INTERNATIONAL STANDARD

ISO 19043

Second edition
2023-05

Natural rubber latex concentrate — Determination of total phosphate content by spectrophotometric method

Concentré de latex de caoutchouc naturel — Détermination de la teneur totale en phosphate par méthode spectrophotométrique

Reference number
ISO 19043:2023(E)

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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 document 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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For an explanation of 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 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 19043:2015), which has been technically revised.

The main changes are as follows:

— the CAS numbers of the chemicals have been added;

— in 6.4, the requirement for potassium dihydrogen phosphate (stock solution) has been changed to include both the commercially available standard solution and the prepared one;

— in 7.1, "The difference between two readings" has been changed into "The difference between the results of duplicate determinations";

— in Clause 8, "Report the result as the mean of the duplicate determinations" has been added;

— in Annex A, the precision data have been updated by the results of an ITP evaluated in accordance with ISO 19983.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Natural rubber latex concentrate — Determination of total phosphate content by spectrophotometric method

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.

1 Scope

This document specifies a method for the determination of total phosphate content of natural rubber latex concentrate. This method is not necessarily suitable for latex from natural sources other than the *Hevea brasiliensis.*

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

Approximately 20 g of concentrated latex, of which the total solids content has been determined, is coagulated with hydrochloric acid (CAS 7647-01-0).

The coagulated latex is removed and the serum filtered through filter paper.

The residual phosphate present in a known volume of the serum is determined by measuring absorbance with a spectrophotometer at wavelength 470 nm.

5 Apparatus

5.1 Balance, accurate to 0,1 mg.

5.2 Volumetric pipettes, of capacity 10 cm^3 and 25 cm^3, complying with the requirements of ISO 648, class A.

6 Reagents

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

6.1 Hydrochloric acid (CAS 7647-01-0), 370 g/kg.

6.2 Hydrochloric acid (CAS 7647-01-0) 1:24, mix 40 cm^3 of 370 g/kg hydrochloric acid (6.1) with water and make up to 1 000 cm^3.

6.3 Vanadate molybdate:

a) Dissolve 25 g ammonium molybdate (CAS 13106-76-8) in 300 cm^3 water.

b) Dissolve 1,25 g ammonium metavanadate (CAS 7803-55-6) in 300 cm^3 water. Heat to dissolve completely. Cool to room temperature before mixing with 330 cm^3 of 370 g/kg hydrochloric acid (6.1) and leave to cool.

c) Mix solutions 6.3 a) and 6.3 b) and make up to 1 000 cm^3 with water.

6.4 Potassium dihydrogen phosphate (CAS 7778-77-0) (stock solution) with a phosphorous concentration of 500 mg/dm^3. Either use a commercially available standard solution or prepare as follows:

— Dissolve 2,196 8 g potassium dihydrogen phosphate and make up to 1 000 cm^3 with water.

7 Procedure

7.1 General

Carry out the procedure in duplicate, using separate test portions obtained from the same homogenized sample. The difference between the results of duplicate determinations shall not exceed 30 mg/kg.

7.2 Determination of total solids content

Determine the total solids content of the concentrated latex in accordance with ISO 124.

7.3 Preparation of standard phosphate solutions

7.3.1 From the 500 mg/dm^3 stock solution (6.4), pipette 0 cm^3, 1 cm^3, 2 cm^3, 3 cm^3, 4 cm^3, 5 cm^3, 10 cm^3.

Then dilute with water and make up to 50 cm^3 to make the first dilution of concentration 0 mg/dm^3, 10 mg/dm^3, 20 mg/dm^3, 30 mg/dm^3, 40 mg/dm^3, 50 mg/dm^3, 100 mg/dm^3, respectively.

7.3.2 Pipette 10 cm^3 of each solution (7.3.1) into 50 cm^3 volumetric flasks.

Into each flask add 10 cm^3 vanadate molybdate (6.3).

Then make up the volume to 50 cm^3 with hydrochloric acid solution (6.2). The final concentration will be 0 mg/dm^3, 2 mg/dm^3, 4 mg/dm^3, 6 mg/dm^3, 8 mg/dm^3,10 mg/dm^3 and 20 mg/dm^3.

7.3.3 Leave the solutions (7.3.2) for 20 min and filter the total 50 cm^3 solutions through an 8 µm pore size filter paper[1)] and measure absorbance with a spectrophotometer at wavelength 470 nm. Measure in duplicate with different portions and take the average values.

Use 0 mg/dm^3 solutions as a blank.

Prepare a calibration curve by plotting the concentration of potassium dihydrogen phosphate at 0 mg/dm^3, 2 mg/dm^3, 4 mg/dm^3, 6 mg/dm^3, 8 mg/dm^3, 10 mg/dm^3 and 20 mg/dm^3 (7.3.2) against the absorbance. Determine the slope from the calibration curve (S).

7.4 Determination of phosphate content

Weigh about 20 g (m_0) of homogenous concentrated latex to the nearest 0,1 mg in a 100 cm^3 beaker. Coagulate with 25 cm^3 hydrochloric acid solution (6.2) and warm in water bath without stirring at 70 °C for 5 min to coagulate the latex completely.

Squeeze coagulum with a glass rod to obtain the serum as much as possible and filter the serum through the 8 µm pore size filter paper.

Pipette 10 cm^3 of the filtered serum (V_1) into a 50 cm^3 volumetric flask and add 10 cm^3 of vanadate molybdate (6.3), then dilute with hydrochloric acid solution (6.2) to 50 cm^3.

Wait for 20 min. Filter the solution through a filter paper.

Measure absorbance with a spectrophotometer at wavelength 470 nm against the blank (without latex).

8 Expression of results

The phosphate content, P, expressed in mg/kg, is calculated by Formula (1):

$$P = \frac{0{,}05 \times A \times V_0 \times 3{,}066\ 1 \times 1\ 000}{S \times m_1 \times V_1} \quad (1)$$

where

A is the absorbance with spectrophotometer at wavelength 470 nm;

V_0 is the volume, in cubic centimetres, of total serum calculated by Formula (2), assuming that the density of serum = 1 g/cm^3;

S is the calibration curve slope versus the phosphate concentration;

m_1 is the mass, in gram, of the dried test portion calculated by Formula (3);

V_1 is the volume, in cubic centimetres, of serum pipetted from total serum to volumetric flask;

3,066 1 is the constant of conversion of P to PO_4^{3-}.

1) Whatman No.40 is an example of a 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 this product.

Report the result as the mean of the duplicate determinations.

$$V_0 = 25 + (m_0 - m_1) \tag{2}$$

where m_0 is the mass of the test portion, in g.

$$m_1 = \frac{m_0 \times w_{TS}}{100} \tag{3}$$

where w_{TS} is the total solids content, expressed as a percentage by mass, of the concentrated latex.

9 Precision data

See Annex A.

10 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 19043:2023;

b) all details necessary for the identification of the sample;

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

d) the results and the units in which they have been expressed;

e) the date of the test;

f) any unusual features noted during the determination;

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

Annex A
(informative)

Precision data

A.1 General

The interlaboratory test programme (ITP) was conducted in China and India in 2022. The precision evaluated was a type-1 precision in accordance with method A of ISO 19983:2017, 6.7.1.

Ten laboratories participated in the ITP. Three different materials of natural rubber latex concentrate with low, medium and high phosphate content were used in the ITP. Test results from each laboratory were obtained on two different days at intervals of one week. The laboratories determined four measurements each day with two days set of tests.

A.2 Precision results

The precision results are given in Table A.1. The results were obtained using outlier deletion procedures as described in ISO 19983.

a) **Repeatability:** the difference between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test method, exceed the tabulated repeatability on average not more than once in 20 cases.

b) **Day-to-day repeatability:** the difference between two-day test (value) averages, found on nominally identical material samples under normal and correct operation of the test method, exceed the tabulated day-to-day repeatability on average not more than once in 20 cases.

c) **Reproducibility:** the difference between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, exceed the tabulated reproducibility on average not more than once in 20 cases.

Table A.1 — Precision data

Material	Mean value	Within laboratory, within-day			Within laboratory, day-to-day			Between laboratories			Number of laboratories[a]
		s_r	r	(r)	s_{rD}	r_D	(r_D)	s_R	R	(R)	
Low content	160	9	25	15,62	12	34	21,25	24	68	42,50	10
Medium content	240	11	31	12,92	17	48	20,00	35	99	51,67	10
High content	318	14	40	12,58	19	54	16,98	44	124	38,99	10

[a] The final number of laboratories in the ITP after deletion of outliers.

s_r is the repeatability standard deviation;

r is the repeatability, in measurement units;

(r) is the relative repeatability, in %;

s_{rD} is the day-to-day repeatability standard deviation;

r_D is the day-to-day repeatability, in measurement units;

(r_D) is the relative day-to-day repeatability, in %;

s_R is the reproducibility standard deviation;

R is the reproducibility, in measurement units;

(R) is the relative reproducibility, in %.