INTERNATIONAL STANDARD

ISO 6101-6

Second edition
2018-09

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 6: Determination of magnesium content

Caoutchouc — Dosage du métal par spectrométrie d'absorption atomique —

Partie 6: Dosage du magnésium

Reference number
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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).

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 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 6101-6:2011), which has been technically revised.

The main changes compared to the previous edition are as follows:

— The procedure for the destruction of organic matter has been further detailed;

— Formula (2) in 8.5 has been changed to magnesium content expressed as milligram per kilogram;

— Precision data have been updated in Annex B.

A list of all parts in the ISO 6101 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.

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 6:
Determination of magnesium content

WARNING 1 — 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 and to determine applicability of any national regulatory conditions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies an atomic absorption spectrometric method for the determination of the magnesium content of natural rubber latex concentrate, raw natural rubber and products made from natural rubber.

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 123, *Rubber latex — Sampling*

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

ISO 247-1, *Rubber — Determination of ash — Part 1: Dry combustion technique*

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

ISO 835, *Laboratory glassware — Graduated 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*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological database for use in standardization at the following address:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

A test portion is ashed at 550 °C ± 25 °C for raw natural rubber or at 950 °C ± 25 °C for rubber products having a high carbon black content in accordance with ISO 247-1. The ash is dissolved in dilute nitric acid. The solution is aspirated into an atomic absorption spectrometer and the absorption is measured at a wavelength of 285,2 nm, using a magnesium hollow-cathode lamp as the magnesium emission source.

5 Reagents

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

5.1 Concentrated nitric acid, ρ_{20} = 1,41 g/cm^3, 65 % (m/m) to 70 % (m/m).

5.2 Dilute nitric acid, 1,6 % (by mass), carefully pipette 11,5 cm^3 of concentrated nitric acid (5.1) into a 1 000 cm^3 one-mark volumetric flask, making up to the mark with water, and mix thoroughly.

5.3 Standard magnesium stock solution, containing 1 g of Mg per cubic decimetre.

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

Grind metallic magnesium, purity greater than 99 % (by mass). Weigh 1 g to the nearest 0,1 mg in a 250 cm^3 conical flask (6.13) and dissolve it in a mixture of 100 cm^3 of dilute nitric acid (5.2) and 10 cm^3 of concentrated nitric acid (5.1). Transfer it to a 1 000 cm^3 one-mark volumetric flask (6.4), dilute to the mark with dilute nitric acid (5.2) and mix thoroughly.

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

5.4 Standard magnesium solution, containing 10 µg of Mg per cubic centimetre, carefully pipette 10 cm^3 of standard magnesium stock solution (5.3) into a 1 000 cm^3 one-mark volumetric flask, making up to the mark with dilute nitric acid (5.2), and mix thoroughly. Prepare this solution preferably on the day of use.

6 Apparatus

6.1 Atomic absorption spectrometer, fitted with a burner fed with acetylene and compressed air and also fitted with a magnesium hollow-cathode lamp capable of emitting radiation of the required wavelengths. A high-brightness lamp is advisable.

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.

6.2 Balance, accurate to 0,1 mg.

6.3 Muffle furnace, capable of being maintained at a temperature of 550 °C ± 25 °C or 950 °C ± 25 °C.

6.4 One-mark volumetric flasks, glass-stoppered, of capacities 50 cm^3, 100 cm^3 and 1 000 cm^3, in accordance with the requirements of ISO 1042, class A.

6.5 Volumetric pipettes, of capacities 0,5 cm^3, 1 cm^3, 5 cm^3, 10 cm^3, 20 cm^3 and 50 cm^3, in accordance with the requirements of ISO 648, class A.

6.6 Graduated pipette, of capacity 1 cm^3, in accordance with the requirements of ISO 835, class A.

6.7 Steam bath.

6.8 Borosilicate-glass rod, for use as a stirrer.

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

6.10 Ashless filter paper.

6.11 Electrical heating plate or **sand bath.**

6.12 Watch glasses, for covering the crucibles (6.9).

6.13 Conical flask, of capacity 250 cm^3.

7 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.

8 Procedure

WARNING — All recognized health and safety precautions shall be observed when carrying out the procedures specified in this document.

8.1 Preparation of test portion

8.1.1 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 (6.9). The size of the test portion shall be judged by prior knowledge of the approximate amount of magnesium present.

8.1.2 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.

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

8.2 Preparation of test solution

8.2.1 Ashing of test portion (destruction of organic matter)

Ash in accordance with ISO 247-1 in the muffle furnace (6.3) maintained at 550 °C ± 25 °C for raw natural rubber or at 950 °C ± 25 °C for rubber products. After ashing, allow the crucible and its contents to cool to ambient temperature.

If the ash is black, caused by small amounts of carbon black, add 1 cm^3 of concentrated nitric acid (5.1) to the ash, evaporate to dryness on an electrical heating plate or sand bath (6.11) and return to the muffle furnace and ash for 10 min to 15 min.

8.2.2 Dissolution of inorganic residue

Add 10 cm^3 of dilute nitric acid (5.2) to the cooled residue. Cover with a watch glass (6.12) and heat on a steam bath (6.7) 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 with dilute nitric acid (5.2) to the mark. Proceed in accordance with 8.4

8.3 Preparation of calibration graph

8.3.1 Preparation of standard solutions

Into a series of five 100 cm^3 one-mark volumetric flasks, introduce, using pipettes, the volumes of standard magnesium solution (5.4) indicated in Table 1. Make up to the mark with dilute nitric acid (5.2) and mix thoroughly.

Prepare the set of calibration solutions on the same day as the determination.

Table 1 — Standard calibration solutions

Volume of standard magnesium solution cm^3	Mass of magnesium contained in 1 cm^3 µg
50,0	5
20,0	2
10,0	1
5,0	0,5
0,0	0

8.3.2 Spectrometric measurements

Switch on the spectrometer (6.1) sufficiently in advance to ensure stabilization. With the magnesium hollow-cathode lamp suitably positioned, adjust the wavelength to 285,2 nm and the sensitivity and slit aperture according to the characteristics of the instrument.

Adjust the pressure and flow rate 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 standard is at or below the level corresponding to the rubber being tested.

Aspirate water through the burner after each measurement.

8.3.3 Plotting the calibration graph

Plot a graph having, for example, the masses, in micrograms, of magnesium 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 a least-squares fitting method.

8.4 Determination

8.4.1 Spectrometric measurements

Carry out duplicate spectrometric measurements at a wavelength of 285,2 nm on the test solution prepared in 8.2.2, following the procedure specified in 8.2.

8.4.2 Dilution

If the instrument response for the test solution is greater than that found for the calibration solution having the highest magnesium content, dilute the test solution 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 so that, after making up to the mark, the magnesium concentration will lie within the range covered by the calibration solutions. Make up to the mark with dilute nitric acid (5.2) and mix thoroughly. Repeat the spectrometric measurements.

Under certain circumstances, the method of standard additions may be used (see Annex A).

8.4.3 Blank determination

Carry out a blank test in parallel with the determination, using dilute nitric acid (5.2), but omitting the test portion.

8.4.4 Number of determinations

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

8.5 Expression of results

Read the magnesium content of the test solution directly from the calibration graph plotted in 8.3.3.

The magnesium content of the test portion, expressed as a percentage by mass, is given by Formula (1):

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

where

$\rho(\mathrm{Mg})_\mathrm{t}$ is the magnesium content, in µg/cm^3, of the test solution, read from the calibration graph;

$\rho(\mathrm{Mg})_\mathrm{b}$ is the magnesium content, in µg/cm^3, of the blank test solution, read from the calibration graph;

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

f is the test solution dilution factor, if required (see 8.4.2), given by:

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

where V is the volume, in cm^3, of test solution taken in 8.4.2.

The magnesium content of the test portion can also be calculated, in mg/kg, of the test solution, by Formula (2):

$$\frac{50\left[\rho(\mathrm{Mg})_{\mathrm{t}} - \rho(\mathrm{Mg})_{\mathrm{b}}\right]}{m} \times f \tag{2}$$

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

Report the magnesium 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 document, i.e. ISO 6101-6:2018;

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

c) the method of sampling used;

d) the type of spectrometer used;

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

f) any unusual features noted during the determination;

g) any operation not included in this document, 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 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 textbook on atomic absorption and is usually described in the user's manual supplied with the atomic absorption spectrometer.

The following example illustrates the method.

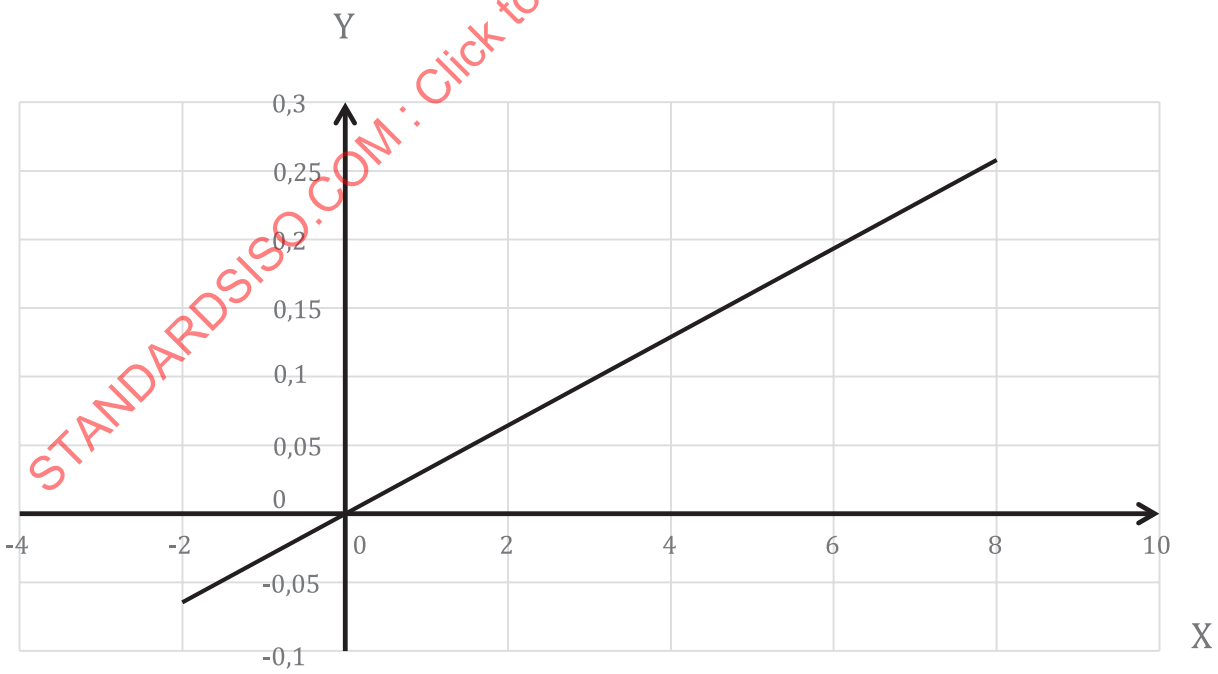
From a test solution prepared as described in 8.2, take four aliquots of the same size. To three of these aliquots, add a different, but known, volume of standard magnesium solution. Make up the volumes to the same total for all four aliquots. Use concentrations which fall on the linear portion of the 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 magnesium 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 X-axis, read off the concentration of magnesium in the test solution.

An example is given in Figure A.1.



Key

X concentration of magnesium in solution, µg/cm^3

Y absorbance

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