INTERNATIONAL STANDARD

ISO 11852

Second edition
2017-04

Rubber — Determination of magnesium content of field natural rubber latex by titration

Caoutchouc — Détermination par titrage de la teneur en magnésium du latex de plantation de caoutchouc naturel

Reference number
ISO 11852:2017(E)

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Contents

Page

Foreword

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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).

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For an explanation on 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 the following URL: 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 second edition cancels and replaces the first edition (ISO 11852:2011), which has been technically revised to be applicable only to field latex. Therefore, all references to concentrated natural rubber latex have been removed and the following changes have been made:

— the title of this document has been changed;

— an introduction has been added to explain the advantages of the method described in this document;

— the definition of natural rubber latex concentrate has been removed from Clause 3;

— the determination of magnesium content of concentrated latex in Clause 5 has been deleted;

— because borax is a substance of high concern and reprotoxic, the buffer solution (6.4) has been changed from borax buffer solution to ammonium chloride/ammonium hydroxide buffer solution;

— the precision data for concentrated latex in Annex A have been deleted.

Introduction

In this document, no additional chemical is required to mask the interference from other divalent ions. The end-point determination in this method is easily determined since most, if not all, of the interferences have been removed during the centrifugation process. Furthermore, the chemical used for masking the interference in the alternative method has an unpleasant odour.

Another advantage of this document is that the centrifuge machine used in the method is already available in laboratory for desludging purposes. Hence, no additional cost is incurred by the laboratory to carry out the test.

Rubber — Determination of magnesium content of field natural rubber latex by titration

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 and to ensure compliance with any national regulatory conditions.

1 Scope

This document specifies a titration method for the determination of the magnesium content of field natural rubber latex.

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 385:2005, *Laboratory glassware — Burettes*

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

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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

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

3.1
magnesium content
amount of magnesium, and possibly also other alkaline-earth metals, present in a sample of natural rubber field latex

Note 1 to entry: When ammonia is added to field latex, the calcium and magnesium ions present in varying concentrations in the serum of the latex are, to a large extent, precipitated as ammonium phosphate complexes, which gradually settle out in the sludge. The results of the test method described in this document are expressed as the magnesium content on the assumption, which is not strictly true, that magnesium is the only divalent alkaline-earth ion remaining in the latex after the sludge has been removed. Calcium ions are also present, occasionally in appreciable amounts.

4 Principle

The latex is centrifuged at between 2 500 m/s^2 (250g) and 5 000 m/s^2 (500g), using a laboratory centrifuge, for 3 min. A known mass of the resultant latex, free of sludge, is diluted with water, and the residual magnesium content present in the latex is determined by titration with the disodium salt of

ethylenediaminetetraacetic acid (EDTA·Na_2) in the presence of a buffer, using Eriochrome Black T[1)] as indicator.

This method is applicable to field latex preserved with ammonia or with a combination of ammonia and formaldehyde, in which the ammonia content on titration is not less than 0,2 % of the latex. The method determines the total concentration of divalent alkaline-earth ions remaining in the latex after the removal of sludge, and this is taken as the magnesium content.

The magnesium content can be expressed either as a percentage of the mass of the latex or in milligrams per kilogram of latex.

5 Apparatus

5.1 Laboratory centrifuge, capable of producing an acceleration between 2 500 m/s^2 (250g) and 5 000 m/s^2 (500g).

5.2 Centrifuge tubes, each of at least 50 cm^3 capacity.

5.3 pH-meter, equipped with a glass electrode and a saturated calomel electrode of the sleeve or sintered-disc type and capable of reading to 0,1 pH-units. Calibrate the pH-meter by using buffer solutions of pH 4,0, 7,0 and 10,0.

5.4 Burette, of capacity 10 cm^3 or 50 cm^3, complying with the requirements of ISO 385:2005, class A.

5.5 Balance, accurate to 0,1 mg.

5.6 Volumetric pipette, of capacity 10 cm^3, complying with the requirements of ISO 648:2008, class A.

5.7 Beaker, of capacity 400 cm^3.

6 Reagents

Use reagents of recognized analytical grade and deionized water or water of equivalent purity.

6.1 Magnesium sulfate solution, 0,005 M.

Dissolve 1,231 6 g of magnesium sulfate heptahydrate ($MgSO_4·7H_2O$) in water. Make up to 1 dm^3 in a volumetric flask. 1 cm^3 of this solution is equivalent to 1 cm^3 of 0,005 mol/dm^3 EDTA·Na_2.

6.2 EDTA·Na_2 solution, 0,005 M.

6.2.1 Preparation

Dissolve approximately 1,86 g of EDTA·Na_2 in water and make up to 1 dm^3. Standardize by titrating against standard magnesium sulfate solution (6.1).

6.2.2 Standardization

Pipette 10 cm^3 of the standard magnesium sulfate solution into a beaker (5.7). Add 200 cm^3 of water and adjust the pH to 10,3 by adding buffer solution (6.4). Add about 0,1 g of Eriochrome Black T indicator (6.3) and titrate with the EDTA·Na_2 solution (6.2). The colour change is from red to permanent blue.

1) Eriochrome is a registered trademark of Huntsman Petrochemical, LLC. Eriochrome Black T is an example of suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

The concentration of the EDTA·Na_2 solution, c(EDTA·Na_2), is given, in mol/dm^3, by Formula (1):

$$c(\text{EDTA} \cdot \text{NA}_2) = \frac{10 \times 0{,}005}{V} \quad (1)$$

where V is the volume of EDTA·Na_2 solution used, in cm^3.

6.3 Eriochrome Black T indicator.

Grind together, in a small pestle and mortar, 0,3 g of Eriochrome Black T and 100 g of sodium or potassium chloride to give a homogeneous mixture.

6.4 Buffer solution.

Dissolve 67,5 g of ammonium chloride (NH_4Cl) in 250 cm^3 of deionized water, mix with 570 cm^3 of 25 % ammonium hydroxide (NH_4OH) and make up to 1 dm^3 with deionized water. The solution should have a pH of about 10,5.

7 Procedure

Carry out the procedure in duplicate, using separate test portions obtained from the same homogenized sample. If the individual results differ from their mean by more than 0,05 percentage points when the results are expressed as a percentage or by more than 5 mg/kg when the results are expressed in mg/kg, repeat the determination.

Fill each of two centrifuge tubes to 90 % of its capacity with field latex and place in the centrifuge. Balance the two tubes and centrifuge the latex for 3 min at between 2 500 m/s^2 (250g) and 5 000 m/s^2 (500g).

Taking care not to disturb the sludge at the bottom of the tube, weigh approximately 2 g to 3 g of the supernatant latex into a beaker containing 100 cm^3 of water. Mix well.

Check the pH of the latex solution and, if it is less than 10,3, add sufficient buffer solution (6.4) to raise the pH above this value. Note that, if the ammonia concentration is less than 0,35 % with respect to the latex or less than 0,50 % with respect to the water content for a latex of 30 % dry-rubber content, it is not in fact necessary to adjust the pH in this way.

Add about 0,1 g of Eriochrome Black T indicator (6.3) to the latex solution and stir. Then titrate with 0,005 mol/dm^3 EDTA·Na_2 solution (6.2) until the colour of the solution becomes blue.

NOTE 1 The end-point is a little difficult to detect with latex and it can be useful to have an over-titrated solution at hand for comparison.

NOTE 2 This test method might not be applicable to field natural rubber latex containing preservatives other than ammonia, in particular, tetramethylthiuram disulfide (TMTD) or zinc oxide (ZnO).

8 Expression of results

Calculate the magnesium content expressed as a percentage of the latex, $w_{Mg(\%)}$, using Formula (2):

$$w_{Mg(\%)} = \frac{c(\text{EDTA} \cdot \text{Na}_2) \times V \times 24{,}31}{10 \times m} \quad (2)$$

Calculate the magnesium content expressed in mg/kg of latex, w_{Mg}, using Formula (3):

$$w_{Mg} = \frac{c(\text{EDTA} \cdot \text{Na}_2) \times V \times 24{,}31 \times 10^3}{m} \quad (3)$$

where

$c(\text{EDTA}\cdot\text{Na}_2)$ is the concentration of the standardized $\text{EDTA}\cdot\text{Na}_2$ solution used, in mol/dm^3;

V is the volume of $\text{EDTA}\cdot\text{Na}_2$ solution used, in cm^3;

m is the mass of field latex taken, in g.

Take as the test result the average of the two determinations:

— rounded to two decimal places when the magnesium content is expressed as a percentage;

— rounded to the nearest whole number when the content is expressed in milligrams per kilogram.

9 Precision

See Annex A.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 11852;

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

c) the method of sampling used;

d) the type of instrument used;

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 document or in the International Standards to which reference is made, as well as any incident which might have affected the result;

h) the date of the test.

Annex A
(informative)

Precision

A.1 The precision of the test method was determined in accordance with ISO/TR 9272. Refer to ISO/TR 9272 for terminology and other statistical details.

A.2 The precision data are given in Table A.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 the 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*.

A.3 The results contained in Table A.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 2010 in which six laboratories took part, performing duplicate analyses on two samples (A and B) which were prepared from field latex.

Before the bulk 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.4 A type 1 precision was evaluated based on the method of sampling used for the interlaboratory test programme.

A.5 The repeatability, *r* (in measurement units), of the test method has been established as the appropriate value tabulated in Table A.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) are considered to come from different, or non-identical, sample populations.

A.6 The reproducibility, *R* (in measurement units), of the test method has been established as the appropriate value tabulated in Table A.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) are considered to come from different, or non-identical, sample populations.

Table A.1 — Precision data for field latex

Average result mg/kg	Within-laboratory		Between laboratories	
	s_r	r	s_R	R
123	3,72	10,53	3,76	10,63

r is the repeatability (in measurement units);
s_r is the within-laboratory standard deviation;
R is the reproducibility (in measurement units);
s_R is the between-laboratory standard deviation.

A.7 In test method terminology, bias is the difference between an average test value and a 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, therefore, cannot be determined for this particular method.

Bibliography

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

[2] ISO 17403, *Rubber — Determination of magnesium content of field and concentrated natural rubber latices by titration (cyanide-free method)*

ICS 83.040.10

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