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Rubber — Determination of magnesium content of raw natural rubber and rubber products by atomic absorption spectrometry

Caoutchouc — Détermination de la teneur en magnésium du caoutchouc naturel brut et des produits en caoutchouc par spectrométrie d'absorption atomique

ICS 83.040.10; 83.140.01

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Foreword

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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 11853 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Rubber — Determination of magnesium content of raw natural rubber and rubber products by atomic absorption spectrometry

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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.

CAUTION — Certain procedures specified in this International Standard may 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 International Standard 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, *Natural rubber latex concentrates – Determination of total solids content*

ISO 247, *Rubber -- Determination of ash*

ISO 648:2008, *Laboratory glassware – One mark 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 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 rubber
natural rubber usually in bales or packages, forming the starting material for the manufacturing of rubber articles

3.3
magnesium content
content of magnesium present in a sample of natural rubber latex concentrate, and in all forms of raw natural rubber and rubber product

4 Principle

A test portion is ashed at 550 ºC ± 25 ºC. The ash is dissolved in 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.

a) ***Nitric acid*** (density = 1.41 mg/m^3);

b) ***Dilute nitric acid*** (1.6% v/v) by carefully pipette 11,5 cm^3 of the nitric acid (5.a) into a 1000 cm^3 one-mark volumetric flask (6.4) and dilute to the mark with distilled water or water of equivalent purity;

c) ***Standard magnesium stock solution*,** containing 1 g of Mg per cubic decimetre; Use a commercially available standard magnesium solution.

d) ***Standard magnesium solution*,** containing 10 mg of Mg per cubic decimetre. Carefully pipette 10 cm^3 of the standard magnesium stock solution (5.c) into a 1000 cm^3 one-mark volumetric flask (6.4) dilute to the mark with dilute nitric acid (5.b) and mix thoroughly. Prepare this solution preferably on the day of use. 1 cm^3 of this standard stock solution contains 10 μg of Mg.

6 Apparatus

6.1 ***Atomic absorption spectrometer***, fitted with a burner fed with acetylene and air compressor 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 ºC ± 25 ºC.

6.4 ***One-mark volumetric flasks***, glass-stopper, of capacities 25 cm^3, 50 cm^3, 100 cm^3, 200 cm^3, 500 cm^3 and 1000 cm^3, complying with the requirements of ISO 1042:1998, class A.

6.5 ***Volumetric pipettes***, of capacities 1 cm^3, 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.

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

6.9 ***Ashless filter paper***.

7 Sampling

Carry out sampling in accordance with one of the methods specified in ISO 123 for natural rubber latex, ISO 1795 for raw, natural rubber and ISO 247 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, 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 the homogenized piece, sampled and prepared in accordance with ISO 1795.

8.1.3 For product made from natural rubber, take the test portion from the homogenized piece, sampled and prepared in accordance with ISO 247.

8.2 Preparation of test solution

8.2.1 Ashing of test portion (Destruction of organic matter)

Weigh, to the nearest 0.1 mg, approximately 3 g of rubber sample. Wrap the rubber sample in an ashless filter paper and place in an unetched crucible of nominal 50 cm^3 capacity. The crucible containing the rubber sample is heated over a bunsen burner, or electric heater, until all volatile material has been 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. Insert into muffle furnace set at a temperature of 550 ^oC ± 25 ^oC 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 (1.6% v/v) to the cooled residue. Cover with a watch glass and heat on a steam bath for at least 30 minutes. Allow to cool to ambient temperature. Filter the contents of the crucible into a 50 cm^3 standard flasks, rinsing the crucible and making up to the mark with dilute nitric acid (1.6% v/v).

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 (6.4), introduce, using pipettes (6.5) the volumes of standard magnesium solution (5.d) indicated in Table 1. Dilute to the mark with 1.6% (v/v) nitric acid (5.b) and mix thoroughly.

Prepare the set of calibration solutions on the same day prior to 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

8.4 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 rates of the air and of the acetylene in accordance with the manufacturer's instruction 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 level found in the sample of rubber being tested.

Aspirate water through the burner after each measurement.

8.5 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 fit method.

8.6 Determination

8.6.1 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.

8.7 Dilution

If the instruments response for the test solution is greater than that found for the calibration solution having the highest magnesium content, dilute, as appropriate, with 1.6% (v/v) nitric acid solution (4.1.2) in accordance with the following procedure.

Pipette carefully a suitable volume (V cm3) of the test solution into a 100 cm3 one-mark volumetric flask (6.4) so that the magnesium concentration lies within the range covered by the calibration solutions. Dilute to the mark with 1.6% (v/v) nitric acid (5.b). Repeat the spectrometric measurements.

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

8.8 Blank determination

Carry out a blank test in parallel with the determination, using 1.6% (v/v) nitric acid (5.b), but omitting the test portion.

8.9 Number of determinations

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

8.10 Expression of results

Read the magnesium content of the test solution directly from the calibration graph plotted in 8.5. The magnesium content, expresses as a percentage by mass, of the test portion, is given by the formula:

$$\frac{\rho(Mg)_t - \rho(Mg)_b}{200m} \times f$$

where

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

$\rho(Mg)_b$ 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 8.7) given by: $f = \frac{100}{V}$

where

V is the volume, in cubic centimetres, of test solutions taken in 8.7

Alternatively, the magnesium content is given by following formula:

$$\frac{\rho(Mg)_t - \rho(Mg)_b}{200m}$$

where

$\rho(Mg)_t$ is the magnesium content, expressed as a percentage by mass, in micrograms per cubic centimeter, of the test solution, given by:

$$\rho(Mg)_t = \frac{A_t \times \rho(Mg)_n}{A_n}$$

$\rho(Mg)_b$ is the magnesium content, in micrograms per cubic centimetre of the blank test solution, given by:

$$\rho(Mg)_b = \frac{A_b \times \rho(Mg)_n}{A_n}$$