INTERNATIONAL STANDARD

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Fourth edition
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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 127:2012(E)

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Contents

Page

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 127 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 fourth edition cancels and replaces the third edition (ISO 127:1995), in which the following modifications have been undertaken:

- the normative references have been updated;
- the first sentence in Clause 8 has been modified.

It also incorporates the Amendment ISO 127:1995/Amd.1:2006, the precision data from which have been included as a new informative annex (Annex C);

Rubber, natural latex concentrate — Determination of KOH number

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 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 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 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.

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

[ISO 1382:2008]

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, $c(\text{KOH}) = 0{,}1\ \text{mol/dm}^3$, carbonate-free.

4.2 **Potassium hydroxide**, standard volumetric solution, $c(\text{KOH}) = 0{,}5\ \text{mol/dm}^3$, 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.

Determine the concentration of the formaldehyde solution as described in Annex A.

5 Apparatus

Standard laboratory glassware, plus the following:

5.1 **pH-meter**, conforming to 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 it has been checked as giving 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 the equation

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

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;

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.

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

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 (e.g. 15 s) intervals, recording the resultant equilibrium pH after each addition. Continue until the end-point has been passed.

If the initial pH is 10,3 or higher, omit the initial addition of 5 cm^3 at one time and proceed directly to “add 0,5 mol/dm^3 potassium hydroxide solution (4.2) in 1 cm^3 increments” as described above.

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 agree to within 5 % (by mass).

8 Expression of results

Calculate the KOH number, K, using the equation

$$K = \frac{561c \times V}{w_{\text{TS}} \times m}$$

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 the equation

$$K_{\text{BA}} = 91 \times \frac{w_{\text{BA}}}{w_{\text{TS}}}$$

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 International Standard;

b) all details necessary for 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 International Standard or regarded as optional;

g) the date of the test.

Annex A
(informative)

Determination of formaldehyde

The method, using a standard solution of ammonia, given in the first and second editions of this International Standard 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. Having regard to the consistent quality of analytical-grade concentrated formaldehyde solution, the majority of users prepare a standard solution of formaldehyde directly.

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

A.1 Reagents

A.1.1 Sodium sulfite, anhydrous, analytical grade.

A.1.2 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0{,}25$ mol/dm^3.

A.1.3 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.2 Procedure

Prepare a solution of 125 g of anhydrous sodium sulfite (A.1.1) 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)[1] and swirl to mix thoroughly. Allow to stand for 5 min, then titrate with 0,25 mol/dm^3 sulfuric acid (A.1.2) to the first colourless end-point using thymolphthalein (A.1.3) as indicator. Run a blank determination with the sodium sulfite solution.

A.3 Expression of results

Calculate the formaldehyde content, expressed as a percentage by mass, of the formaldehyde solution from the formula

$$\frac{30{,}03(V_1 - V_2) \times 2c(H_2SO_4)}{10m_1}$$

where

V_1 is the volume, in cubic centimetres, of sulfuric acid (A.1.2) used for the titration of the test portion of formaldehyde solution;

V_2 is the volume, in cubic centimetres, of sulfuric acid (A.1.2) required for the blank;

$c(H_2SO_4)$ is the actual concentration, in moles of H_2SO_4 per cubic decimetre, of the sulfuric acid;

m_1 is the mass, in grams, of the test portion of formaldehyde solution.

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

Annex B
(informative)

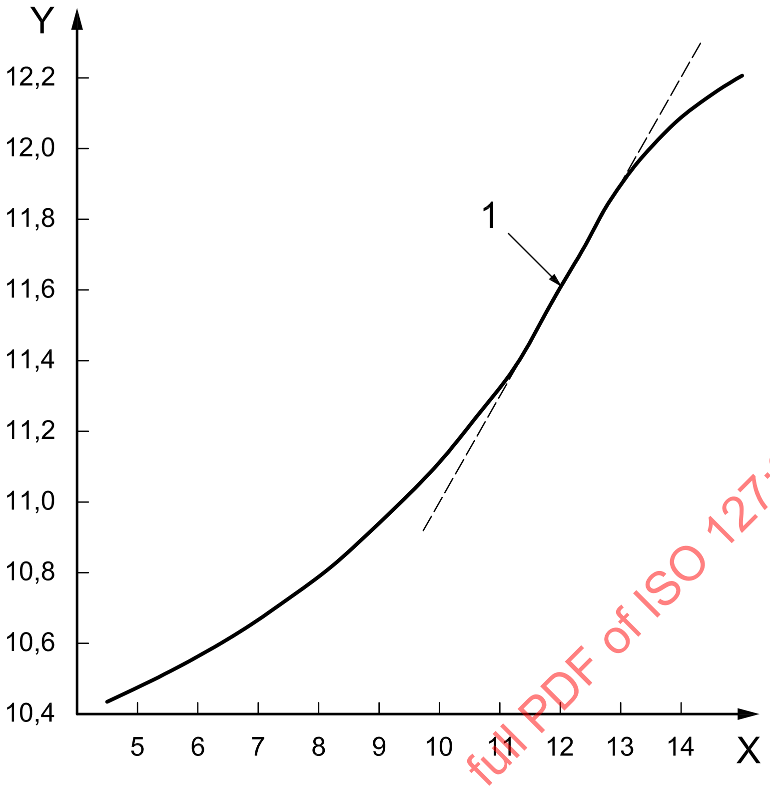
Example of a typical titration and calculation of the end-point

Table B.1 — Example of a typical titration to show variation in pH

Volume of KOH solution added cm^3	pH reading	First difference ΔpH/cm^3	Second difference ΔpH/cm^3
Initial	10,09		
5	10,46		
		0,09	
6	10,55		0,01
		0,10	
7	10,65		0,01
		0,11	
8	10,76		0,03
		0,14	
9	10,90		0,04
		0,18	
10	11,08		0,06
		0,24	
11	11,32		0,07
		0,31	
12	11,63		−0,01
		0,30	
13	11,93		−0,09
		0,21	
14	12,14		

In this example, the first difference reaches a maximum of 0,31 between 11 cm^3 and 12 cm^3 of potassium hydroxide solution. The precise point of inflexion is calculated from the ratio of the adjacent second-difference values, i.e. $0{,}07/(0{,}07 + 0{,}01) = 0{,}875$ of the difference between 11 cm^3 and 12 cm^3, i.e. 11,875 cm^3.

Figure B.1 presents these data graphically to show the point of inflexion.



Key

X volume of potassium hydroxide solution (cm^3)

Y pH

1 end-point

Figure B.1 — Illustrative curve showing change of pH during titration

Annex C
(informative)

Precision statement

C.1 Background

C.1.1 The precision of this method was determined in accordance with ISO/TR 9272:1986. Refer to this document for terminology and explanations of statistical concepts.

C.1.2 The precision details in this precision statement give an estimate of the precision of this test method with the materials used in the particular interlaboratory programme as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that the parameters are applicable to those particular materials and the specific test protocol of this test method.

C.1.3 The precision results are given in Table C.1. The precision is expressed on the basis of a 95 % confidence level for the values established for repeatability r and reproducibility R.

C.1.4 The results contained in Table C.1 are mean values and give an estimate of the precision of this test method as determined in an interlaboratory test programme (ITP) conducted in 2001. Thirteen laboratories performed triplicate analyses on two samples, A and B, which were prepared from highly ammoniated latex. The bulk latex was strained and then homogenized by thorough blending and stirring prior to being subsampled into 1-litre bottles labelled A and B. 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 which had been given to the participants in the ITP.

C.1.5 A type 1 precision was determined, based on the sampling method used for the latex samples in the ITP.

C.2 Repeatability

The repeatability r (in measurement units) of this test method has been established as the appropriate value tabulated in Table C.1. Two single test results, obtained in the same laboratory under normal test conditions, that differ by more than the tabulated value of r (for any given level) should be considered to have come from different (non-identical) sample populations.

C.3 Reproducibility

The reproducibility R (in measurement units) of this test method has been established as the appropriate value tabulated in Table C.1. Two single test results, obtained under normal test conditions, that differ by more than the tabulated value of R (for any given level) should be considered to have come from different (non-identical) sample populations.