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Rubber, natural latex concentrate — Determination of KOH number

Latex concentré de caoutchouc naturel — Détermination de l'indice de potasse

Reference number
ISO 127:2018(E)

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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.

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).

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

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 fifth edition cancels and replaces the fourth edition (ISO 127:2012), which has been technically revised to add in Clause 7 the calculation of water required to dilute latex.

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.

Rubber, natural latex concentrate — Determination of KOH number

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 the KOH number of natural rubber latex concentrate which is preserved wholly or in part with ammonia.

The method is applicable to latices containing boric acid.

The method is not applicable to latices preserved with potassium hydroxide. It is not necessarily suitable for latices from natural sources other than *Hevea brasiliensis*, or for latices of synthetic rubber, compounded latex, vulcanized latex or artificial dispersions of rubber.

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 123, *Rubber latex — Sampling*

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

ISO 125, *Natural rubber latex concentrate — Determination of alkalinity*

ISO 976, *Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH*

ISO 1802, *Natural rubber latex concentrate — Determination of boric acid content*

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:

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

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

3.1
KOH number
<rubber latex> number of grams of potassium hydroxide equivalent to the acid radicals combined with ammonia in latex containing 100 g of total solids

[SOURCE: ISO 1382:2012, 2.249, modified — Note 1 to entry has been deleted.]

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water free of dissolved carbon dioxide, or water of equivalent purity.

4.1 Potassium hydroxide, standard volumetric solution, c(KOH) = 0,1 mol/dm^3, carbonate-free.

4.2 Potassium hydroxide, standard volumetric solution, c(KOH) = 0,5 mol/dm^3, carbonate-free.

4.3 Formaldehyde, 45 g to 50 g in 1 dm^3 of solution [c(HCHO) = 1,5 mol/dm^3 to 1,67 mol/dm^3], acid-free, prepared by diluting concentrated formaldehyde with water and neutralizing with 0,1 mol/dm^3 potassium hydroxide solution (4.1), using as indicator the faint pink colour of phenolphthalein.

Annex A describes the method to determine the concentration of the formaldehyde solution.

5 Apparatus

Standard laboratory glassware, plus the following.

5.1 pH-meter, in accordance with ISO 976 but capable of being read to 0,01 units.

5.2 Glass electrode, of a type suitable for use in solutions of pH up to 12,0.

5.3 Mechanical stirrer, with earthed motor and glass paddle, or **magnetic stirrer**.

An automatic titrator may be used provided that it has been proven to give the same result as the standard method.

6 Sampling

Carry out the sampling in accordance with one of the methods specified in ISO 123.

7 Procedure

Calibrate the pH-meter by the method specified in ISO 976. If the total solids, w_{TS}, and alkalinity, A, of the latex are not known, determine them in accordance with ISO 124 and ISO 125, respectively. If the latex contains boric acid and the content is not known, determine it in accordance with ISO 1802.

Carry out the determination in duplicate.

Weigh, to the nearest 0,1 g, into a 400 cm^3 beaker a test portion (mass m) of the test sample containing approximately 50 g of total solids. If necessary, adjust the alkalinity to (0,5 ± 0,1) % ammonia calculated with respect to the water phase by adding, while stirring, the required quantity of formaldehyde solution (4.3).

Calculate the volume, V_f, in cubic centimetres, of formaldehyde solution to be added from Formula (1):

$$V_f = \frac{m(100 - w_{TS})(A - 0{,}5)}{113{,}4c(\mathrm{HCHO})} \quad (1)$$

where

m is the mass, in grams, of the test portion;

w_{TS} is the total solids content, expressed as a percentage by mass, of the latex concentrate;

A is the alkalinity (water phase);

c(HCHO) is the actual concentration, expressed in moles per cubic decimetre, of the formaldehyde solution (4.3).

Dilute the latex with water to about 30 % total solids.

Calculate the volume of water, V_w, required to dilute the latex to about 30 % of the total solids by using Formula (2):

$$V_w = 166{,}7 - (m + V_f) \quad (2)$$

where

m is the mass, in grams, of the test portion;

V_f is the volume, in cubic centimetres, of formaldehyde solution;

V_w is the quantity, in grams, of water required.

Insert the electrodes of the pH-meter (5.1) into the diluted latex concentrate and record the pH.

a) If the initial pH is less than 10,3, slowly add 5 cm^3 of 0,5 mol/dm^3 potassium hydroxide solution (4.2) while stirring slowly with the glass paddle or magnetic stirrer (5.3). Record the resultant equilibrium pH reading. With continued stirring, add 0,5 mol/dm^3 potassium hydroxide solution (4.2) in 1 cm^3 increments at regular intervals (e.g. 15 s), recording the resultant equilibrium pH after each addition. Continue until the end-point has been passed.

b) If the initial pH is 10,3 or higher, add 0,5 mol/dm^3 potassium hydroxide solution (4.2) in 1 cm^3 increments at regular intervals (e.g. 15 s), recording the resultant equilibrium pH after each addition. Continue until the end-point has been passed.

The end-point of the titration is the point of inflexion of the titration curve of the pH-value against the volume, in cubic centimetres, of potassium hydroxide solution. At this point, the slope of the curve, i.e. the first differential, reaches a maximum and the second differential changes from a positive to a negative value. The end-point shall be calculated from the second differential on the assumption that the change from a positive to a negative value bears a linear relation to the addition of potassium hydroxide during the 1 cm^3 interval involved.

An example of a typical titration and the calculation of the end-point is given in Annex B.

The results of duplicate determinations shall be within 5 % (by mass).

8 Expression of results

Calculate the KOH number, K, using Formula (3):

$$K = \frac{561 c \times V}{w_{TS} \times m} \quad (3)$$

where

c is the actual concentration, expressed in moles of KOH per cubic decimetre, of the potassium hydroxide solution (4.2);

V is the volume, in cubic centimetres, of the nominally 0,5 mol/dm^3 potassium hydroxide solution (4.2) required to reach the end-point;

w_{TS} is the total solids content, expressed as a percentage by mass, of the latex concentrate;

m is the mass, in grams, of the test portion.

If the latex concentrate contains boric acid, subtract the KOH number equivalent to the boric acid from the KOH number obtained above. Calculate the KOH number equivalent to the boric acid present, K_{BA}, from Formula (4):

$$K_{BA} = 91 \times \frac{w_{BA}}{w_{TS}} \quad (4)$$

where

w_{BA} is the boric acid content, expressed as a percentage by mass;

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

9 Precision

See Annex C.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 127:2018;

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

c) all details necessary for complete identification of the pH-meter used;

d) the result obtained;

e) the correction applied for boric acid, if present;

f) details of any operation not included in this document or regarded as optional;

g) the date of the test.

Annex A
(informative)

Determination of formaldehyde

A.1 General

The method, using a standard solution of ammonia, given in the first and second editions of this document for determining the concentration of the formaldehyde solution appears not to have been widely used due to the fact that standard solutions of ammonia are considered to be unsatisfactory. Taking into account the consistent quality of analytical-grade concentrated formaldehyde solution, the majority of users prepare directly a standard solution of formaldehyde.

Where it is necessary to determine the concentration of the diluted formaldehyde, a variety of methods exist and users may refer to the *Encyclopaedia of Industrial Chemical Analysis*, Vol. 13, published by Interscience (1971). The method given below is for information only.

A.2 Reagents

A.2.1 Sodium sulfite, anhydrous, analytical grade.

A.2.2 Sulfuric acid, standard volumetric solution, $c(H_2SO_4)$ = 0,25 mol/dm^3.

A.2.3 Thymolphthalein, indicator solution.

Dissolve 80 mg of thymolphthalein in 100 cm^3 of ethyl alcohol and dilute with 100 cm^3 of distilled water.

A.3 Procedure

Prepare a solution of 125 g of anhydrous sodium sulfite (A.2.1) in 500 cm^3 of water and dilute to 1 dm^3. Transfer 100 cm^3 of the solution to a 500 cm^3 conical flask.

Accurately weigh into the flask 6,0 g to 8,0 g of the nominally 50 g/dm^3 formaldehyde solution (4.3) and swirl to mix thoroughly.

NOTE When analysing concentrated formaldehyde solution, 1,8 g to 2,0 g of solution is a more convenient amount to take.

Allow to stand for 5 min, then titrate with 0,25 mol/dm^3 sulfuric acid (A.2.2) to the first colourless end-point using thymolphthalein (A.2.3) as indicator.

Run a blank determination with the sodium sulfite solution.

A.4 Expression of results

Calculate the formaldehyde content, F, expressed as a percentage by mass, of the formaldehyde solution from Formula (A.1):

$$F = \frac{30{,}03(V_1 - V_2) \times 2c(H_2SO_4)}{10\, m_1} \qquad \text{(A.1)}$$

where