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**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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# Contents

Page

Foreword.....	iv
<b>1 Scope.....</b>	<b>1</b>
<b>2 Normative references.....</b>	<b>1</b>
<b>3 Terms and definitions.....</b>	<b>2</b>
<b>4 Sampling.....</b>	<b>2</b>
<b>5 Methods of test.....</b>	<b>2</b>
<b>6 Test report.....</b>	<b>3</b>
<b>Annex A (normative) Determination of total copper content.....</b>	<b>4</b>
<b>Annex B (normative) Determination of total manganese content.....</b>	<b>7</b>
<b>Annex C (normative) Determination of total iron content.....</b>	<b>10</b>
<b>Annex D (normative) Determination of specific surface area.....</b>	<b>13</b>
<b>Annex E (normative) Determination of the specific surface area by multipoint nitrogen adsorption test (BET test).....</b>	<b>19</b>
<b>Annex F (normative) Determination of granule size fractions of granulated precipitated silica.....</b>	<b>20</b>
<b>Annex G (normative) Determination of CTAB surface area.....</b>	<b>22</b>
<b>Annex H (informative) Classification of silicas and typical physical and chemical properties.....</b>	<b>28</b>
<b>Bibliography.....</b>	<b>30</b>

[ISO 5794-1:2022](https://standards.iteh.ai/catalog/standards/sist/1a704499-b243-4a6b-9e99-e50c1cad0a2e/iso-5794-1-2022)

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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.

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](http://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](http://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 of the voluntary nature of standards, 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 [www.iso.org/iso/foreword.html](http://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 fifth edition cancels and replaces the fourth edition (ISO 5794-1:2010), which has been technically revised.

The main changes are as follows:

- update of the normative references;
- addition of CAS Registry Numbers<sup>1)</sup> for reagents and chemicals;
- addition of information on the manufacturer of instruments in [Annex D](#), footnotes 1 and 2;
- withdrawal of D.6.2 on the use of a nomogram for the calculation of the specific surface area.

A list of all parts in the ISO 5794 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

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1) CAS Registry Number<sup>®</sup> is a trademark of CAS corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

# Rubber compounding ingredients — Silica, precipitated, hydrated —

## Part 1: Non-rubber tests

**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.

### 1 Scope

This document specifies methods of test for characterizing precipitated hydrated silica for use as a rubber compounding ingredient.

This document 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.

### 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 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 3262-1:2020, *Extenders — Specifications and methods of test — Part 1: Introduction and general test methods*

ISO 3262-19:2021, *Extenders — 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.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

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

Note 1 to entry: A classification of silicas and typical physical and chemical properties can be found in [Annex H](#).

### 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](#).

**Table 1 — Methods of test**

Property	Method of test
Silica content of ignited sample, % (mass fraction)	ISO 3262-19:2021, Clause 6
Colour	ISO 3262-1:2020, 5.1
Residue on sieve (nominal aperture size 45 µm)	
for silica in powder form	ISO 3262-19:2021, 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 <sup>3</sup>	ISO 787-10
Loss on ignition at 1 000 °C of dried sample	ISO 3262-1:2020, 5.2
pH of slurry	ISO 787-9
Water-soluble matter	ISO 787-8
Total copper content, mg/kg	<a href="#">Annex A</a>
Total manganese content, mg/kg	<a href="#">Annex B</a>
Total iron content, mg/kg	<a href="#">Annex C</a>
Specific surface area, m <sup>2</sup> /g	<a href="#">Annexes D and E</a>
Granule size distribution, %	<a href="#">Annex F</a>
CTAB surface area, m <sup>2</sup> /g	<a href="#">Annex G</a>

## 6 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 5794-1;
- 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 document;
- g) the date of the test.

## 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 applicable health and safety precautions should be taken when performing this method of analysis.**

#### A.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.

**A.2.1 Acetylene** (CAS: 74-86-2), compressed-gas supply. [2022](https://standards.iteh.ai/catalog/standards/sist/1a704499-b243-4a6b-9e99-e50c1cad0a2e/iso-5794-1-2022)

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

**A.2.3 Hydrochloric acid** (CAS: 7647-01-0), 10 % (mass fraction) solution.

Dilute 20 cm<sup>3</sup> of 35 % (mass fraction) hydrochloric acid solution ( $\rho_{20} = 1,18 \text{ Mg/m}^3$ ) with 50 cm<sup>3</sup> of water.

**A.2.4 Hydrofluoric acid** (CAS: 7664-39-3), 40 % (mass fraction) solution ( $\rho_{20} = 1,13 \text{ Mg/m}^3$ ).

**A.2.5 Sulfuric acid** (CAS: 7664-93-9), 98 % (mass fraction) solution ( $\rho_{20} = 1,84 \text{ Mg/m}^3$ ).

**A.2.6 Nitric acid** (CAS: 7697-37-2), 68 % (mass fraction) solution ( $\rho_{20} = 1,42 \text{ Mg/m}^3$ ).

**A.2.7 Copper** (CAS: 7440-50-8), standard solution corresponding to 1 g of copper/dm<sup>3</sup>.

Dissolve 1,000 g  $\pm$  0,001 g of high-purity copper turnings in a mixture of 10 cm<sup>3</sup> of water and 5 cm<sup>3</sup> of nitric acid ([A.2.6](#)) in a 100 cm<sup>3</sup> beaker. Boil under a fume hood to expel oxides of nitrogen. Cool, transfer to a 1 dm<sup>3</sup> volumetric flask, make up to the mark with water and mix. 1 cm<sup>3</sup> of this standard solution contains 1 000  $\mu\text{g}$  of copper.

**A.2.8 Copper**, standard solution corresponding to 50 mg of copper/dm<sup>3</sup>.

Pipette 50,0 cm<sup>3</sup> of the 1 g/dm<sup>3</sup> standard copper solution ([A.2.7](#)) into a 1 dm<sup>3</sup> volumetric flask, add 5 cm<sup>3</sup> of nitric acid ([A.2.6](#)), make up to the mark with water and mix. 1 cm<sup>3</sup> of this standard solution contains 50  $\mu\text{g}$  of copper.



**A.2.9 Copper**, standard solution corresponding to 10 mg of copper/dm<sup>3</sup>.

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

NOTE Commercially available standard copper solutions can 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 and the following shall be used.

**A.3.1 Platinum dish**, of capacity approximately 35 cm<sup>3</sup>.

**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.

#### A.4.3 Preparation of the calibration graph

##### A.4.3.1 Preparation of standard calibration solutions

Into a series of six 50 cm<sup>3</sup> volumetric flasks, transfer the volumes of 10 mg/dm<sup>3</sup> 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 <sup>3</sup>	Corresponding copper content µg/cm <sup>3</sup>
0,5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

##### 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<sup>3</sup> of hydrofluoric acid solution (A.2.4) and 0,5 cm<sup>3</sup> 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<sup>3</sup> of hydrochloric acid solution (A.2.3) and transfer to a 10 cm<sup>3</sup> volumetric flask, rinsing the dish with two 1 cm<sup>3</sup> 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.

### 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.

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<sup>3</sup> of the test solution to 50 cm<sup>3</sup> with water, repeat the measurements and take the dilution into account in the expression of results.

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## 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,  $w(\text{Cu})$ , expressed in milligrams per kilogram, from Formula (A.1).

$$w(\text{Cu}) = 10(\rho_1 - \rho_2)/m \quad (\text{A.1})$$

where

$\rho_1$  is the copper content, in micrograms per cubic centimetre, of the test solution;

$\rho_2$  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.

## 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 applicable 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.

**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](#).

**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** (CAS: 7439-96-5), standard solution corresponding to 1 g of manganese/dm<sup>3</sup>.

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

1 cm<sup>3</sup> of this standard solution contains 1 000 µg of manganese.

**B.2.8 Manganese**, standard solution corresponding to 50 mg of manganese/dm<sup>3</sup>.

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

1 cm<sup>3</sup> of this standard solution contains 50 µg of manganese.

**B.2.9 Manganese**, standard solution corresponding to 10 mg of manganese/dm<sup>3</sup>.

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

1 cm<sup>3</sup> of this standard solution contains 10 µg of manganese.

NOTE Commercially available standard manganese solutions can be used, if preferred, instead of the solutions described in [B.2.7](#), [B.2.8](#) and [B.2.9](#).

### 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<sup>3</sup> volumetric flasks, transfer the volumes of 10 mg/dm<sup>3</sup> 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 ( <a href="#">B.2.9</a> ) cm <sup>3</sup>	Corresponding manganese content µg/cm <sup>3</sup>
0,5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

##### 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](#).