BS ISO 976:2013

BSI Standards Publication

Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH

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National foreword

This British Standard is the UK implementation of ISO 976:2013. It supersedes BS 6057-3.9:1996 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Rubber — Raw, natural and synthetic, including latex and carbon black.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Published by BSI Standards Limited 2013

ISBN 978 0 580 77195 8
ICS 83.040.10

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 December 2013.

Amendments/corrigenda issued since publication

Date	Text affected

BS ISO 976:2013

INTERNATIONAL STANDARD

ISO 976

Fourth edition
2013-12-01

Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH

Caoutchouc et plastiques — Dispersions de polymères et latex de caoutchouc — Détermination du pH

Reference number
ISO 976:2013(E)

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Published in Switzerland

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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 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 materials (including latex) for use in the rubber industry*.

This fourth edition cancels and replaces the third edition (ISO 976:1996), which has been technically revised. It also incorporates the Amendment (ISO 976:1996/Amd.1:2006). The following are the main changes:

— the normative references were updated;

— in (subclause) 4.2, the contact details of a provider of combined electrodes were deleted;

— the precision data were updated.

INTERNATIONAL STANDARD ISO 976:2013(E)

Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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 pH of polymer dispersions and rubber latices (natural and synthetic) by means of a pH-meter equipped with a combined glass and silver reference electrode.

The method is also suitable for prevulcanized latex and compounds containing polymer dispersions or rubber latices, including adhesives.

NOTE The accuracy of the method decreases at pH values above 11.

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 123, *Rubber latex — Sampling*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Reagents

Use commercially available analytical-grade buffer solutions of known pH or, in the absence of commercial buffer solutions, prepare the solutions required (3.1, 3.2 and 3.3) using only reagents of recognized analytical grade and carbon dioxide-free distilled water or water of equivalent purity (grade 3 as defined in ISO 3696).

3.1 Buffer solution, of nominal pH 7.

Dissolve 3,40 g of potassium dihydrogen phosphate (KH_2P0_4) and 3,55 g of disodium hydrogen phosphate (Na_2HP0_4) in water and make up to 1 000 cm^3 in a volumetric flask.

The pH of this solution is 6,87 at 23 °C.

Store the solution in a glass or polyethylene vessel, which is resistant to chemicals.

3.2 Buffer solution, of pH 4.

Dissolve 10,21 g of potassium hydrogen phthalate ($KOOC\ C_6H_4\ COOH$) in water and make up to 1 000 cm^3 in a volumetric flask.

The pH of this solution is 4,00 at 23 °C.

Store the solution in a glass or polyethylene vessel, which is resistant to chemicals.

3.3 Buffer solution, of nominal pH 9

Dissolve 3,814 g of sodium tetraborate decahydrate ($Na_2B_4O_7\ 10H_2O$) in water and make up to 1 000 cm^3 in a volumetric flask.

The pH of this solution, when freshly prepared, is 9,20 at 23 °C.

Store the solution in a glass or polyethylene vessel, which is resistant to chemicals, and fitted with a soda-lime carbon dioxide trap. Replace the solution after one month.

Alkaline buffer solutions are unstable; they absorb carbon dioxide from the atmosphere. When an alkaline buffer has been used for calibration, the accuracy can be verified by means of the buffer solution of pH 4.

3.4 Reference electrolyte.

Use 3 mol/dm^3 potassium chloride solution saturated with silver chloride.

4 Apparatus

Use usual laboratory equipment and the following.

4.1 pH-meter, with an input impedance of at least 10^{12} Ω, a resolution of 0,01 pH units and equipped for temperature compensation.

4.2 Combined electrode, in which the glass electrode is surrounded concentrically by the silver reference electrode. The reference electrolyte (3.4) is kept in electrical contact with the test sample by a chemically inert diaphragm, e.g. a retractable sleeve made of polytetrafluoroethylene or glass. This is supplied with the electrode by the electrode manufacturer.

A typical combined electrode is shown in Figure 1.

The glass electrode used shall be one recommended by the manufacturer as suitable over the pH range to be encountered (0 to 14 in the case of polychloroprene latices).

Electrical contact between the electrolyte and the test sample is maintained through a thin film of electrolyte between the sleeve and the electrode.

The electrode functions linearly between pH 0 and the appearance of the alkaline error, which, depending on the sodium ion concentration, usually does not appear until the pH is over 11.

4.3 Magnetic stirrer and magnetic bar.

4.4 Electrode holder.

5 Sampling

Carry out the sampling of the rubber latex or polymer dispersion in accordance with one of the methods specified in ISO 123 or ISO 15528.

6 Procedure

6.1 General

In order to reduce thermal and electrical hysteresis effects, ensure that the temperatures of the test samples, electrode, demineralized or distilled rinsing water and buffer solutions are as close to one

another as possible. The temperatures of the test samples and buffer solutions shall not differ by more than 1 °C. The temperature for the determination shall be 23 °C ± 3 °C (27 °C ± 3 °C in tropical countries).

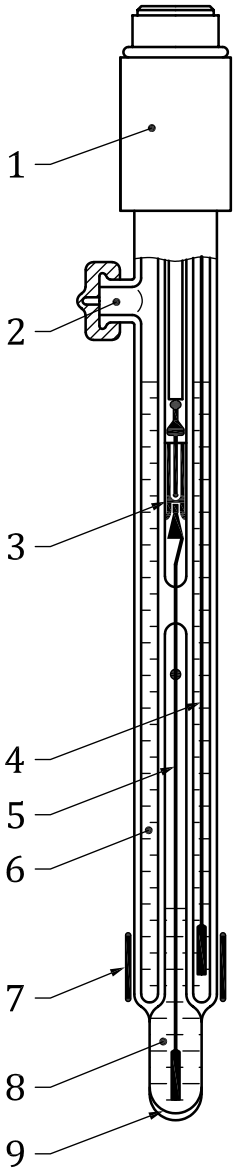
NOTE The pH variation over the range of 20 °C to 30 °C is negligible. In addition, the temperature compensator in the instrument should be set at the actual temperature.

6.2 Maintenance of the electrode

The combined electrode (4.2) shall be maintained following the manufacturer's instructions, paying particular attention to the following points.

a) Refill the electrode with the reference electrolyte (3.4) through the filling hole, first removing the filling cap if one is fitted. Slightly withdraw the seated sleeve to eliminate any latex deposits and allow a drop of the electrolyte to appear before reseating the sleeve. Prior to calibration and measurement, remove the cap on the electrolyte filling hole to allow the reference electrolyte to be at atmospheric pressure.

b) When it is not in use, keep the electrode with the junction immersed in the electrolyte.



Key

1 sliding head
2 filling hole
3 connector
4 reference electrode
5 internal electrode
6 reference electrolyte (3.4)
7 seated PTFE or glass diaphragm (sleeve)
8 internal buffer
9 membrane

Figure 1 — Typical combined electrode

6.3 Calibration of the pH-meter

6.3.1 Switch on the pH-meter (4.1) and allow the electronic circuit to stabilize. Calibrate the pH-meter following the manufacturer's instructions. If not available, proceed as follows.

6.3.2 Select two commercial buffer solutions (see Clause 3), one of nominal pH 7 (i.e. close to the zero point of the electrode) and the other differing from the first by about 3 pH units and of a higher or lower pH corresponding to the sample to be tested. In the event that commercial buffer solutions are not available, use the appropriate prepared buffer solutions (3.1 and 3.2 or 3.3).

6.3.3 Allow the temperature of the buffer solutions, the test sample, and the electrode to equilibrate at the specified temperature (see 6.1). Record the temperature and adjust the temperature correction on the pH-meter to correspond.

6.3.4 Rinse the electrode with distilled or demineralized water (see Clause 3), and then with the buffer solution of nominal pH 7, so that the liquid runs down the length of the electrode.

6.3.5 Introduce an adequate volume of the same buffer solution into a suitable clean, dry glass or inert plastic vessel. Immerse the electrode in it, taking care that the level of reference electrolyte in the electrode remains about 5 cm higher than the level of the buffer solution to prevent any contamination of the electrode.

Stir gently and allow the reading to stabilize. Adjust the pH-meter using the zero-point adjustment control, so that the reading corresponds to the pH of the buffer solution. Withdraw the electrode and discard the portion of buffer solution.

6.3.6 Rinse the electrode with water, followed by the chosen buffer solution, pH 4 (3.2) or pH 9 (3.3), as described in 6.3.4.

Instead of the prepared solution of pH 9 (3.3), commercial buffer solution with a pH in the range 9 to 11 can also be used, if available.

6.3.7 Immerse the electrode in a quantity of the chosen buffer solution as described in 6.3.5. Allow the reading to stabilize before adjusting the meter to the pH of the buffer solution, using the gradient adjustment control and without touching the zero-point control.

Ensure that the electrode gradient is in the range -55,6 mV/pH unit to - 61,5 mV/pH unit, i.e. between 95 % and 103 % of the theoretical value (- 58,57 mV/pH unit at 23 °C).

If the electrode is outside this range, carry out the maintenance procedure specified in 6.2.

Discard the portion of buffer solution.

6.4 Measurement of the pH of the test sample

6.4.1 Mix the test sample thoroughly to ensure that it is homogeneous.

6.4.2 Rinse the electrode and measuring vessel, first with distilled or demineralized water, and then with some of the sample to be tested, as described in 6.3.4. Transfer an adequate volume into the vessel (an additional clean, dry vessel can be used) and immerse the electrode in it as described in 6.3.5. Stir gently.

Allow the pH-meter reading to stabilize and record the pH.

Clean the electrode by rinsing with distilled or demineralized water to remove any latex before it dries.

6.4.3 Repeat the operations specified in 6.4.2 with a fresh portion of the test sample.

If the new reading does not differ from the first by more than 0,1 pH unit, the determination is complete.

If the two readings differ by more than 0,1 pH unit, make two further determinations, having first carried out all the checks necessary to detect any sources of error.

If a series of consecutive determinations is to be made, recalibrate the pH-meter in accordance with 6.3 at 30 min intervals or more frequently depending on the change found at each successive check.

7 Expression of results

Calculate the mean of the two readings that agree and round to the nearest 0,1 of a pH unit.

Express the results in units of pH at 23 °C if the determination was carried out at this temperature. Otherwise, specify the temperature of the determination.

8 Test report

The test report shall contain the following information:

a) a reference to this International Standard, i.e. ISO 976;

b) sufficient information to identify the sample;

c) the pH of the rubber latex or polymer dispersion, expressed to the nearest 0,1 pH unit, and the temperature of the determination;

d) any particular features noted during the test;

e) any departure from the procedures specified in this International Standard or the International Standards to which it refers, as well as any operation considered as optional;

f) the date and the place of the test.

Annex A
(informative)

Precision

A.1 General

The precision of this method was determined in accordance with ISO/TR 9272. Make reference to this Technical Report for terminology and explanations of statistical concepts.

The precision details in this precision statement provide an estimate of the precision of this test method with the materials used in the particular interlaboratory programme (ITP) 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.

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

The results in Table A.1 are mean values and give an estimate of the precision of this test method as determined in the ITP conducted in 2011. Sixteen laboratories performed duplicate 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-l 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.

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

A.2 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.3 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 different 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.4 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, therefore, cannot be determined for this particular test method.

Table A.1 — Precision data for determination of pH

Mean	Within-laboratory		Between laboratories	
	s_r	r	s_R	R
10,48	0,018	0,05	0,197	0,56

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-laboratories standard deviation

Bibliography

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

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