BS ISO 6101-3:2014

BSI Standards Publication

Rubber — Determination of metal content by atomic absorption spectrometry

Part 3: Determination of copper content

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National foreword

This British Standard is the UK implementation of ISO 6101-3:2014. It supersedes BS 7164-28.1:1997 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

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

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Rubber — Determination of metal content by atomic absorption spectrometry —

Part 3:
Determination of copper content

Caoutchouc — Détermination de la teneur en métal par spectrométrie d'absorption atomique —

Partie 3: Dosage du cuivre

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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).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 6101-3:1997), which has been technically revised.

ISO 6101 consists of the following parts, under the general title *Rubber — Determination of metal content by atomic absorption spectrometry*:

- *Part 1: Determination of zinc content*
- *Part 2: Determination of lead content*
- *Part 3 Determination of copper content*
- *Part 4: Determination of manganese content*
- *Part 5: Determination of iron content*
- *Part 6: Determination of magnesium content*

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 3:
Determination of copper content

WARNING — Persons using this part of ISO 6101 should be familiar with normal laboratory practice. This part of ISO 6101 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

This part of ISO 6101 specifies an atomic absorption spectrometric method for the determination of the copper content of rubbers.

The method is applicable to raw rubber and rubber products having copper contents above 1 ppm. Copper contents below this limit can be determined, provided that suitable adjustments are made to the mass of the test portion and/or to the concentrations of the solutions used. The use of the standard additions method can lower the bottom limit of detection.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 247, *Rubber — Determination of ash*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 1772, *Laboratory crucibles in porcelain and silica*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

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

3 Principle

A test portion is ashed at 550 °C ± 25 °C in accordance with ISO 247, method A. The ash is dissolved in hydrochloric acid or nitric acid. The solution is aspirated into an atomic absorption spectrometer and the absorption is measured at a wavelength of 324,7 nm, using a copper hollow-cathode lamp as the copper emission source. Any silicates are volatilized by sulfuric and hydrofluoric acid.

NOTE ISO 6955:1982 defines the spectrometric terms used in this part of ISO 6101.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, ρ_{20} = 1,18 Mg/m^3, 37 % (m/m).

4.2 Hydrochloric acid, diluted 1 + 2.

Dilute 1 volume of concentrated hydrochloric acid (4.1) with 2 volumes of water.

4.3 Sulfuric acid, ρ_{20} = 1,84 Mg/m^3, 95 % (m/m) to 97 % (m/m).

4.4 Sulfuric acid, diluted 1 + 3.

Add carefully 1 volume of concentrated sulfuric acid (4.3) to 3 volumes of water.

4.5 Hydrofluoric acid, ρ_{20} = 1,13 Mg/m^3, 38 % (m/m) to 40 % (m/m).

4.6 Hydrogen peroxide, 30 % (m/m) solution.

4.7 Concentrated nitric acid, ρ_{20} = 1,41 Mg/m^3.

4.8 Dilute nitric acid, 1,6 % (by mass), prepared by carefully pipetting 11,5 cm^3 of concentrated nitric acid (4.7) into a 1 000 cm^3 one-mark volumetric flask, making up to the mark with water and mixing thoroughly.

4.9 Standard copper stock solution, containing 1 g of Cu per 1 000 cm^3.

Either use a commercially available standard copper solution, or prepare as follows:

Weigh, to the nearest 0,1 mg, 1 g of electrolytic copper (purity ≥99,9 %) and dissolve in 50 cm^3 of concentrated hydrochloric acid (4.1). Add 15 cm^3 of hydrogen peroxide solution (4.6). After dissolution, decompose the excess hydrogen peroxide by boiling. Allow to cool and transfer quantitatively to a 1 000 cm^3 one-mark volumetric flask (see 5.5). Dilute to the mark and mix thoroughly.

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

4.10 Standard copper solution, containing 10 mg of Cu per 1 000 cm^3.

Carefully pipette 10 cm^3 of the standard copper stock solution (4.9) into a 1 000 cm^3 one-mark volumetric flask (see 5.5) and dilute to the mark with 1 + 2 hydrochloric acid (4.2) or dilute nitric acid (4.8), and mix thoroughly. Prepare this solution on the day of use.

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

5 Apparatus

Ordinary laboratory apparatus, plus the following.

5.1 Atomic absorption spectrometer, fitted with a burner fed with acetylene and air, compressed to at least 60 kPa and 300 kPa, respectively, and also fitted with a copper hollow-cathode lamp as the copper emission source.

The instrument shall be operated in accordance with the manufacturer's instructions for optimum performance.

Alternatively, an electrothermal atomization device (graphite furnace) may be used. It shall be operated by a competent person in accordance with the manufacturer's instructions for optimum performance.

5.2 Balance, accurate to 0,1 mg.

5.3 Muffle furnace, capable of being maintained at a temperature of 550 °C ± 25 °C.

5.4 Glass filter crucible, filter pore size 16 µm to 40 µm (porosity grade P40 — according to ISO 4793).

5.5 One-mark volumetric flasks, glass-stoppered, of capacities 50 cm^3, 100 cm^3, 200 cm^3, 500 cm^3 and 1 000 cm^3, complying with the requirements of ISO 1042, class A.

5.6 Volumetric pipettes, of capacities 5 cm^3, 10 cm^3, 20 cm^3, and 50 cm^3, complying with the requirements of ISO 648, class A.

5.7 Graduated pipette, of capacity 1cm^3, complying with the requirements of ISO 835, class A.

5.8 Electric hotplate, or heated sand bath.

5.9 Steam bath.

5.10 Platinum or borosilicate-glass rod, for use as a stirrer.

5.11 Crucible, of platinum, and of capacity 50 cm^3 to 150 cm^3 depending on the test portion size.

5.12 Crucible, of silica, porcelain, or borosilicate glass, of capacity 50 cm^3 to 150 cm^3 depending on the test portion size, complying with the requirements of ISO 1772.

5.13 Ashless filter paper.

6 Sampling

Carry out sampling as follows:

- raw rubber: in accordance with ISO 1795;
- latex: in accordance with ISO 123;
- products: to be representative of the whole batch.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,1 mg, approximately 1 g to 5 g of rubber product and 5 g to 10 g of raw rubber, milled or finely cut, into an appropriate crucible. Wrap the test portion in an ashless filter paper and place into an appropriate crucible (5.11 or 5.12). The size of the test portion shall be judged by prior knowledge of the approximate amount of copper present.

For natural rubber latex concentrate, take a portion of thoroughly mixed latex containing about 10 g of total solids, make into a thin film by pouring the portion onto a glass plate, dry to constant mass as specified in ISO 124 and cut into small pieces.

For raw, natural rubber, take the test portion from a test sample prepared in accordance with ISO 1795.

7.2 Preparation of test solution

7.2.1 Destruction of organic matter

Ash the test portion in accordance with method A of ISO 247, in the muffle furnace (5.3), maintained at 550 °C ± 25 °C. If the ash is black, caused by small amounts of carbon black, stir carefully with the platinum or borosilicate-glass rod (5.10) and continue heating.

7.2.2 Dissolution of inorganic residue using hydrochloric acid

After ashing, allow the crucible and its contents to cool to ambient temperature. Add 20 cm^3 of concentrated hydrochloric acid (4.1). Heat the mixture on the steam bath (5.9) for at least 10 min. Do not let the reaction mixture boil. Allow to cool to ambient temperature and transfer the solution quantitatively, with the aid of water, to a 50 cm^3 one-mark volumetric flask (5.5). If the ash is not totally dissolved, proceed as follows.

— Transfer the solution and the undissolved ash quantitatively, with the aid of water, to a platinum crucible (5.11). Add a few drops of concentrated sulfuric acid (4.3) and 5 cm^3 of hydrofluoric acid (4.5). Heat on the electric hotplate or heated sand bath (5.8) in a fume cupboard and evaporate to dryness, while stirring with a platinum rod (5.10). Repeat this digestion with the same quantities of sulfuric and hydrofluoric acids two more times.

— Allow to cool to ambient temperature, add 20 cm^3 of concentrated hydrochloric acid (4.1), heat for 10 min and transfer quantitatively, with the aid of water, to a 50 cm^3 one-mark volumetric flask (5.5).

— Dilute to the mark with water and mix thoroughly. Insoluble matter can settle and, if so, shall be filtered off using a filter crucible (5.4) just before making spectrometric measurements in accordance with 7.3.

Test solutions should contain approximately 12 % hydrochloric acid. If evaporation, etc., has reduced or increased this concentration, adjust accordingly with concentrated hydrochloric acid (4.1) or water.

7.2.3 Dissolution of inorganic residue using nitric acid — Alternative method

Add 10 cm^3 of dilute nitric acid (4.8) to the cooled residue. Cover with a watch glass and heat on a steam bath (5.9) for at least 30 min. Allow to cool to ambient temperature. Filter the contents of the crucible into a 50 cm^3 one-mark volumetric flask, rinsing the crucible, and making up to the mark with dilute nitric acid (4.8).

7.3 Preparation of the calibration graph

7.3.1 Preparation of calibration solutions

7.3.1.1 Into a series of five 100 cm^3 one-mark volumetric flasks (see 5.5), introduce, using pipettes (see 5.6) the volumes of standard copper solution (4.10) indicated in Table 1. Dilute to the mark with 1 + 2 hydrochloric acid solution (4.2) or dilute nitric acid (4.8) and mix thoroughly.

Table 1 — Standard calibration solutions

Volume of standard copper solution cm^3	**Mass of copper contained in 1 cm^3** µg
50	5
20	2
10	1

Table 1 *(continued)*

Volume of standard copper solution cm^3	**Mass of copper contained in 1 cm^3** µg
5	0,5
0	0

7.3.1.2 Prepare the set of calibration solutions immediately prior to the determination.

7.3.2 Spectrometric measurements

Switch on the spectrometer (5.1) sufficiently in advance to ensure stabilization. With the copper hollow-cathode tube suitably positioned, adjust the wavelength to 324,7 nm and the sensitivity and slit aperture according to the characteristics of the instrument.

Adjust the pressures and flow rates of the air and of the acetylene in accordance with the manufacturer's instructions so as to obtain a clear blue, non-luminous, oxidizing flame, suited to the characteristics of the particular spectrometer being used.

Aspirate the set of calibration solutions in succession into the flame and measure the absorbance of each solution twice, averaging the readings. Take care that the aspiration rate is constant throughout this process. Ensure also that at least one calibration solution is at or below the analyte level found in the rubber being tested.

Aspirate water through the burner after each measurement.

7.3.3 Plotting the calibration graph

Plot a graph having, for example, the masses, in micrograms, of copper contained in 1 cm^3 of the calibration solutions as abscissae and the corresponding values of absorbance, corrected for the absorbance of the calibration blank, as ordinates. Represent the points on the graph by the best straight line as judged visually or as calculated by the least-squares fit method.

7.4 Determination

7.4.1 Spectrometric measurements

Carry out duplicate spectrometric measurements at a wavelength of 324,7 nm on the test solution prepared in 7.2.2 or 7.2.3, following the procedure specified in 7.3.2.

7.4.2 Dilution

If the instrument response for the test solution is greater than that found for the calibration solution having the highest copper content, dilute, as appropriate, with 1 + 2 hydrochloric acid solution (4.2) or dilute nitric acid (4.8) in accordance with the following procedure.

Pipette carefully a suitable volume (V cm^3) of the test solution into a 100 cm^3 one-mark volumetric flask (see 5.5) so that the copper concentration lies within the range covered by the calibration solutions. Dilute to the mark with 1 + 2 hydrochloric acid (4.2) or dilute nitric acid (4.8). Repeat the spectrometric measurements.

NOTE To increase the reliability of the test method, the standard-additions method can be used (see Annex A).

7.5 Blank determination

Carry out a blank test in parallel with the determination, using 1 + 2 hydrochloric acid (4.2) or dilute nitric acid (4.8), but omitting the test portion.

If the preparation of the test solution involved the use of sulfuric acid and hydrofluoric acid, prepare the blank test solution by repeating that procedure, but omitting the test portion.

7.6 Number of determinations

Carry out the procedure in duplicate, using separate test portions cut from the same homogenized sample.

8 Expression of results

8.1 Read the copper content of the test solution directly from the calibration graph plotted in 7.3.3. The copper content of the test portion, expressed as a percentage by mass, is given by Formula (1):

$$\frac{\rho(Cu)_\mathrm{t} - \rho(Cu)_\mathrm{b}}{200m} \times f \qquad (1)$$

where

$\rho(Cu)_\mathrm{t}$ is the copper content, in micrograms per cubic centimetre, of the test solution, read from the calibration graph;

$\rho(Cu)_\mathrm{b}$ is the copper content, in micrograms per cubic centimetre, of the blank test solution, read from the calibration graph;

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

f is the test solution dilution factor, if required (see 7.4.2), given by:

$$f = \frac{100}{V}$$

where

V is the volume, in cubic centimetres, of test solution taken in 7.4.2.

8.2 The copper content of the test portion can also be calculated, in micrograms per cubic centimetre, of the test solution, by Formula (2):

$$\frac{\rho(Cu)_\mathrm{t} - \rho(Cu)_\mathrm{b}}{200m} \times f \qquad (2)$$

where

$\rho(Cu)_\mathrm{t}$ is the copper content, in micrograms per cubic centimetre, of the test solution, given by Formula (3):

$$\rho(Cu)_\mathrm{t} = \frac{A_\mathrm{t} \times \rho(Cu)_\mathrm{n}}{A_\mathrm{n}} \qquad (3)$$

$\rho(Cu)_\mathrm{b}$ is the copper content, in micrograms per cubic centimetre, of the blank test solution, given by Formula (4):

$$\rho(Cu)_b = \frac{A_b \times \rho(Cu)_n}{A_n} \quad (4)$$

where

A_t is the absorbance of the test solution;

A_b is the absorbance of the blank test solution;

A_n is the absorbance of the standard calibration solution having the copper content closest to that of the test solution;

$\rho(Cu)_n$ is the copper content, in micrograms per cubic centimetre, of the standard calibration solution having the absorbance closest to that of the test solution;

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

f is the test solution dilution factor, if required (see 7.4.2), given by:

$$f = \frac{100}{V}$$

where

V is the volume, in cubic centimetres, of the test solution taken in 7.4.2.

8.3 The test result is the average of two determinations, rounded to two decimal places when the copper concentration is expressed as a percentage and to the nearest whole number when the concentration is expressed in milligrams per kilogram.

8.4 Report the copper content as a percentage if greater than or equal to 0,1 % or as milligrams per kilogram if less than 0,1 %.

9 Precision

See Annex B.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard (ISO 6101-3);

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

c) the method of sampling used;

d) the type of instrument used (flame or graphite furnace spectrometer);

e) the results obtained and the units in which they have been expressed;

f) any unusual features noted during the determination;

g) any operations not included in this part of ISO 6101, or in the International Standards to which reference is made, as well as any incident which might have affected the results.

Annex A
(informative)

Method of standard additions

The method of standard additions provides the analyst with a powerful tool for increasing the accuracy of an atomic absorption analysis.

It is used with samples containing unknown concentrations of matrix materials, with samples which are difficult to duplicate with blanks and/or when it is necessary to lower the limits of detection.

The method of standard additions can be found in any standard text book on atomic absorption and is usually described in the user's manual supplied with the atomic absorption spectrometer.

The following example illustrates the method.

- From a test solution prepared as described in 7.2, take four aliquots of the same size. To three of these aliquots, add a different, but known volume of standard copper solution. Make up the volumes to the same total for all four aliquots. Use concentrations which fall on the linear portion of the calibration graph.
- Measure the absorbance of each of the four solutions so obtained.
- Plot absorbance on the y-axis and the concentration, in micrograms of copper per cubic centimetre of solution, on the x-axis.
- Extrapolate the straight line to intersect the x-axis (zero absorbance). At the point of intersection with the y-axis, read the concentration of copper in the test solution.

An example is given in Figure A.1.

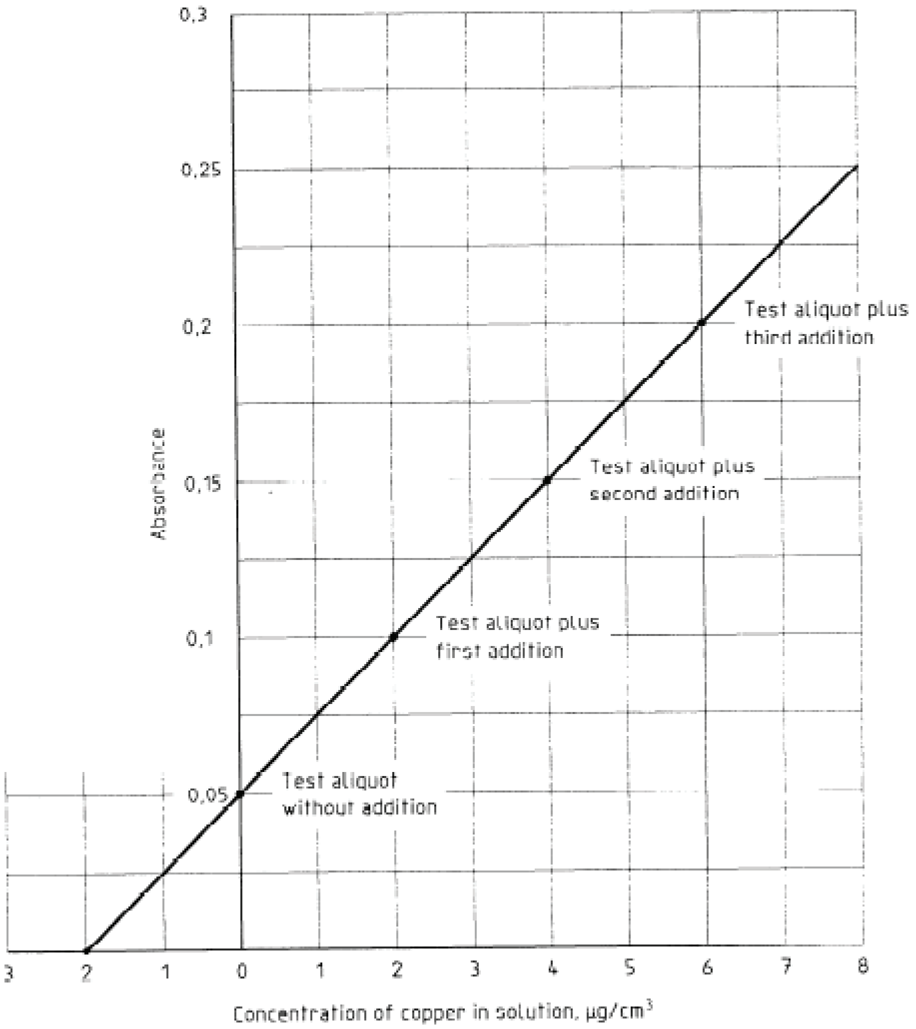

Figure A.1 — Example of graph obtained using the method of standard additions

Annex B
(informative)

Precision

The precision of the test method was determined in accordance with ISO/TR 9272. Refer to that document for terminology and other statistical details.

The precision data are given in Table B.1. The precision parameters should not be used for acceptance or rejection of any group of materials without documentation that the parameters are applicable to those particular materials and specific test protocols of the test method. The precision is expressed on the basis of a 95 % confidence level for the values established for repeatability, r, and reproducibility, R.

The results contained in Table B.1 are average values and give an estimate of the precision of this test method as determined in an interlaboratory test programme, carried out in 2012, in which seven laboratories took part, performing duplicate analyses on two samples, A and B, prepared from high-ammonia natural rubber latex concentrate. Before the bulk latex was sub-sampled into two bottles labelled A and B, it was filtered and homogenized by thorough stirring. Thus, essentially, samples A and B were the same and were treated as such in the statistical computations. Each participating laboratory was required to carry out the test, using these two samples, on the dates given to them.

A type 1 precision was evaluated, based on the method of sampling used for the interlaboratory test programme.

Repeatability: The repeatability r (in measurement units) of the test method has been established as the appropriate value tabulated in Table B.1. Two single test results, obtained in the same laboratory under normal test method procedures, that differ by more than the tabulated value of r (for any given level) should be considered to have come from different or non-identical sample populations.

Reproducibility: The reproducibility R (in measurement units) of the test method has been established as the appropriate value tabulated in Table B.1. Two single test results, obtained in different laboratories under normal test method procedures, that differ by more than the tabulated value of R (for any given level) should be considered to have come from different or non-identical sample populations.

Bias: In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias cannot therefore be determined for this particular method.

Table B.1 — Precision data for the determination of copper in natural rubber latex concentrate

Average result	Within-laboratory		Between laboratories	
µg/cm^3	s_r	r	S_R	R
2,57	0,41	1,16	0,81	2,30

s_r is the within-laboratory standard deviation

r is the repeatability (in measurement units)

S_R is the between-laboratory standard deviation

R is the reproducibility (in measurement units)

Bibliography

[1] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

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