ประกาศกระทรวงอุตสาหกรรม

ฉบับที่ ๔๗๗๘ (พ.ศ. ๒๕๕๙)

ออกตามความในพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม

พ.ศ. ๒๕๑๑

เรื่อง กำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม

ยาง - การหาปริมาณแมกนีเซียมในน้ำยางสดและน้ำยางข้นโดยการไทเทรต

อาศัยอำนาจตามความในมาตรา ๑๕ แห่งพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม พ.ศ. ๒๕๑๑ ซึ่งแก้ไขเพิ่มเติมโดยพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม (ฉบับที่ ๗) พ.ศ. ๒๕๕๘ รัฐมนตรีว่าการกระทรวงอุตสาหกรรมออกประกาศกำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม ยาง - การหาปริมาณแมกนีเซียมในน้ำยางสดและน้ำยางข้นโดยการไทเทรต มาตรฐานเลขที่ มอก. 2658 - 2558 ไว้ดังมีรายละเอียดต่อท้ายประกาศนี้

ทั้งนี้ ให้มีผลตั้งแต่วันที่ประกาศในราชกิจจานุเบกษาเป็นต้นไป

ประกาศ ณ วันที่ ๑ กุมภาพันธ์ พ.ศ. ๒๕๕๙

อรรชกา สีบุญเรือง

รัฐมนตรีว่าการกระทรวงอุตสาหกรรม

มอก. 2658–2558

ISO 11852:2011

มาตรฐานผลิตภัณฑ์อุตสาหกรรม
ยาง – การหาปริมาณแมกนีเซียมในน้ำยางสดและน้ำยางข้นโดยการไทเทรต

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ กำหนดขึ้นโดยรับ ISO 11852:2011 Rubber – Determination of magnesium content of field and concentrated natural rubber latex by titration มาใช้โดยวิธีพิมพ์ซ้ำ (reprinting) ในระดับเหมือนกันทุกประการ (identical) โดยใช้ ISO ฉบับภาษาอังกฤษเป็นหลัก

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ กำหนดวิธีหาปริมาณแมกนีเซียมของน้ำยางสดและน้ำยางข้นโดยวิธีไทเทรต

รายละเอียดให้เป็นไปตาม ISO 11852:2011

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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 11852 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*.

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

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.

1 Scope

This International Standard specifies titration methods for the determination of the magnesium content of field and concentrated natural rubber latex, respectively.

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

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.

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
magnesium content
amount of magnesium, and possibly also other alkaline-earth metals (see the Note), present in a sample of natural rubber field latex or latex concentrate

NOTE 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 methods described in this International Standard 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 Field latex

4.1 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 as indicator.

This method is applicable only 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.

4.2 Apparatus

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

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

4.2.3 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 4,00, 7,00 and 10,00.

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

4.2.5 Balance, accurate to 0,1 mg.

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

4.3 Reagents

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

4.3.1 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.

4.3.2 EDTA.Na_2 solution, 0,005 M.

4.3.2.1 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 (4.3.1).

4.3.2.2 Standardization.

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 buffer solution (4.3.4). Add about 0,1 g of Eriochrome black T indicator (4.3.3) and titrate with the EDTA.Na_2 solution. The colour change is from red to permanent blue.

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

$$c(\text{EDTA.Na}_2) = \frac{10 \times 0{,}005}{V}$$

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

4.3.3 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.

4.3.4 Buffer solution.

Dissolve 4 g of borax ($Na_2B_4O_7.10H_2O$) in approximately 80 cm^3 of water. In another flask, dissolve 1 g of sodium hydroxide (NaOH) and 0,5 g of sodium sulfate (Na_2SO_4) in 10 cm^3 of water. Mix the two solutions together and make up to 100 cm^3. Ensure that all the reagents used contain less than 1 mg of magnesium per kilogram.

4.4 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 (4.3.4) 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 (4.3.3) to the latex solution and stir. Then titrate with 0,005 mol/dm^3 EDTA.Na_2 solution (4.3.2) until the colour of the solution becomes blue.

NOTE 1 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.

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

4.5 Expression of results

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

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

Calculate the magnesium content expressed in mg/kg of latex, w_{Mg}, using the following equation:

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

where

c(EDTA.Na_2) is the concentration of the standardized EDTA.Na_2 solution used, in mol/dm^3;

V is the volume of EDTA.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.

5 Concentrated latex

5.1 Principle

Approximately 10 g of concentrated latex, of which the total solids content has been determined, is diluted with 10 cm^3 of deionized water and coagulated with 5 cm^3 of 25 % acetic acid. The coagulated latex is removed, leaving behind a clear serum. The residual magnesium present in a known volume of the serum is determined by titration with the disodium salt of ethylenediaminetetraacetic acid ($EDTA.Na_2$) in the presence of a buffer, using Eriochrome Black T as indicator.

5.2 Apparatus

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

5.2.2 Balance, accurate to 0,1 mg.

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

5.3 Reagents

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

5.3.1 Dilute acetic acid, 25 % by volume.

Mix 250 cm^3 of acetic acid with deionized water and make up to 1 dm^3 with deionized water.

5.3.2 Ammonium chloride/ammonium hydroxide 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.

5.3.3 Potassium cyanide solution, (4 % by mass/volume).

Dissolve 4 g of potassium cyanide (KCN) in 100 cm^3 of deionized water.

CAUTION — Potassium cyanide is very toxic and hazardous when inhaled, when in contact with the skin and if swallowed. Contact with acids liberates a very toxic gas. Personal protective equipment shall be worn at all times. Work under a hood. Titrated solutions containing residual KCN shall be disposed of using a proper disposal procedure for hazardous chemicals.

5.4 Procedure

5.4.1 General

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.

5.4.2 Determination of total solids content

Take a portion of thoroughly mixed concentrated latex containing about 10 g of latex. Determine the total solids content of the concentrated latex in accordance with ISO 124.

5.4.3 Determination of magnesium content

Weigh accurately about 10 g of concentrated latex into a 50 cm^3 beaker and dilute with 10 cm^3 of deionized water. Coagulate with 5 cm^3 of 25 % acetic acid (5.3.1). Remove the coagulated latex, leaving behind a clear serum. Pipette 10 cm^3 of the serum into a flask and adjust the pH of the mixture with an appropriate volume of buffer solution (5.3.2) to above 10,0. Add 4 cm^3 of 4 % KCN (5.3.3). Titrate the magnesium present in the resulting solution with 0,005 mol/dm^3 EDTA.Na_2 solution (4.3.2), using Eriochrome Black T (4.3.3) as indicator, until the colour of the solution becomes blue.

5.5 Expression of results

Calculate the magnesium content expressed as a percentage of the total solids content (TSC) of the latex, $w_{Mg(\%TSC)}$, using the following equation:

$$w_{Mg(\%TSC)} = \frac{c(\text{EDTA.Na}_2) \times V \times 24{,}31 \times \frac{V_t}{V_s}}{10 \times m \times \text{TSC}}$$

Calculate the magnesium content expressed in mg/kg of the total solids in the latex, w_{Mg}, using the following equation:

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

where

c(EDTA.Na_2) is the concentration of the standardized EDTA.Na_2 solution used, in mol/ dm^3;

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

V_t is the total volume of serum produced, in cm^3 (see below);

V_s is the volume of serum used for the titration (= 10 cm^3);

m is the mass of concentrated latex taken, in g;

TSC is the total solids content of the concentrated latex, in percent.

Calculate the total volume of serum produced from the following equation:

$$V_t = \left(\frac{m - m_s}{\rho} \right) + V_w + V_a$$

where

m is the mass of concentrated latex taken, in g;

m_s is the mass of solids in the concentrated latex taken, in g, calculated from the equation $m_s = m \times \text{TSC}$;

V_w is the volume of deionized water used (= 10 cm^3);

V_a is the volume of acetic acid used (= 5 cm^3).

ρ is the density of the serum, taken as 1 Mg/m^3.

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 of the TSC;

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

6 Precision

See Annex A.

7 Test report

The test report shall include the following information:

a) a reference to this International Standard;

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 International Standard 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 statement

A.1 Field latex

A.1.1 The precision of the test method was determined in accordance with ISO/TR 9272. 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 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.1.3 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.1.4 A type 1 precision was evaluated based on the method of sampling used for the interlaboratory test programme.

A.1.5 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.1.6 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	Within-laboratory		Between laboratories	
mg/kg	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.1.7 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 cannot therefore be determined for this particular method.

A.2 Concentrated latex

A.2.1 The precision of the test method was determined in accordance with ISO/TR 9272. 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 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.2.3 The results contained in Table A.2 are average values for three different samples and give an estimate of the precision of this test method as determined in an interlaboratory test programme carried out in 2010 in which nine laboratories took part.

A.2.4 A type 1 precision was evaluated based on the method of sampling used for the interlaboratory test programme.

A.2.5 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 value of r (for any given level) are considered to come from different, or non-identical, sample populations.

A.2.6 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 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.2 — Precision data for concentrated latex

Sample	Average result	Within-laboratory		Between laboratories	
	mg/kg	s_r	r	s_R	R
A	29	1,27	3,56	3,76	10,64
B	51	2,04	5,78	4,99	14,13
C	88	2,72	7,70	9,35	26,45
For the meanings of the symbols used, see Table A.1.					

A.2.7 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 cannot therefore be determined for this particular method.

Bibliography

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

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