the general title *Rubber compounding ingredients — Silica, precipitated, hydrated*:

- *Part 1: Non-rubber tests*
- *Part 2: Evaluation procedures in styrene-butadiene rubber*

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INTERNATIONAL STANDARD **ISO 5794-1:2010(E)**

Rubber compounding ingredients — Silica, precipitated, hydrated —

Part 1:
Non-rubber tests

WARNING — Persons using this part of ISO 5794 should be familiar with normal laboratory practice. This part of ISO 5794 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 ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 5794 specifies methods of test for characterizing precipitated hydrated silica for use as a rubber compounding ingredient. A definition of precipitated hydrated silica is given.

ISO 5794-2 specifies the test formulation, mixing equipment, mixing procedure and methods of test for use in determining the physical properties of styrene-butadiene rubber compounded with precipitated hydrated silica.

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2 Normative references

The following referenced documents are indispensable for the application 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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

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

ISO 787-9, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension*

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

ISO 787-18, *General methods of test for pigments and extenders — Part 18: Determination of residue on sieve — Mechanical flushing procedure*

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

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

ISO 3262-19:2000, *Extenders for paints — Specifications and methods of test — Part 19: Precipitated silica*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
precipitated hydrated silica
material composed of amorphous particles obtained from soluble silicates by precipitation from aqueous solution

4 Sampling

Sampling shall be carried out in accordance with ISO 15528.

5 Methods of test

The properties of precipitated hydrated silica shall be determined by the methods of test referred to in Table 1.

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Table 1 — Methods of test

Property	Method of test
Silica content of ignited sample, % (by mass)	ISO 3262-19:2000, Clause 6
Colour	ISO 3262-1:1997, 5.1
Residue on sieve (nominal aperture size 45 µm)	
for silica in powder form	ISO 3262-19:2000, Clause 8
for silica in other forms	ISO 787-18
Volatile matter at 105 °C (loss on heating)	ISO 787-2 (Use a test portion of 2 g weighed to the nearest 0,1 mg.)
Density, Mg/m^3	ISO 787-10
Loss on ignition at 1 000 °C of dried sample	ISO 3262-1:1997, 5.2
pH of slurry	ISO 787-9
Water-soluble matter	ISO 787-8
Total copper content, mg/kg	See Annex A
Total manganese content, mg/kg	See Annex B
Total iron content, mg/kg	See Annex C
Specific surface area, m^2/g	See Annexes D, E
Granule size distribution, %	See Annex F
CTAB surface area, m^2/g	See Annex G

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6 Test report

The test report shall include the following information:

a) a reference to this part of ISO 5794;

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

c) the results of the tests:

    1) silica content,

    2) colour,

    3) residue on sieve (and the test method used),

    4) volatile matter at 105 °C,

    5) density,

    6) loss on ignition at 1 000 °C,

    7) pH of slurry,

    8) water-soluble matter,

    9) total copper content,

    10) total manganese content,

    11) total iron content,

    12) specific surface area by nitrogen adsorption (and the test method used),

    13) granule size distribution (fraction retained on the 75 µm and 300 µm screens, and the fines content),

    14) specific surface area by CTAB adsorption,

d) any unusual observations noted during the test;

e) any deviations from the test method and the reason for them;

f) any tests performed not covered by this part of ISO 5794;

g) the date of the test.

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Annex A
(normative)

Determination of total copper content

A.1 Principle

A test portion is digested with hydrofluoric acid and sulfuric acid and the silicon is volatilized as silicon tetrafluoride.

Any metals in the digested test portion are dissolved in hydrochloric acid, then the solution is diluted and aspirated into the flame of an atomic absorption spectrometer set at a wavelength of 324,7 nm.

The method is applicable to the determination of copper contents up to 125 mg/kg, and there is provision for extending the range to 1 250 mg/kg.

WARNING — All recognized health and safety precautions should be taken when performing this method of analysis.

A.2 Reagents and materials

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All reagents shall be of recognized analytical grade. The water used shall be grade 3 water as defined in ISO 3696:1987.

A.2.1 **Acetylene**, compressed-gas supply.

A.2.2 **Air**, compressed-gas supply.

A.2.3 **Hydrochloric acid**, 10 % (by mass) solution.

Dilute 20 cm^3 of 35 % (by mass) hydrochloric acid solution (ρ_{20} = 1,18 Mg/m^3) with 50 cm^3 of water.

A.2.4 **Hydrofluoric acid**, 40 % (by mass) solution (ρ_{20} = 1,13 Mg/m^3).

A.2.5 **Sulfuric acid**, 98 % (by mass) solution (ρ_{20} = 1,84 Mg/m^3).

A.2.6 **Nitric acid**, 68 % (by mass) solution (ρ_{20} = 1,42 Mg/m^3).

A.2.7 **Copper**, standard solution corresponding to 1 g of Cu per cubic decimetre.

Dissolve 1,000 g ± 0,001 g of high-purity copper turnings in a mixture of 10 cm^3 of water and 5 cm^3 of nitric acid (A.2.6) in a 100 cm^3 beaker. Boil under a fume hood to expel oxides of nitrogen. Cool, transfer to a 1 dm^3 volumetric flask, make up to the mark with water and mix. 1 cm^3 of this standard solution contains 1 000 µg of copper.

A.2.8 **Copper**, standard solution corresponding to 50 mg of Cu per cubic decimetre.

Pipette 50,0 cm^3 of the 1 g/dm^3 standard copper solution (A.2.7) into a 1 dm^3 volumetric flask, add 5 cm^3 of nitric acid (A.2.6), make up to the mark with water and mix. 1 cm^3 of this standard solution contains 50 µg of copper.

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A.2.9 Copper, standard solution corresponding to 10 mg of Cu per cubic decimetre.

Pipette 50,0 cm^3 of the 50 mg/dm^3 standard copper solution (A.2.8) into a 250 cm^3 volumetric flask, add 1 cm^3 of nitric acid (A.2.6), make up to the mark with water and mix. 1 cm^3 of this standard solution contains 10 µg of copper.

NOTE Commercially available standard copper solutions may be used, if preferred, instead of the solutions described in A.2.7, A.2.8 and A.2.9.

A.3 Apparatus

Usual laboratory equipment, plus the following:

A.3.1 Platinum dish, of capacity approximately 35 cm^3.

A.3.2 Atomic absorption spectrometer, fitted with an air/acetylene burner.

A.3.3 Analytical balance, capable of weighing to 0,1 mg.

A.4 Procedure

A.4.1 Test portion

Weigh, to the nearest 0,1 mg, approximately 2 g of sample into the platinum dish (A.3.1).

A.4.2 Blank test

Carry out a blank test simultaneously with the determination, using the same reagents and same procedures, but omitting the test portion.

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A.4.3 Preparation of the calibration graph

A.4.3.1 Preparation of standard calibration solutions

Into a series of six 50 cm^3 volumetric flasks, transfer the volumes of 10 mg/dm^3 standard copper solution (A.2.9) indicated in Table A.1, dilute to the mark with water and mix.

Table A.1 — Standard calibration solutions for determination of copper

Volume of standard copper solution (A.2.9) cm^3	Corresponding copper content µg/cm^3
0,5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

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A.4.3.2 Spectrometric measurements

Aspirate in turn each of the standard calibration solutions prepared in A.4.3.1 into the flame of the atomic absorption spectrometer (A.3.2) and record their absorbances at a wavelength of 324,7 nm, following the instructions of the instrument manufacturer.

Aspirate water into the flame after each measurement.

A.4.3.3 Plotting the graph

Plot a graph having, for example, the copper contents, in micrograms per cubic centimetre, as the abscissae and the corresponding values of absorbance as the ordinates.

A.4.4 Determination

A.4.4.1 Preparation of the test solution

Add 10 cm^3 of hydrofluoric acid solution (A.2.4) and 0,5 cm^3 of sulfuric acid solution (A.2.5) to the test portion prepared in A.4.1, in the platinum dish (A.3.1).

Place the dish and contents on a heated sand tray and evaporate under a fume hood until the evolution of dense white fumes ceases.

Dissolve any residue in 5 cm^3 of hydrochloric acid solution (A.2.3) and transfer to a 10 cm^3 volumetric flask, rinsing the dish with two 1 cm^3 portions of water and adding the rinsings to the flask. Make up to the mark with water, and transfer the solution to a dry polyethylene bottle.

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A.4.4.2 Spectrometric measurements

Aspirate the test solution prepared in A.4.4.1 and the blank test solution (see A.4.2) into the flame of the atomic absorption spectrometer and measure their absorbances at 324,7 nm, following the instructions of the instrument manufacturer. Repeat this procedure and record the mean values of the absorbance of the test solution and the blank test solution.

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Aspirate water into the flame after each measurement.

If the absorbance of the test solution is greater than that of the standard calibration solution having the highest copper content, dilute 5 cm^3 of the test solution to 50 cm^3 with water, repeat the measurements and take the dilution into account in the expression of results.

A.5 Expression of results

By reference to the calibration graph, determine the copper contents corresponding to the absorbances of the test solution and the blank test solution.

Calculate the total copper content of the sample, $w(\mathrm{Cu})$, expressed in milligrams per kilogram, from the equation

$$w(\mathrm{Cu}) = 10(\rho_1 - \rho_2)/m$$

where

ρ_1 is the copper content, in micrograms per cubic centimetre, of the test solution;

ρ_2 is the copper content, in micrograms per cubic centimetre, of the blank test solution;

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

If the test solution was diluted as described in A.4.4.2, multiply the right-hand side of the equation by 10.

Express the result to the nearest 0,1 mg/kg.

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Annex B
(normative)

Determination of total manganese content

B.1 Principle

The principle is the same as for the determination of total copper content (see Annex A), except that the absorbance of the test solution is measured at 279,5 nm and is compared with the absorbance of manganese standard calibration solutions. The method is applicable to the determination of manganese contents up to 125 mg/kg, and there is provision for extending the range to 1 250 mg/kg.

WARNING — All recognized health and safety precautions should be taken when performing this method of analysis.

B.2 Reagents and materials

All reagents shall be of recognized analytical grade. The water used shall be grade 3 water as defined in ISO 3696:1987.

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B.2.1 **Acetylene**, as specified in A.2.1.

B.2.2 **Air**, as specified in A.2.2.

B.2.3 **Hydrochloric acid solution**, as specified in A.2.3.

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B.2.4 **Hydrofluoric acid solution**, as specified in A.2.4.

B.2.5 **Sulfuric acid solution**, as specified in A.2.5.

B.2.6 **Nitric acid solution**, as specified in A.2.6.

B.2.7 **Manganese**, standard solution corresponding to 1 g of Mn per cubic decimetre.

Dissolve 1,000 g ± 0,001 g of high-purity, oxide-free manganese in a mixture of 50 cm^3 of water and 5 cm^3 of nitric acid (B.2.6) in a 400 cm^3 beaker. Boil under a fume hood to expel oxides of nitrogen. Cool, transfer to a 1 dm^3 volumetric flask, make up to the mark with water and mix.

1 cm^3 of this standard solution contains 1 000 µg of manganese.

B.2.8 **Manganese**, standard solution corresponding to 50 mg of Mn per cubic decimetre.

Pipette 50,0 cm^3 of the 1 g/dm^3 standard manganese solution (B.2.7) into a 1 dm^3 volumetric flask, add 5 cm^3 of nitric acid (B.2.6), dilute to the mark with water and mix.

1 cm^3 of this standard solution contains 50 µg of manganese.

B.2.9 **Manganese**, standard solution corresponding to 10 mg of Mn per cubic decimetre.

Pipette 50,0 cm^3 of the 50 mg/dm^3 standard manganese solution (B.2.8) into a 250 cm^3 volumetric flask, add 1 cm^3 of nitric acid (B.2.6), make up to the mark with water and mix.

1 cm^3 of this standard solution contains 10 µg of manganese.

NOTE Commercially available standard manganese solutions may be used, if preferred, instead of the solutions described in B.2.7, B.2.8 and B.2.9.

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B.3 Apparatus

As specified in A.3.

B.4 Procedure

B.4.1 Test portion

See A.4.1.

B.4.2 Blank test

See A.4.2.

B.4.3 Preparation of the calibration graph

B.4.3.1 Preparation of standard calibration solutions

Into a series of six 50 cm^3 volumetric flasks, transfer the volumes of 10 mg/dm^3 standard manganese solution (B.2.9) indicated in Table B.1, dilute to the mark with water and mix.

Table B.1 — Standard calibration solutions for determination of manganese

Volume of standard manganese solution (B.2.9) cm^3	**Corresponding manganese content** µg/cm^3
0,5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

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B.4.3.2 Spectrometric measurements

Aspirate in turn each of the standard calibration solutions prepared in B.4.3.1 into the flame of the atomic absorption spectrometer and record their absorbances at a wavelength of 279,5 nm, following the instructions of the instrument manufacturer.

Aspirate water into the flame after each measurement.

B.4.3.3 Plotting the graph

Plot a graph having, for example, the manganese contents, in micrograms per cubic centimetre, as the abscissae and the corresponding values of absorbance as the ordinates.

B.4.4 Determination

B.4.4.1 Preparation of the test solution

See A.4.4.1.

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