BS ISO 5794-1:2022
Incorporating corrigendum September 2022

BSI Standards Publication

Rubber compounding ingredients — Silica, precipitated, hydrated

Part 1: Non-rubber tests

Documents
Document Sharing Hub

Full document details: BS ISO 5794-1:2022

National foreword

This British Standard is the UK implementation of ISO 5794-1:2022. It supersedes BS ISO 5794-1:2010, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Raw materials (including latex) for use in the rubber industry.

A list of organizations represented on this committee can be obtained on request to its committee manager.

Contractual and legal considerations

This publication has been prepared in good faith, however no representation, warranty, assurance or undertaking (express or implied) is or will be made, and no responsibility or liability is or will be accepted by BSI in relation to the adequacy, accuracy, completeness or reasonableness of this publication. All and any such responsibility and liability is expressly disclaimed to the full extent permitted by the law.

This publication is provided as is, and is to be used at the recipient's own risk.

The recipient is advised to consider seeking professional guidance with respect to its use of this publication.

This publication is not intended to constitute a contract. Users are responsible for its correct application.

© The British Standards Institution 2022
Published by BSI Standards Limited 2022

ICS 83.040.20

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2022.

Amendments/corrigenda issued since publication

Date	Text affected
30 September 2022	Correction to incorrectly typeset text

Documents
Document Sharing Hub

Full document details: BS ISO 5794-1:2022

INTERNATIONAL STANDARD

ISO 5794-1

Fifth edition
2022-06

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

Reference number
ISO 5794-1:2022(E)

Documents
Document Sharing Hub
© ISO 2022

Full document details: BS ISO 5794-1:2022

COPYRIGHT PROTECTED DOCUMENT

© ISO 2022

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

© ISO 2022 – All rights reserved

Documents
Document Sharing Hub

Full document details: BS ISO 5794-1:2022

Contents

Page

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

© ISO 2022 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022

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 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.

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®[1)] 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.

1) CAS Registry Number® 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.

© ISO 2022 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022

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 2022 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022

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^3	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	Annex A
Total manganese content, mg/kg	Annex B
Total iron content, mg/kg	Annex C
Specific surface area, m^2/g	Annexes D and E
Granule size distribution, %	Annex F
CTAB surface area, m^2/g	Annex G

© ISO 2022 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022

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.

© ISO 2022 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022

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.

A.2.2 Air, compressed-gas supply.

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

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

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

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

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

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

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 copper/dm^3.

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.

© ISO 2022 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022

A.2.9 Copper, standard solution corresponding to 10 mg of copper/dm^3.

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 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^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.

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

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.

© ISO 2022 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022

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.

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^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(Cu), expressed in milligrams per kilogram, from Formula (A.1).

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

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.

© ISO 2022 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 5794-1:2022