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pH measurement —

Part 1: Specification for pH scale

Confirmed
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Committees responsible for this British Standard

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British Pharmacopoeia Commission
Chemical Industries Association
Department of Trade and Industry (National Physical Laboratory)
Electricity Supply Industry in England and Wales
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Society of Chemical Industry
Society of Dairy Technology
Society of Glass Technology
University of Newcastle-upon-Tyne

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Contents

Foreword

This British Standard was first published in 1950 and was prepared by a technical committee directly under the aegis of the Chemical Divisional Council following recommendations made at a conference, held in 1948 at the request of the Royal Society, to discuss the need for a standard pH scale. It was revised in 1961 to extend the temperature range covered in the definition of pH from between 0 °C and 60 °C to between 0 °C and 95 °C. This revision, in two Parts, of BS 1647 has been prepared under the direction of the Laboratory Apparatus Standards Committee by the technical committee responsible for BS 2586 and BS 3145. This Part, together with Part 2, supersedes BS 1647:1961 which is withdrawn.

At the time of preparation of this revision of this British Standard, discussions were under way with a view to harmonizing the standards and recommendations were being prepared by four international organizations. These were: Subcommittee 1 (General methods) of TC 47 (Chemistry) of the International Organization for Standardization (ISO), Subcommittee D (Analysing equipment) of TC 66 (Electronic measuring equipment) of the International Electrotechnical Commission (IEC), the International Organization for Legal Metrology (OIML) and the International Union for Pure and Applied Chemistry (IUPAC).

The technical committee responsible for this Part of this standard played a leading role in this work and, as a safe-guard against the possibility of agreement not being reached in the short term, recommended the publication of this revision. It is based on a critical re-examination of some of the earlier experimental work and on new work carried out at the University of Newcastle-upon-Tyne.

The object of this Part of this standard is to ensure that, when different workers refer to a stated value of pH to two or more decimal places, they mean the same thing to within 0.01 in the temperature range 0 °C to 60 °C and approaching 0.01 at higher temperatures. It is occasionally desirable to measure and to quote pH values to more than two decimal places, but the significance of such additional figures may sometimes be uncertain. The principal change in this revision is to include more details of the procedures for making pH measurements in order that they may have the greatest possible reproducibility and repeatability.

The definition adopted for pH is an operational one, i.e. it is based on the operation used to determine it using an electrochemical cell, referred to as the "operational cell" in this Part of this standard. No simple physical interpretation of pH values can be made in terms of either hydrogen ion concentration or hydrogen ion activity except when the test solution is a dilute aqueous electrolyte solution of simple composition. For this reason, pH should be regarded as simply a reproducible scale related to "acidity", and this is all that is required for most technological purposes. For the interpretation of pH values, see Appendix A.

When the test solution contains organic matter, suspended or dissolved, reproducibility may not be high but is usually adequate for the purposes for which the pH measurement is required. The definition of the pH scale in terms of a single reference value standard solution has been reaffirmed. This definition differs from most other definitions of pH scales given in national standards (see review [1]) which, in common with the scale adopted by the National Bureau of Standards (NBS), USA [2] are multi-standard scales. Nevertheless, within 0.02, and possibly less, in the pH range 3 to 9 and the temperature range 0 °C to 60 °C, there is no practical difference between the pH scale defined in this Part of this standard and the NBS recommendations. A general account of the problems of pH standardization and measurement can be found elsewhere (see [3] and [4]).

The pH value of an unknown solution is measured with a pH meter against the pH scale specified in this standard using the operational standard solutions specified in Part 2 of this standard for calibration of the pH meter.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope

This Part of BS 1647 specifies requirements for a pH scale in terms of a single reference value standard aqueous solution of potassium hydrogen phthalate with a molality of 0.05 mol/kg, and the (theoretical) slope factor. This pH scale is appropriate for pH measurements of aqueous solutions at temperatures in the range 0 °C to 95 °C using, for calibration, the operational standard solutions specified in Part 2 of this standard.

NOTE 1 Practical pH measurements are based on one or more of the operational reference standard solutions according to circumstances and the reference value standard solution is included in the list of operational standard reference solutions from which the choice is to be made.

NOTE 2 The procedures described in Appendix B are suitable for all pH measurements, including those where other solvents and suspended matter are present, but where the electrode system is calibrated against reference materials in aqueous solution. **A.4** gives information about scales for non-aqueous and mixed aqueous solvents.

The best repeatability generally obtainable is ± 0.003 but may be considerably worse than this in solutions containing reactive materials and suspended matter, especially at the extremes of the pH range 0 to 14 and at temperatures above 60 °C.

Guidance on interpretation of pH measurements is given in Appendix A and guidance on accuracy of pH measurements is given in Appendix B.

NOTE 3 The titles of the publications referred to in this Part of this standard are listed on the inside back cover.

2 Definitions

For the purposes of this Part of BS 1647 the following definitions apply.

2.1
operational pH cell

an electrochemical cell that is the basis of practical pH measurements, consisting of an electrode responsive to hydrogen ions (hydrogen gas electrode, or glass electrode) and a reference electrode dipping into the test solution

2.2
reference electrode

an external electrode system that incorporates a filling solution and a means, such as a ceramic plug or frit, of forming the liquid junction

NOTE Usually this reference system has either a mercury/calomel (*di*mercury dichloride) or a silver/silver chloride inner element in contact with a concentrated solution of potassium chloride as filling solution.

2.3
liquid junction

any junction between two electrolyte solutions of different composition

NOTE Across such a junction there arises a potential difference called the liquid junction potential. In the operational cell (**2.1**) the junction is between the test solution or the pH standard reference solution and the filling solution or the bridge solution (**2.5**) of the reference electrode.

2.4
filling solution (of a reference electrode)

a solution containing the anion to which the reference electrode of the operational pH cell is reversible, e.g. chloride for the silver/silver chloride electrode

NOTE In the absence of a bridge solution (**2.5**), a high concentration of filling solution, comprising cations and anions of almost equal mobility, is employed as a means of maintaining a small and approximately constant liquid junction potential on substitution of the test solution for the standard reference solution(s).

2.5
bridge solution (of a double junction reference electrode)

a solution of high concentration of an inert salt, preferably comprising cations and anions of almost equal mobility, optionally interposed between the reference electrode filling solution (**2.4**) and both the test solution and the standard reference solution, when the test solution and the filling solution are chemically incompatible

NOTE This procedure introduces a second liquid junction into the operational cell and is usually formed in a similar way to the first.

2.6
residual liquid junction (potential) error

an error arising from the assumption that the liquid junction potential does not vary with the composition and concentration of the test and standard solutions

2.7
pH glass electrode

an electrode responsive to hydrogen ions, usually consisting of a bulb, or other suitable form, of special glass attached to a stem of high resistance material with internal reference electrode and internal filling solution system (see BS 2586)

NOTE Other geometrical forms may be appropriate for special applications, e.g. a capillary electrode for measuring the pH of blood.

2.8
pH meter

a high impedance instrument by means of which either the pH of a solution or the potential difference between a pair of electrodes immersed in a solution is indicated (see BS 3145).

2.9
slope factor, *k*

the temperature-dependent proportionality factor between potential and pH, calculated from the expression

(In 10) RT/F

where

R is the universal gas constant;

T is the absolute temperature;

F is the Faraday constant.

NOTE The slope factor is tabulated in Table 3.

2.10
pH reference value standard solution

a solution that is the basis for the definition of a pH scale in the temperature range 0 °C to 95 °C

2.11
pH operational reference standard solutions

solutions with pH values assigned by the operational cell method (see Part 2 of this standard) with reference to values of the reference value pH standard at each temperature

2.12
internal reference electrode (of a pH glass electrode)

an electrode, e.g. silver/silver chloride, terminating the screened input cable of the pH meter, and in contact with the internal filling solution

2.13
test solution

the solution of which the pH value is required

2.14
accuracy class

a classification of pH measurements, the accuracy of which is designated by a number representing the limit of error expressed as a percentage of the upper limit of the normal range of pH (0 to 14) as given in Table 1

Table 1 — Accuracy classes for pH measurements

Accuracy class	Tolerance of pH value, ±
0.02	0.003
0.1	0.014
0.3	0.042
1.5	0.20

3 pH scale

The pH scale shall be a straight line drawn through a single pH value at a particular temperature specified for the reference value standard solution as specified in clause **4** and having a slope factor as specified in clause **5**.

NOTE The procedure by which pH values have been assigned to this reference value standard solution is that specified in Part 2 of this standard. The convention of Bates and Guggenheim [5] has been adopted for the single ion activity coefficient of the chloride ion using equation (1).

$$-\lg\gamma_{\mathrm{cl}^-} = AI^{1/2}/[1 + 1.5I^{1/2}] \qquad (1)$$

where

I is the ionic strength (not greater than 0.1 mol kg^{-1});

A is the temperature dependent parameter of the Debye — Huckel Theory ($A = 0.5108$ mol$^{-1/2}$ kg$^{1/2}$ at 25 °C).

4 Reference value standard solution

The reference value standard solution shall consist of 0.05 mol of potassium hydrogen phthalate in 1 kg of pure water, prepared as specified in Part 2 of this standard.

The pH value of the reference value standard solution at any temperature in the range 0 °C to 95 °C shall be in accordance with the following equation:

$$\mathrm{pH(S)} = A + B\,(T - 298.15) + C\,(T - 298.15)^2 \qquad (2)$$

where

T = temperature (in K);

A = 4.00480;

B = 0.0010007;

C = 0.000032745.

NOTE 1 Values of the pH of the reference value standard solution at various temperatures are given in Table 2.

NOTE 2 Values of the pH reference value standard solution for the temperature range 100 °C to 200 °C, not covered by this standard, have been reported [6].

NOTE 3 Equation (2) is based on recent work [7] taking note of earlier work [8, 9, 10].

pH values shall not differ from the values given in Table 1 by more than ± 0.003 at 25 °C, by more than ± 0.005 between 5 °C and 20 °C, by more than ± 0.01 between 30 °C and 65 °C and by more than ± 0.02 above 65 °C.

Table 2 — pH values of the reference value standard solution at various temperatures

Temperature, *t*	pH	Temperature, *t*	pH
°C		°C	
0	4.000	50	4.050
5	3.998	55	4.064
10	3.997	60	4.080
15	3.998	65	4.097
20	4.001	70	4.116
25	4.005	75	4.137
30	4.011	80	4.159
35	4.018	85	4.183
37	4.022	90	4.21
40	4.027	95	4.24
45	4.038		

5 Slope factor

The slope factor, k, shall be in accordance with the following equation:

$$k = \frac{RT}{F} \, \text{In } 10 \qquad (3)$$

where

R is the universal gas constant = 8.31441 J·K^{-1} mol^{-1};

T is the temperature (in K);

F is the Faraday constant = 9.648456×10^4 C·mol^{-1};

In 10 = 2.302585.

NOTE Values of k at various temperatures are given in Table 3.

Table 3 — Values of the slope factor

Temperature, *t*	Slope factor, *k*	Temperature, *t*	Slope factor, *k*
°C	mV	°C	mV
0	54.199	50	64.120
5	55.191	55	65.112
10	56.183	60	66.104
15	57.175	65	67.096
20	58.167	70	68.088
25	59.159	75	69.081
30	60.152	80	70.073
35	61.144	85	71.065
37	61.540	90	72.057
40	62.136	95	73.049
45	63.128		

Appendix A Interpretation of pH measurements

A.1 Activity and concentration

Originally pH was defined as referring to the hydrogen ion *concentration* of a solution, then later to the hydrogen ion *activity* of a solution. If either is represented by $[H^+]$ then

$$pH = -\lg\,[H^+] \tag{4}$$

Although this expression can be used as a notional definition and to give an interpretation to pH values under certain limiting conditions, neither the hydrogen ion concentration nor hydrogen ion activity can be rigorously derived from electromotive force measurements. Indeed, it is for this reason that the operational definition of clause **3** has gained universal acceptance.

It should be noted that pH is a dimensionless quantity. It is not strictly correct to write, in isolation, the logarithm of a quantity other than a dimensionless number, and the full form of equation (4) is either

$$\begin{aligned} pH &= -\lg \frac{m}{m_o}\,{}^{\gamma}H \text{ or} \\ pH &= -\lg \frac{c}{c_o}\,{}^{y}H \end{aligned} \tag{5}$$

where

m_o and C_o	are arbitrary constants, representing the standard state condition numerically equal to either 1 mol/kg or 1 mol/L respectively;
m	is the molality of hydrogen ion;
c	is the molarity of hydrogen ion;
γ_H, Y_H	are single ion activity coefficients of hydrogen ion on the molality and molarity scales respectively.

For most purposes the difference between molarity and molality for dilute aqueous solutions can be ignored. The effect on the pH value of expressing the concentration in these different units depends on the density of water and is about 0.001 at 25 °C rising to 0.02 at 100 °C.

A.2 The usual range of pH

The hydrogen ion and hydroxyl ion concentrations in aqueous solutions are related through their equilibrium with water molecules as follows:

$$H_2O \rightleftarrows H^+ + OH^-$$

NOTE The symbol H^+ is used to represent the hydrated hydrogen ion, which occurs as H_3O^+ and higher hydrates.

In dilute aqueous solutions, the concentration of water is negligibly affected by its dissociation or, expressed alternatively, the activity of water is effectively unity, so the equilibrium constant for the process can be formulated as

$$K_w = [H^+]\,[OH^-] \tag{6}$$

where

K_w is the autoprotolysis constant or ionic product of water.

At ambient temperatures its value is found to be close to 10^{-14} $(\text{mol/L})^2$ but it is strongly temperature-dependent.

In pure water, the dissociation is extremely small and activity coefficients are effectively unity, so at ambient temperature

$$[H^+] = [OH^-] = K_w{}^{1/2} = 10^{-7}\ \text{mol/L} \tag{7}$$

and the solution is at the neutral point, pH = 7. This value is 7.5 at 0 °C and 6.5 at 60 °C.

For a 1 mol/L solution of a strong monoprotic, inorganic acid, $[H^+]$ is approximately 10° mol/L and therefore this solution has pH = 0. For a 1 mol/L solution of a strong alkali, $[OH^-]$ is approximately 10° mol/L; therefore $[H^+] = 10^{-14}$ mol/L and pH = 14. The range of pH measurements is therefore usually considered to be 0 to 14. However, if 10 mol/L solutions of acid and alkali are considered, the range of the pH scale is approximately – 1 to 15.

A.3 Activity coefficient

The quantity pH specified in this Part of this standard and measured by the procedures described in Appendix B has no simple interpretation. However, within a restricted range of dilute aqueous solutions (not exceeding 0.1 mol/L) in the pH range 2 to 12, i.e. for solutions closely matching the concentrations and compositions of the reference value and operational reference standard solutions, the definition is such that

$$pH = -\lg\,(cy \pm/c_o) \pm 0.02 \tag{8}$$

where c denotes the hydrogen ion concentration (molarity or molality) and $y \pm$ denotes the mean ionic activity coefficient of a typical uni-univalent electrolyte in solution. The uncertainty of ± 0.02 in pH corresponds to an uncertainty of ± 3.9 % in concentration.

A.4 Non-aqueous media

Using standard solutions prepared using non-aqueous and mixed aqueous solvents it is possible to set up pH scales for other solvent systems. The reference value standard solution (or solutions, since multi-standard scales appear to be favoured) is established by the method described in Appendix B. So far, such scales are available for very few systems, e.g. methanol/water, see [11], and heavy water (D_2O), see [12]. Often for the operational measurement the reference electrode filling solution is made up in the new solvent and not in water, but this is not essential.

A.5 pH measurements on blood and body fluids

For pH measurements on blood (reference range for normal subjects: pH = 7.35 to 7.45) it is customary to calibrate the pH meter assembly with 0.025 mol/L phosphate buffer (see **E.6** of BS 1647-2:1984) or, since a two-point standardization is almost always used, also with the so-called "physiological" phosphate standard reference solution buffer (see **E.7** of BS 1647-2:1984). Both of these reference solutions have an ionic strength of 0.1 mol/L which is close to the ionic strength of blood and isotonic saline (0.15 mol/L to 0.16 mol/L) so the residual liquid junction potential is expected to be small. It seems likely that standard reference solutions with ionic strengths matching those of blood and body fluids will be developed soon (see reference [13]) and a special pH scale appropriate to this ionic strength will be required, as in the case of sea-water and various mixed aqueous solvents where residual liquid junction potentials are large. Such considerations are important if any attempt is to be made to interpret pH measurements in terms of hydrogen ion activity or concentration in order to deduce speciation in solution. An example of this is the use of pH measurements and P_{CO_2} determination to obtain hydrogen carbonate ion concentrations in blood. Such measurements are complicated also by the effect of the protein content and suspended matter on the liquid junction potential and, to overcome this, flowing junctions are often employed. Adsorption of proteins can also affect the response of glass electrodes. The precision of pH measurements of serum has been studied (see [14]) suggesting a standard deviation of repeatability and reproducibility of 0.011 and 0.026 in pH respectively. These values are of the same order as, or greater than, the systematic difference (0.013) between the operationally assigned pH(S) value for 0.025 mol/L phosphate buffer at 37 °C and that based on cells without liquid junctions. The latter have been preferred in current medical work because the value for the "physiological" pH standard reference solution aligning with the pH scale defined in this Part of this standard was not previously available. A change in the values adopted for the reference solution affects values obtained for hydrogen carbonate ion; for example, the difference of 0.013 in pH corresponds to 0.8 mmol HCO_3, or 0.17 kPa for P_{CO_2} (1.3 mmHg P_{CO_2}).

The difference lies within the uncertainty of estimation of activity coefficients and hence neither value could be said to be more fundamental than the other.

Appendix B Outline procedures for measurement of pH values

B.1 General

The procedures which should be adopted for pH measurements depend on the particular application[1] and the accuracy required in the measurement, and are conveniently divided into four accuracy classes (see **2.14**) as described in **B.2** to **B.5**. A suitable text such as that of Bates [3] should be consulted for further details.

B.2 Measurements of accuracy class 0.02 (± 0.003 in pH)

For measurements of accuracy class 0.02, use very carefully selected glass electrodes showing only small drifts with time. Most commercially available reference electrodes are not capable of giving adequate reproducibility of performance for measurements of this class. Use silver/silver chloride electrodes freshly prepared as described in BS 2586, in contact with 3.5 mol/L KCl saturated with AgCl.

Use a pH meter complying with the requirements of BS 3145 and having a scale interval of 0.005 or a representation unit of 0.001.

B.3 Measurements of accuracy class 0.1 (± 0.014 in pH)

B.3.1 For measurements of accuracy class 0.1, it may be necessary to select the electrodes to be used with care. The procedures for checking the operation of commercial pH glass electrodes and reference electrodes and of pH combination electrodes given in BS 2586 should be followed.

B.3.2 Use a pH meter conforming with the requirements of BS 3145 with a scale interval 0.05 or representation unit 0.01. Calibrate the pH meter and electrode assembly with one operational standard reference solution before and after the test solution measurement.

NOTE 1 To minimize errors from all sources, the bracketing procedure may be employed in which two operational standard reference solutions are used, one on each side of, and as close as possible to, the pH of the sample, all solutions being at the same temperature. The measurements on the two operational standard reference solutions are often used to check, and reduce if necessary, the effective value of the slope factor (see Table 1). To facilitate this, some pH meters are provided with a slope factor adjustment control which permits a correction to be made for linear but less than theoretical behaviour of the glass electrode. In order to use this facility, the isopotential control (preferably referred to as the output zero control) should be set to the pH value of the first reference solution, and the slope factor control adjusted so that the readout coincides with the pH of the second reference solution. Unless the output zero control is adjusted in this way, the slope factor control and set buffer control will not be independent. pH meters without output zero control usually have a fixed zero value of pH = 7.0. This method of correction should be used with caution as variations in liquid junction potential error, carbon dioxide contamination, alkaline errors of the glass electrode and memory effects may each lead to an apparent drop below the theoretical slope factor value. Doubts about the satisfactory functioning of the glass electrode should be resolved by carrying out the check tests described in BS 2586.

NOTE 2 Even when using the best available glass electrodes, errors due to the glass electrode may occur in alkaline solutions, particularly those containing sodium ions. Such errors may be measured at particular pH values using the procedures and test solutions described in BS 2586.

NOTE 3 When transferring between solutions, electrodes may be washed with a little of the solution in which they are to be placed. The alternative techniques of washing with distilled water and/or swabbing with filter or tissue paper are less satisfactory as they may introduce an electrostatic charge into the high impedance measuring circuit with the result that drifting potential values may be observed.

NOTE 4 Above 45 °C the calomel electrode may be subject to temperature hysteresis because of calomel disproportionation reaction, and the silver/silver chloride electrode should therefore be used above 45 °C.

NOTE 5 Suspended matter, proteins, etc. may coat the surface of the glass electrode and also interfere with the formation of the liquid junction at the reference electrode.

B.4 Measurements of accuracy class 0.3 (± 0.042 in pH)

Accuracy class 0.3 is adequate for most routine pH measurements at or near room temperature. For extreme conditions, check the performance of both glass and reference electrodes after exposure to the test solution. Use a pH meter complying with the requirements of BS 3145 with a scale interval or representation unit suitable for the measurements.

B.5 Measurements of accuracy class 1.5 (± 0.2 in pH)

Accuracy class 1.5 covers most pH measurements of a simple diagnostic nature requiring a crude index of acidity or alkalinity. Few precautions are necessary.

B.6 Expression of results

In reporting measurements of pH(*X*), the following information should be provided:

a) manufacturer and type of glass and reference electrode; method of forming the liquid junction at the reference electrode;

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[1] See, for example, BS 2690-109.

b) manufacturer, type and scale interval or representation unit (see BS 3145) of pH meter;

c) method used to calibrate the pH meter system, for example: *either*

   1) system calibrated with operational standard pH(OS) = . . . at . . . K; *or*

   2) system calibrated with 2 operational standards pH(OS) . . . and . . . at . . . K.

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BS 1647-2, *Specification for reference value standard solutions and operational reference standard solutions.*

BS 2586, *Specification for glass and reference electrodes for the measurement of pH.*

BS 2690, *Methods of testing water used in industry.*

BS 2690-109, *Alkalinity, acidity, pH value and carbon dioxide.*

BS 3145, *Specification for laboratory pH meters.*

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