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Rubber — Determination of magnesium content of field and concentrated natural rubber latices by titration (cyanide-free method)

Caoutchouc — Détermination par titrage de la teneur en magnésium du latex de plantation et du latex concentré de caoutchouc naturel (méthode sans cyanure)

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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).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 3, *Raw material (including latex) for use in the rubber industry*.

Rubber — Determination of magnesium content of field and concentrated natural rubber latices by titration (cyanide-free method)

1 Scope

This International Standard specifies a cyanide-free titration method for the determination of the magnesium content in field and concentrated natural rubber latex.

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

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

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, which has been subjected to some process of concentration

3.2
field natural rubber latex
natural rubber latex with or without a preservative and prior to concentration or any other processing

Note 1 to entry: The preservative is added to maintain the original state of the latex as it came from the tree.

3.3
magnesium content
content of magnesium equivalent to alkaline-earth metals, mainly magnesium and calcium, present in all soluble forms that may be titrated with ethylenediaminetetraacetic acid in field or concentrated natural rubber latex

4 Apparatus

4.1 Burette, 50 cm^3 graduated.

4.2 Analytical balance, accurate to 0,1 mg.

4.3 Volumetric pipettes, of capacities 2 cm^3, 5 cm^3 and 10 cm^3 complying with the requirements of ISO 648, class A.

5 Reagents

Use reagents of recognized analytical quality. And, wherever water is specified, use distilled water or water of equivalent purity.

5.1 Magnesium sulfate solution.

Dissolve 1,232 4 g magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$) in water and make up to 1 dm^3 in a flask. 1 cm^3 of this solution contains magnesium which is equivalent to 1 cm^3 of 0,005 mol/dm^3 EDTA.

5.2 EDTA solution, 0,005 mol/dm^3.

Dissolve approximately 1,86 g of sodium salt of ethylenediaminetetraacetic acid (EDTA) in water and make up to 1 dm^3. Standardize against the standard solution of magnesium sulfate specified in 5.1.

5.3 Masking agent solution.

Dissolve sufficient sodium hydrogen sulphide hydrate ($NaHS \cdot xH_2O$) to give at least 1,68 g sodium hydrogen sulfide (NaHS). Transfer into a 100 cm^3 volumetric flask and make up to volume with water. 1 cm^3 of this solution is equivalent to 1 cm^3 of 0,3 mol/dm^3 NaHS.

NOTE If the number of hydrate groups is not indicated (xH_2O), NaHS content may be calculated from the percentage of NaHS assay in the specification data. For example, if the percentage of NaHS assay equals 60,0 %, 2,80 g of $NaHS \cdot xH_2O$ is necessary to give 1,68 g of NaHS.

5.4 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.

5.5 Buffer solution of ammonium chloride/ammonium hydroxide.

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.

5.6 Standardization of EDTA.

Pipette 10 cm^3 of the standard magnesium sulfate solution into a beaker. Add 200 cm^3 of water and adjust the pH to 10,3 by adding 6 cm^3 of the buffer solution. Add 0,1 g of Eriochrome black T indicator and titrate with EDTA solution.

5.7 Calcium hydroxide powder.

6 Method A — Determination of magnesium content of field latex

6.1 Principle

This method determines the soluble concentration of divalent alkaline earth ions present in the latex after the dilution. The results are expressed as magnesium content on the assumption that other divalent alkaline earth ions, such as calcium, are only present at the micromolar concentration level.

The latex is diluted with water. The soluble magnesium content in the latex is determined by titration with the sodium salt of EDTA in the presence of NH_4Cl/NH_4OH buffer using sulfide releasing agent as masking agent and Eriochrome Black T as indicator.

The magnesium content is expressed as a percentage of the total solids content of latex.

6.2 Procedure

Weigh approximately 2,0 g of the field latex into a conical flask and dilute to 100 cm^3 with water.

Add 2,0 cm^3 of ammonium chloride/ammonium hydroxide buffer solution to keep the pH of latex solution between 10,0 and 10,5.

Then add 1,0 cm^3 of 0,3 mol/dm^3 NaHS to the latex solution, mix well and leave the solution for at least 10 s.

Add 0,1 g of Eriochrome black T indicator to the latex solution and mix well.

Then titrate with the standard 0,005 mol/dm^3 EDTA solution until the colour of the solution loses the last trace of red and becomes pure blue.

At the end of titration, add approximately 0,5 g of calcium hydroxide into the mixture, shake well and leave for at least 1 min before disposing.

NOTE If the pH value of the solution is higher than 11 after adding 2,0 cm^3 of ammonium chloride/ammonium hydroxide buffer solution, the volume of buffer could be reduced in order to keep pH value in the range of 10,0 to 10,5.

6.3 Number of determinations

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

6.4 Calculation of results

Calculate the magnesium content expressed as a percentage of the latex or a percentage of the total solid content using the following formula:

$$Mg_{\text{latex}} = \frac{M_{\text{EDTA}} \times V_{\text{EDTA}} \times 24{,}31 \times 100}{1\,000 \times W}$$

or

$$Mg_{\text{TSC}} = \frac{M_{\text{EDTA}} \times V_{\text{EDTA}} \times 24{,}31 \times 10\,000}{1\,000 \times W \times TSC}$$

where

Mg_{latex} is the magnesium content expressed as a percentage of the latex, in percent (%);

Mg_{TSC} is the magnesium content expressed as a percentage of the total solid content, in percent (%);

M_{EDTA} is the concentration of EDTA solution used, in moles per cubic decimetre (mol/dm^3);

V_{EDTA} is the volume of EDTA solution used, in cubic centimetres (cm^3);

W is the mass, of field latex taken, in grams (g);

TSC is the total solid content of field latex, expressed in percent (%).

6.5 Expression of result

The test result is the average of two determinations, rounded to two decimal places when the magnesium concentration is expressed as a percentage.

7 Method B — Determination of magnesium content of concentrated latex

7.1 Principle

Serum from concentrated latex is prepared by diluting 10 g of concentrated latex in 10 cm^3 water, before coagulating with acetic acid. A certain portion of serum is then buffered to pH in the range of 10,0 to 10,5. Then masking agent, NaHS, and Eriochrome black T indicator are added before titration with ethylenediaminetetraacetic acid.

7.2 Procedure

7.2.1 Test portion

Take a portion of thoroughly mixed concentrated latex containing about 10,0 g of total solids. Determine the total solid content (TSC) of concentrated latex according to ISO 124.

7.2.2 Preparation of test solution

Dilute 10,0 g of concentrated latex sample with 10,0 cm^3 water and coagulate with 5,0 cm^3 of 25 % acetic acid solution until clear serum is obtained.

Pipette 10,0 cm^3 of serum into a conical flask and add 4,0 cm^3 of ammonium chloride/ammonium hydroxide buffer solution pH 10,5 to raise the pH to the range of 10,0 to 10,5.

Add 1,0 cm^3 of masking agent solution, 0,3 mol/dm^3 NaHS, to the latex solution and mix well for at least 10 s. Add 0,1 g of Eriochrome black T indicator to the latex solution and mix well.

Titrate the residual magnesium content present in the resultant serum with the sodium salt of ethylenediaminetetraacetic acid (EDTA) in the presence of a buffer using Eriochrome Black T as indicator.

At the end of titration, add approximately 0,5 g of calcium hydroxide into the mixture, shake well and leave for at least 1 min before disposing.

NOTE If the pH value of solution is higher than 11 after adding 4,0 cm^3 of ammonium chloride/ammonium hydroxide buffer solution, the volume of buffer could be reduced in order to keep pH value in the range of 10,0 to 10,5. The end-point is a little difficult to detect with latex and it is advisable to have an over-titrated solution at hand for comparison.

7.3 Number of determinations

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

7.4 Calculation of results

Calculate the magnesium content, expressed as a percentage on the total solid content, using the following formula:

$$Mg_{\text{Based on TSC}} = \frac{M_{\text{EDTA}} \times V_{\text{EDTA}} \times 24{,}31 \times W_{\text{Total serum}} \times 100}{1000 \times V_{\text{Pipetted serum}} \times \left(\frac{W_{\text{Latex sample}} \times TSC}{100} \right)}$$

where

$Mg_{\text{Based on TSC}}$ is the magnesium content expressed as a percentage of the total solid content, in percent (%);

M_{EDTA} is the concentration of EDTA, in moles per cubic decimetre (mol/dm^3);

V_{EDTA} is the volume of EDTA in titration, in cubic centimetres (cm^3);

$V_{\text{Pipetted serum}}$ is the volume of pipetted serum, in cubic centimetres (cm^3);

$W_{\text{Latex sample}}$ is the mass of the latex sample, in grams (g);

TSC is the total solid content, expressed in percent (%);

$W_{\text{Total serum}}$ is the mass of total serum, in grams (g);

$$W_{\text{Total serum}} = W_{\text{Latex sample}} - \left(\frac{W_{\text{Latex sample}} \times TSC}{100} \right) + V_{\text{Added water}} + V_{\text{Added acid}}$$

where

$V_{\text{Added water}}$ is the volume of water added to dilute the sample at the beginning of the procedure (7.2.2), in cubic centimetres (cm^3);

$V_{\text{Added acid}}$ is the volume of 25 % acetic acid added to the serum for coagulation (7.2.2), in cubic centimetres (cm^3).

7.5 Expression of result

The test result is taken as the average of two determinations, rounded to three decimal places when the concentration is expressed in % magnesium on the TSC.

8 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 17403;

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

c) the method of sampling;

d) the type of instruments used;

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

f) any unusual features noted during the determination;

g) any operations not included in this International Standard to which reference is made, as well as any incident which might have affected the results.

Annex A
(informative)

Precision

A.1 Method A — Determination of magnesium content of field latex

A.1.1 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.

A.1.2 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 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.1.3 The results contained in Table A.1 are average values and give an estimate of the precision of this test method. The results were obtained from an interlaboratory test programme (ITP) carried out in 2012 where six laboratories took part in performing duplicate analyses on two samples namely A and B which were prepared from high-ammonia latex.

Before the bulk was sub-sampled into three bottles labelled A, B and C, it was filtered and homogenized by thorough stirring. Thus essentially samples A, B and C 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.1.4 A Type 1 precision was evaluated based on the method of sampling used for the ITP.

A.1.5 **Repeatability**: 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 *r* (for any given level) should be considered to have come from different, or non-identical, sample populations.

A.1.6 **Reproducibility**: 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 the same laboratory under normal test method procedures that differ by more than the tabulated *R* (for any given level) should be considered to have come from different, or non-identical, sample populations.

A.1.7 **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 A.1 — Level 1 and type 1 precision data for magnesium content in field latex

Material	Mean	Within-laboratory			Between laboratories			Number of laboratories
		s_r	r	(r)	s_R	R	(R)	
A	0,020	0,002	0,004	21,3	0,009	0,026	131,0	6
B	0,039	0,002	0,006	14,4	0,012	0,033	83,6	6
C	0,068	0,002	0,004	6,4	0,020	0,056	82,4	6

s_r is the within-laboratory standard deviation (in measurement units);

r is the repeatability (in measurement units);

(r) is the relative repeatability;

s_R is the between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R is the reproducibility (in measurement units);

(R) is the relative reproducibility.

A.2 Method B — Determination of magnesium content of concentrated natural rubber latex

A.2.1 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.

A.2.2 The precision data are given in Table A.2. 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.

A.2.3 The results contained in Table A.2 are average values and give an estimate of the precision of this test method. The results were obtained from an ITP carried out in 2012 where eight laboratories took part in performing duplicate analyses on two samples namely D and E which were prepared from high-ammonia latex. Before the bulk was sub-sampled into two bottles labelled D and E, it was filtered and homogenized by thorough stirring. Thus essentially, samples D and E 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.2.4 A Type 1 precision was evaluated based on the method of sampling used for the ITP.

A.2.5 Repeatability: the repeatability r (in measurement units) of the test method has been established as the appropriate value tabulated in Table A.2. Two single test results, obtained in the same laboratory under normal test method procedures that differ by more than the tabulated r (for any given level) should be considered to have come from different, or non-identical, sample populations.

A.2.6 Reproducibility: the reproducibility R (in measurement units) of the test method has been established as the appropriate value tabulated in Table A.2. Two single test results, obtained in the same laboratory under normal test method procedures that differ by more than the tabulated R (for any given level) should be considered to have come from different, or non-identical, sample populations.

A.2.7 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.