INTERNATIONAL STANDARD

ISO 6101-6

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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.

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 6101-6 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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 6:
Determination of magnesium 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.

IMPORTANT — Certain procedures specified in this part of ISO 6101 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 part of ISO 6101 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 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 123, *Rubber latex —Sampling*

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

ISO 247, *Rubber — Determination of ash*

ISO 648:2008, *Laboratory glassware — Single-volume pipettes*

ISO 835:2007, *Laboratory glassware — Graduated pipettes*

ISO 1042:1998, *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

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

3.1

natural rubber latex concentrate

natural rubber latex from *Hevea brasiliensis*, containing ammonia and/or other preservatives, and which has been subjected to some process of concentration

3.2

raw natural rubber

natural rubber, usually in bales or packages, forming the starting material for the manufacture of rubber articles

4 Principle

A test portion is ashed at 550 °C ± 25 °C, or at 950 °C ± 25 °C for rubber products having a high carbon black content. 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, density = 1,41 g/cm^3.

5.2 Dilute nitric acid, 1,6 % (by mass), prepared by carefully pipetting 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 mixing thoroughly.

5.3 Standard magnesium stock solution, containing 1 mg of Mg per cubic centimetre. Use a commercially available standard magnesium solution.

5.4 Standard magnesium solution, containing 10 µg of Mg per cubic centimetre, prepared by carefully pipetting 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 mixing 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 air compressed to at least 60 kPa and 300 kPa, respectively, and also fitted with a magnesium hollow-cathode lamp as the magnesium emission source. The instrument shall be operated 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, complying with the requirements of ISO 1042:1998, class A.

6.5 Volumetric pipettes, of capacities 5 cm^3, 10 cm^3, 20 cm^3 and 50 cm^3, complying with the requirements of ISO 648:2008, class A.

6.6 Graduated pipette, of capacity 1 cm^3, complying with the requirements of ISO 835:2007, 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, complying with the requirements of ISO 1772.

6.10 Ashless filter paper.

7 Sampling

Carry out sampling in accordance with one of the methods specified in ISO 123 for natural rubber latex, in accordance with ISO 1795 for raw, natural rubber and on a milled or finely cut test sample representative of the whole sample for products made from natural rubber.

8 Procedure

8.1 Test portion

8.1.1 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.

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

8.1.3 For products made from natural rubber, take the test portion from a milled or finely cut test sample.

8.2 Preparation of test solution

8.2.1 Ashing of test portion (destruction of organic matter)

Weigh out, to the nearest 0,1 mg, a test portion of approximately 3 g. Wrap the test portion in an ashless filter paper and place in a crucible of nominal capacity 50 cm^3. Heat the crucible containing the test portion over a bunsen burner or electric heater until all volatile material has evolved. Take care that the rubber does not ignite. If any material is lost due to spurting or frothing, repeat the above procedure with a new test portion. Place in a muffle furnace set at a temperature of 550 °C ± 25 °C (or 950 °C ± 25 °C for rubbers with a high carbon black content) and leave overnight or until all carbonaceous material is burned off.

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 and heat on a steam bath 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 (5.2).

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	5
20	2
10	1
5	0,5
0	0

8.3.2 Spectrometric measurements

Switch on the spectrometer 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.

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 the formula:

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

where

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

$\rho(\mathrm{Mg})_\mathrm{b}$ is the magnesium 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 8.4.2), given by:

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

where V is the volume, in cubic centimetres, of test solution taken in 8.4.2.

The magnesium content of the test portion can also be calculated, in micrograms per cubic centimetre, of the test solution, by the following formula:

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

where

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

$$\rho(\mathrm{Mg})_\mathrm{t} = \frac{A_\mathrm{t} \times \rho(\mathrm{Mg})_\mathrm{n}}{A_\mathrm{n}}$$

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

$$\rho(\mathrm{Mg})_\mathrm{b} = \frac{A_\mathrm{b} \times \rho(\mathrm{Mg})_\mathrm{n}}{A_\mathrm{n}}$$

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 magnesium content closest to that of the test solution;
$\rho(\mathrm{Mg})_n$	is the magnesium 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 8.4.2), given by:

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

where V is the volume, in cubic centimetres, of the test solution taken in 8.4.2.

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 part of ISO 6101;

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 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 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.

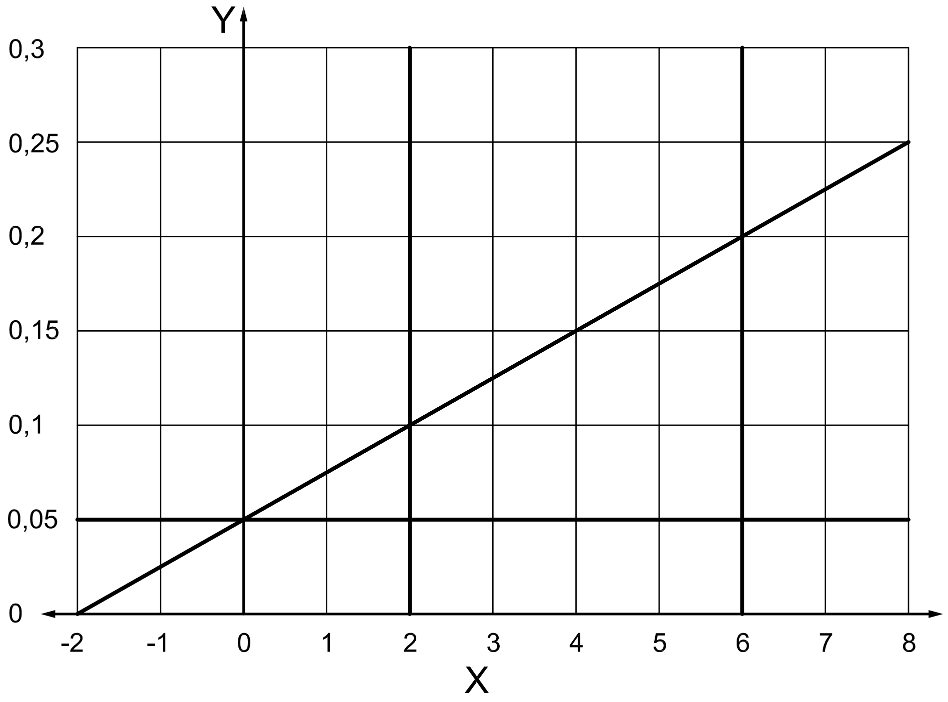
From a test solution prepared as described in 8.2, take four aliquots of the same size. To three of these aliquots, add a different, but known, volume of standard magnesium solution. Make up the volumes to the same total for all four aliquots. Use concentrations which fall on the linear portion of the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

Annex B
(informative)

Precision

The precision of the test method was determined in accordance with ISO/TR 9272[1]. Refer to this 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 2007, in which nine 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 magnesium in natural rubber latex concentrate

Average result	Within-laboratory		Between laboratories	
µg/cm^3	s_r	r	s_R	R
10,61	0,43	0,80	3,27	6,08

$r = 1{,}86 \times s_r$

where

r is the repeatability (in measurement units);

s_r is the within-laboratory standard deviation.

$R = 1{,}86 \times s_R$

where

R is the reproducibility (in measurement units);

s_R is the between-laboratory standard deviation.

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

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