

Specification for

Glass and reference electrodes for the measurement of pH

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Cooperating organizations

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Foreword

This British Standard, which has been prepared under the direction of the Laboratory Apparatus Standards Committee, was first published in 1955 in an attempt to reduce differences which had been encountered in certain characteristics of glass electrodes, such as dimensions, efficiency of performance and electrical resistance. The 1965 revision and the present revision are the result of further consideration in the light of continuing developments and technological advances.

There is a variety of shapes and configurations for the pH-sensitive region of glass electrodes, appropriate to the wide range of applications in which they are used. Accordingly, unlike the previous editions, the present revision does not require adherence to specified dimensions. Manufacturers are, however, recommended to provide certain information regarding dimensions as suggested in 8.2.

The division of electrodes into categories such as “general purpose”, “wide range”, etc. has been abandoned but, based on development of a new method of testing the performance of glass electrodes, the manufacturer should now only claim as the usable pH range of a glass electrode those limits of pH between which its performance is in accordance with the requirements of this British Standard. Furthermore, the sodium ion error of the glass electrode at selected pH values greater than 7 and the acid error at pH values less than 1 can now be readily measured by the manufacturer and checked by the user using procedures described in Appendix A.

An additional important feature of this revision is the inclusion of reference electrodes. The correct functioning of the reference electrode in pH measurements is of equal importance to that of the glass electrode, although this has not always been appreciated. Tests are now incorporated, in Appendix B, which enable the characteristics of reference electrodes to be assessed; these tests will aid in the selection of electrodes of proper design for particular applications.

In accordance with current practice, various types of connector to the pH meter are permitted. Standardization in this area is desirable and it is the hope of the technical committee responsible for this British Standard that the bayonet normal connector (BNC) type will be adopted internationally in the near future.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, 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.

Section 1. General

1 Scope

This British Standard specifies the performance requirements and other essential characteristics of glass electrodes responsive to hydrogen ions, reference electrodes and glass combination electrodes for general laboratory use.

2 References

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

3 Definitions

For the purposes of this British Standard the following definitions apply.

3.1

pH glass electrode

electrode responsive to hydrogen ions and consisting of a bulb, or other suitable form made of a special glass, attached to a stem of high resistance material, complete with internal reference electrode (see 3.9) and internal filling solution (see 3.10)

3.2

pH-sensitive region

bulb, or other suitable form, of special glass composition which makes the electrode hydrogen ion responsive

3.3

pH meter

high impedance instrument by means of which either the pH value of a solution, or the potential difference between electrodes immersed in a solution, is indicated

3.4

screened cable

coaxial shielded cable connecting the internal reference electrode system to a plug for input to the measuring device (pH meter)

NOTE Some glass electrodes may be provided with detachable screened cables.

3.5

screened (pH glass) electrode

pH glass electrode in which the screening is continued almost down the whole length of the stem of the electrode

3.6

pH combination or dual electrode

electrode in which a pH glass electrode and an external reference electrode system together with the liquid junction are combined in a single probe. In the usual construction of pH combination electrodes, the reference electrode is concentric with, and outside the stem of, the glass electrode, which does not then require additional electrical screening

3.7

zero point

value of the pH of a solution which, in combination with a stated external reference electrode, gives zero e.m.f. from the cell

3.8

slope factor

temperature dependent factor relating potential and pH, tabulated in BS 1647

3.9

internal reference electrode (of pH glass electrode)

electrode, for example silver/silver chloride, terminating the screened input cable to the pH meter, and in contact with the internal filling solution

3.10

internal filling solution (of pH glass electrode)

aqueous electrolyte solution, which may be gelled, containing a fixed concentration of hydrogen ions, i.e. a buffer solution, and a fixed concentration of the ion to which the inner reference electrode is reversible, e.g. chloride ion in the case of silver/silver chloride or calomel electrodes

3.11

liquid junction

any junction between two electrolyte solutions of different composition. Across such a junction there arises a potential difference, called the liquid junction potential. In the operational pH cell (see 3.12) the junction is between the test solution, or the pH standard solution, and the filling solution of the reference electrode

3.12

operational pH cell

electrochemical cell which is the basis of practical pH measurements, consisting of a glass electrode and a reference electrode (see 3.13) dipping in the test solution

3.13 reference electrode

external electrode system which incorporates a means of forming the liquid junction (see 3.11) such as a ceramic plug or frit. Usually, this reference electrode system has either a calomel or a silver/silver chloride inner element in contact with a concentrated solution of potassium chloride (KCl) as filling solution (see 3.17)

3.14 isopotential method

method of correcting for small differences in temperature between test and standard solutions, or between a glass and a remote external reference electrode at different temperature, based on the approximately linear temperature dependence of those contributions to the measured e.m.f. of the cell which are independent of the composition of the test and standard solutions

3.15 isopotential pH point

the pH value at which the e.m.f. of a particular pH glass electrode/reference electrode pair, comprising the operational cell (see 3.12), may be independent of temperature. It is determined as the point of intersection on a graph of e.m.f. of the operational cell versus pH value for two temperatures differing by at least 20 °C

3.16 pH glass electrode error

deviation of a pH glass electrode from the hydrogen ion response function. An example often encountered is the error due to sodium ions at alkaline pH values, which results in a low pH value being indicated

3.17 filling solution (of a reference electrode)

solution containing the anion to which the reference electrode of the operational pH cell is reversible, e.g. chloride for a silver/silver chloride electrode. In the absence of a bridge solution (see 3.18), a filling solution of a high concentration comprising cations and anions of almost equal mobility is employed as a means of maintaining the liquid junction potential small and approximately constant on substitution of test solution for standard solution(s)

3.18 bridge solution (of a double junction reference electrode)

solution of high concentration of inert salt, preferably comprising cations and anions of almost equal mobility, optionally interposed between the reference electrode filling solution (see 3.17) and both the test and pH standard solution, when the test solution and filling solution are chemically incompatible. This procedure introduces a second liquid junction into the operational cell and is usually formed in a similar way to the first

Section 2. pH glass electrodes

4 pH range and usable temperature range

4.1 pH range. The manufacturer shall state (see clauses 7 and 8) the pH range for which a glass electrode is usable. The performance of this glass electrode in the specified range shall be in accordance with the limits laid down in clause 6 and determined using the method described in Appendix A.

4.2 Temperature range. The manufacturer shall state (see clauses 7 and 8) the usable temperature range of the pH glass electrode and, if appropriate, a permissible method of heat sterilization.

NOTE Currently, laboratory pH glass electrodes are available for pH ranges 0 to 11, 0 to 12 or 0 to 14 and temperature range 0 °C to 100 °C. For special purposes, low temperature (below 0 °C) and high temperature (above 100 °C) electrodes may be available from some manufacturers.

5 General construction and electrical requirements

5.1 Electrical resistance

5.1.1 The d.c. resistance of the electrode (membrane resistance) measured at 25 °C shall not exceed 1 GΩ.

5.1.2 For special electrodes, e.g. high temperature electrodes or microelectrodes, a higher d.c. resistance is permitted provided that this is stated (see 8.1). Values at temperatures other than 25 °C may be stated, if appropriate.

NOTE The membrane resistance and insulation resistance may be determined with a meter for measuring high d.c. resistance which applies a voltage less than 500 V. Highly reproducible values are not usually obtainable because of the effect of the polarizing voltage on the glass electrode and the electrode may not be usable for some hours after the test. Alternatively and preferably, the following method may be employed, using the pH meter itself as the measuring instrument and a known high value, screened resistor ($R_s \approx 100 \text{ M}\Omega$). Immerse the pH glass electrode and reference electrode in any standard pH buffer solution at 25 °C. Record the reading (V_1) and then shunt the input with the resistor and record the new value (V_2). The glass electrode resistance is then given by $R_s(V_1 - V_2)/V_2$.

5.2 Screening, insulation and connector.

The screened cable to the internal reference electrode shall be of the low noise, flexible type and terminate in a suitable screened plug appropriate to the pH meter being used. It is recommended that the manufacturer should state [see 8.2 b)] what screened plug has been fitted to the pH glass electrode. The insulation resistance, measured using a suitable meter for high d.c. resistance, between the internal conductor to the internal reference electrode and the screen on the plug, shall be at least 1 000 times greater than the membrane resistance measured at the same temperature.

NOTE 1 A number of suitable screened plug connectors are commonly in use. These include the (television aerial) coaxial connector specified as type 1 in BS 3041-2 as well as foreign types. However, BNC connectors are strongly preferred and recommended.

NOTE 2 With some hand held pH meters, the pH combination electrode plugs directly into the input socket of the meter without the necessity for a screened cable.

5.3 Zero point. The pH value at which the e.m.f. of the electrode system with a stated external reference electrode is zero at 25 °C shall be stated together with an estimate of its tolerance.

5.4 Dimensions. Manufacturers are recommended to provide information regarding dimensions (see 8.2).

5.5 Minimum sample volume. It is recommended that the manufacturer should state the minimum sample volume for the efficient use of the electrode.

5.6 Isopotential pH point. It is recommended that the manufacturer should state the isopotential pH point with a given type of reference electrode.

5.7 Internal reference electrode and internal filling solution. For the purposes of temperature compensation by means of an isopotential method, it is recommended (see 8.2) that the manufacturer should state the internal reference electrode and internal filling solution used.

6 Performance requirements at 25 °C

6.1 Conditioning. The manufacturer shall state how the electrode shall be treated before bringing it into use [see 8.1 c)].

6.2 Usable pH range, acid solutions (pH 0 to 7). The error of the glass electrode, when tested according to the method detailed in Appendix A, and when transferred between buffer solution B6 and any of the buffer solutions B0, B1, B2, B4, B5 (see Table 2), shall not exceed a difference in pH value of 0.02. If the error in solution B0 exceeds 0.02, the lower limit of the usable pH range stated shall be 1 instead of 0.

6.3 Usable pH range, alkaline (pH > 7),

alkali-metal ion-free solutions. The error of the glass electrode when tested according to the method detailed in Appendix A and when transferred between buffer solution B6 and any of the buffer solutions B7, B9, B11, B12, B14 (see Table 2) shall not exceed the appropriate value in Table 1 corresponding to the manufacturer's stated upper limit of its usable pH range.

6.4 Usable pH range, alkaline sodium

ion-containing solutions. If the manufacturer states [see 8.2 f)] the sodium errors of a new glass electrode, then these shall be given at the stated upper limit of the usable pH range and at one or more of the sodium concentrations 10^{-2} , 10^{-1} and 10^0 mol/l, and the errors shall have been determined according to the procedure described in Appendix A.

NOTE 1 A new glass electrode is one, previously unused, which has been conditioned according to the manufacturer's instructions (see 6.1).

NOTE 2 With age, the sodium error of a glass electrode may increase and its response may become more sluggish.

7 Information to be given integrally with the pH glass electrode

The manufacturer shall provide the following information in concise form for glass electrodes (but not necessarily for combination electrodes) by means of inscriptions integral with the electrode construction (e.g. inscribed on the stem or on the cap, or by a sleeve on the screened lead):

- the usable pH range meeting the performance criteria (see 6.2 and 6.3) in solutions free of errors due to alkali metal ions (see 4.1);
- the usable temperature range of the electrode (see 4.2);
- the zero point (see 5.3).

8 Summary of information to be given in the literature provided with pH glass electrodes

8.1 Mandatory requirements. The following information shall be given:

- the information required in clause 7;
- the d.c. resistance at 25 °C or at another stated temperature if this exceeds 1 GΩ (see 5.1);
- the method of bringing the electrode into use (conditioning) (see 6.1);
- whether heat sterilization is possible and, if so, the manufacturer shall recommend a method (see 4.2).
- the tolerance on the zero point (see 5.3).

Table 1 — Maximum permitted glass electrode errors at 25 °C dependent on stated usable upper pH limit

Stated upper limit of usable range at 25 °C	Maximum permitted error at stated pH values				
	7.9 (B7)	9.4 (B9)	11.3 (B11)	12.6 (B12)	14.1 (B14)
11	0.02	0.04	0.12	—	—
12	0.02	0.02	0.06	0.18	—
13	0.02	0.02	0.03	0.07	—
14	0.02	0.02	0.03	0.05	0.10

8.2 Optional requirements. The following information shall be given:

- a) the following dimensions (with tolerances) (see 5.4):
 - 1) the overall length of stem and pH-sensitive region below the cap;
 - 2) the length of the cap into which the connecting cable is secured;
 - 3) the diameter of the cap;
 - 4) the diameter of the stem;
 - 5) the diameter of the pH-sensitive region;
 - 6) the length of cable provided;
- b) the type of plug fitted (see 5.2);
- c) the minimum sample volume (see 5.5);
- d) the internal reference electrode, and the composition of the inner filling solution (see 5.7);
- e) the isopotential (pH) point with a stated reference electrode (see 5.6);
- f) the sodium error at the stated upper limit of the usable pH range for one or more sodium concentrations 10^{-2} , 10^{-1} and 10^0 mol/l (see 6.4).

Section 3. Reference electrodes

9 Requirements

9.1 Temperature range. The manufacturer shall state (see clause 10) the usable temperature range of the reference electrode.

9.2 Electrode system. The manufacturer shall state (see clause 10) which electrode is used for the inner element, the composition and concentration of the filling solution, and the type of device used for forming the liquid junction. The composition and concentration of the bridge solution shall be stated for double junction reference electrodes.

NOTE The bridge solution is commonly used to overcome leakage of chloride from the filling solution which may contaminate the test solution. The variation of the second liquid junction potential thereby introduced, with the composition of the test and standard solutions, may be greater than for the single junction type of reference electrode.

9.3 Electrode potential

9.3.1 Manufacturer's statement. The manufacturer shall state (see 10.1) the potential difference of a new electrode at 25.0 ± 0.5 °C in concentrated potassium chloride solution measured with respect to a silver/silver chloride concentrated potassium chloride electrode as described in B.1.

9.3.2 Tolerance. The measured value of any new sample shall not differ from the manufacturer's stated value by more than ± 5 mV.

NOTE 1 With use, the potential of the electrode may change. With the common type of electrode this can arise from two sources:

- a) a change in potassium chloride concentration due to ingress of water and egress of potassium chloride;
- b) an ingress of ions from test solutions which poison the electrode.

Depending on its design, an electrode may tolerate the effects of these for a considerable time before its electrode potential is affected through a change in species concentration at the insoluble salt/metal interface, although there may also be changes in liquid junction potential.

Tests for ascertaining whether a reference electrode is prone to these effects are described in B.2.

NOTE 2 The effect described in a), which is not important with saturated potassium chloride-filled electrodes while potassium chloride crystals remain, can be minimized by storing electrodes, when out of use, in the filling solution and not in distilled water.

9.4 Leak rate. Except for sealed types, the manufacturer shall state (see 10.1) the leak rate of a typical electrode, under a specified head of filling solution, in micromoles of stated ionic constituent per 24 h, determined for chloride-filled electrodes as described in the test method detailed in B.2.3. For electrodes with other types of filling or bridge solution, an appropriate analytical method should be chosen.

9.5 Electrical resistance

9.5.1 The electrical resistance for any new electrode shall not exceed 100 k Ω .

9.5.2 The manufacturer shall state [see 10.1 g)] the electrical resistance of a typical new electrode, measured by the d.c. or a.c. method described in B.3.

9.6 Connector. It is recommended that the manufacturer should state [see 10.2 a)] the diameter of the plug (2 mm, 3 mm or 4 mm) normally fitted for the reference electrode cable termination. The preferred size is 3 mm.

NOTE Some reference electrodes may be provided with detachable cables.

9.7 Dimensions. The manufacturer is recommended to state the dimensions listed in 10.2 b).

10 Summary of information to be given in literature provided with reference electrodes

10.1 Mandatory requirements. The following information shall be given:

- a) temperature range (see 9.1);
- b) electrode of inner element (see 9.2);
- c) composition and concentration of filling solution (see 9.2);
- d) type of device for forming liquid junction (see 9.2);
- e) potential difference at 25 °C in concentrated potassium chloride solution with respect to a silver/silver chloride electrode (see 9.3);
- f) leak rate (except for sealed electrodes) at a stated head of filling solution (see 9.4);
- g) electrical resistance (see 9.5).

10.2 Optional requirements. The following information shall be given:

- a) diameter of plug fitted (see 9.6);
- b) the following dimensions (with tolerances):
 - 1) the diameter of the stem;
 - 2) the length of the stem;
 - 3) the diameter and length of the cap;
 - 4) the length of cable provided.

Section 4. pH combination or dual electrodes

11 Requirements

11.1 General performance and characteristics. The glass electrode component of a combination electrode shall comply with the requirements of 4.1 regarding pH range and performance.

The manufacturer shall provide the information required in 5.1.2 for the glass electrode component, that required in 9.2, 9.3.1, 9.4 and 9.5.2 for the reference electrode component, and for the complete electrode, the method of conditioning (see 6.1), and the zero point with tolerance (see 5.3). It is recommended that the manufacturer should supply the information required in 5.2, 5.5, 5.6, 5.7 and 6.4, the dimensions as listed in 8.2 a), and the integral labelling described in clause 7.

11.2 Temperature range. The manufacturer shall state [see 12.1 b)] the usable temperature range of a pH combination electrode.

11.3 Screening, insulation and connectors.

The screened cable to the internal reference electrode shall be of the low-noise flexible type and shall terminate in a suitable screened plug appropriate to the pH meter being used. The connection to the reference electrode may be taken to the screen of the plug or to a separate plug. The method adopted should be stated by the manufacturer. The insulation resistance, measured using a suitable meter for high d.c. resistance, between the internal conductor to the internal reference electrode and the reference electrode plug (or, if this is not provided, the screen on the plug) shall be at least 1 000 times greater than the membrane resistance measured at the same temperature. This measurement shall be carried out with the electrode dry (see note 2 to 5.2).

NOTE 1 For the purposes of the tests described in B.2, connection should be made to the screen of the plug if a separate reference electrode plug is not provided.

NOTE 2 For the purposes of the tests described in Appendix A, connection should be made only to the central pin of the screened plug and precautions should be made to ensure that potassium chloride solution does not leak from the junction and significantly change the chloride concentration of the special buffer solutions. Some electrostatic pick-up may be observed as a result of the electrode being unscreened.

NOTE 3 Some combination electrodes may be provided with detachable screened cables.

12 Summary of information to be given in literature provided with combination electrodes

12.1 Mandatory requirements for the pH glass component. The following information shall be given:

- a) the pH range (see 4.1);
- b) the temperature range (see 11.2);
- c) the zero point (see 5.3);
- d) the d.c. resistance at 25 °C or other stated temperature if resistance exceeds 1 G Ω (see 5.1);
- e) the method of bringing the electrode into use (conditioning) (see 6.1).

12.2 Mandatory requirements for the combination electrode. The following information shall be given:

- a) the electrode of the inner element (see 9.2);
- b) the composition and concentration of filling solution (see 9.2);
- c) the type of device for forming the liquid junction (see 9.2);
- d) the potential difference at 25 °C in concentrated potassium chloride solution with respect to a silver/silver chloride electrode (see 9.3);
- e) the leak rate (except for sealed electrodes) (see 9.4);
- f) the electrical resistance (see 9.5.2).

12.3 Optional requirements for the pH glass component. The following information shall be given:

- a) the dimensions with tolerances as given in 8.2 a);
- b) the type of plug fitted (see 5.2);
- c) the internal reference electrode and composition of inner filling solution (see 5.7);
- d) the sodium error at the stated upper limit of the usable pH range for one or more sodium concentrations 10^{-2} , 10^{-1} and 10^0 mol/l (see 6.4).

12.4 Optional requirements for the combination electrode. The following information shall be given:

- a) the minimum sample volume (see 5.5);
- b) the isopotential pH point (see 5.6);
- c) whether a separate reference electrode plug has been fitted and, if so, its diameter (see 11.3);
- d) the following dimensions (with tolerances) (see 5.4):
 - 1) overall length of stem and pH-sensitive region below the cap;
 - 2) the length of the cap into which the connecting cable is secured;
 - 3) the diameter of the cap;
 - 4) the diameter of the stem;
 - 5) the diameter of the pH-sensitive region;
 - 6) the length of the cable provided;
- e) the tolerance on the zero point (see 5.3);
- f) the following information integrally with the electrode:
 - 1) the usable pH range meeting the performance criteria given in 6.2 and 6.3 in solutions free of errors due to alkali metal-ions (see 4.1, 6.2 and 6.3);
 - 2) the usable temperature range of the electrode (see 11.2);
 - 3) the zero point (see 5.3).

Appendix A Method of testing performance of pH glass electrodes

A.1 Principle of the method

With the method of testing given in this appendix, the performance of a glass electrode is assessed by transferring it between two or more special buffer solutions (see Table 2). For example, transfer of an electrode between alkali-metal ion-free buffers of pH values of 6.6 and 12.6 and comparison of the measured potential difference (against the specified reference electrode) gives, by comparison with data given in Table 3, the error of the glass electrode in the solution of pH value 12.6. A similar transfer between a buffer of pH value 12.6 containing no alkali-metal ions and one of the same pH value but containing 1 mol/l of Na⁺ gives, by comparison with the data given in Table 4, the sodium error of the glass electrode at 1 mol/l of Na⁺ and pH value 12.6. The basis of the method is the indirect comparison of glass electrodes with hydrogen glass electrodes utilizing silver/silver chloride reference electrodes in cells without liquid junction. The method permits the performance of glass electrodes to be evaluated independently of that of reference electrodes with liquid junction and hence of the operational cell for pH determination.

If a glass electrode is transferred between two chloride-containing buffer solutions containing previously equilibrated silver/silver chloride electrodes, then the measured potential difference is the difference in e.m.f. between the cells:

$$\text{Ag} | \text{Ag Cl} | \text{buffer 1} + \text{chloride} | \text{pH glass electrode,}$$

$$\text{Ag} | \text{Ag Cl} | \text{buffer 2} + \text{chloride} | \text{pH glass electrode.}$$

This difference will not be the same as that between the two cells:

$$\text{Ag} | \text{Ag Cl} | \text{buffer 1} + \text{chloride} | \text{H}_2/\text{Pt,}$$

$$\text{Ag} | \text{Ag Cl} | \text{buffer 2} + \text{chloride} | \text{H}_2/\text{Pt,}$$

if the pH glass electrode shows an error in one or both of the buffers 1 and 2. A series of special buffer solutions is available as detailed in Table 2, and the correct response of a perfect electrode responding to hydrogen ions when transferred between any pair of these has been determined with hydrogen gas electrodes giving the values shown in Table 3 and Table 4. If the first of the pair of solutions is chosen such that the glass electrode can be assumed to be error-free in that solution, then subtraction of the numerical value of the measured potential difference from the appropriate value given in Table 3 or Table 4 yields directly the error of the glass electrode in the second solution in millivolts. By convention this is positive in alkaline solutions and negative in acid solutions. The error in pH value is obtained by dividing this value in millivolts by the slope factor (see 3.8). If, for example, the two solutions have nominally the same pH value but only one contains Na⁺, the error may be directly ascribed to a sodium error of the electrode.

Table 2 — Composition of special buffer solutions

Solution no.	Buffer composition			pH
B0	1 mol/l hydrochloric acid			0.1
B1	0.1 mol/l hydrochloric acid			1.1
B2	0.05 mol/l glycine hydrochloride	0.05 mol/l glycine		2.5
B4	0.02 mol/l succinic acid	0.02 mol/l sodium hydrogen succinate	0.1 mol/l sodium chloride	4.2
B5	0.02 mol/l sodium hydrogen succinate	0.02 mol/l sodium succinate	0.1 mol/l sodium chloride	5.6
B6	0.05 mol/l Bis-Tris hydrochloride	0.05 mol/l Bis-Tris		6.6
B7	0.1 mol/l Tris hydrochloride	0.05 mol/l Tris		7.9
B9	0.03 mol/l ethanolamine hydrochloride	0.02 mol/l ethanolamine		9.4
B11	0.05 mol/l piperidine hydrochloride	0.075 mol/l piperidine		11.3
B12	0.05 mol/l tetramethylammonium hydroxide	0.1 mol/l tetramethylammonium chloride		12.6
B14	0.8 mol/l tetramethylammonium hydroxide	0.1 mol/l tetramethylammonium chloride		14.1

Table 3 — Observed potential differences for a perfect electrode responding to hydrogen ions transferred between Na⁺-free solutions

Potential differences in millivolts

To:	B0	B1	B2	B4	B5	B6	B7	B9	B11	B12	B14
From: B0	0	120.2	219.9	294.7	374.5	461.8	523.3	640.0	743.2	802.6	896.3
B1	-120.2	0	99.7	174.5	254.3	341.6	403.1	519.8	623.0	682.4	776.1
B2	-219.9	-99.7	0	74.8	154.6	241.9	303.4	420.1	523.3	582.7	676.4
B4	-294.7	-174.5	-74.8	0	79.8	167.1	228.6	345.3	448.5	507.9	601.6
B5	-374.5	-254.3	-154.6	-79.8	0	87.3	148.8	265.5	368.7	428.1	521.8
B6	-461.8	-341.6	-241.9	-167.1	-87.3	0	61.5	178.2	281.4	340.8	434.5
B7	-523.3	-403.1	-303.4	-228.6	-148.8	-61.5	0	116.7	219.9	279.3	373.0
B9	-640.0	-519.8	-420.1	-345.3	-265.5	-178.2	-116.7	0	103.2	162.6	256.3
B11	-743.2	-623.0	-523.3	-448.5	-368.7	-281.4	-219.9	-103.2	0	59.4	153.1
B12	-802.6	-682.4	-582.7	-507.9	-428.1	-340.8	-279.3	-162.6	-59.4	0	93.7
B14	-896.3	-776.1	-676.4	-601.6	-521.8	-434.5	-373.0	-256.3	-153.1	-93.7	0

Table 4 — Observed potential differences for a perfect glass electrode transferred between solutions of various Na⁺ concentrations at approximately constant pH

Potential differences in millivolts

To:	BjNa2	BjNa1	BjNa0	pH
From: Bj				
B6	1.1	6.2	23.7	6.5 ± 0.2
B7	0.3	3.1	9.7	7.9 ± 0.2
B9	1.2	6.7	20.8	9.4 ± 0.2
B11	0.7	5.0	20.9	11.3 ± 0.2
B12	-2.5	-0.5	-3.0	12.6 ± 0.2
B14	-3.7	-3.2	-17.5	14.1 ± 0.2

The buffer solutions selected for pH values greater than 7 contain no alkali metal ions or alkaline earth ions. Sodium salts are then added to these in order to obtain solutions of approximately the same pH value but of various sodium ion concentrations.

NOTE 1 The buffer solutions given in Table 3 and Table 4 are described according to the following example. B6 means the solution of pH value 6.5 (the next lower whole number pH is referred to) and containing no added sodium ions. The same solution but containing 0.01 mol/l Na⁺ ions ($pNa = -\log C_{Na^+} = 2$) is referred to as B6Na2.

Compositions of the special buffer solutions are given in Table 2. Buffer solutions (BjNa2, BjNa1 and BjNa0) containing Na⁺ are made from buffer solutions B6, B7, B9 and B11 by adding sodium perchlorate monohydrate. For buffer solutions B12 and B14, perchlorate will precipitate and so sodium chloride is used. However, it is not possible at these high pH values to make up the sodium-containing buffers by direct addition of solid sodium chloride and they have to be prepared separately. The pH values of all sodium-containing buffers differ from that of the corresponding primary buffer solution by no more than ± 0.2.

The buffer solutions may be used in either of the following two ways.

- a) For testing the response at decreasing or increasing pH in the absence of sodium ions (B6 into B0 to B14).

NOTE 2 It is recommended that B6 be used as a reference solution. Direct transfers between acid and alkaline solutions may lead to greater time variations of the glass electrode readings. Values for such transfers are nevertheless given in Table 3.

NOTE 3 Tetramethylammonium hydroxide may contain small amounts of Na⁺ and K⁺ ions as impurity, so buffers prepared from it may not be entirely alkali-metal ion-free.

NOTE 4 The sodium ions present in B4 and B5 have no adverse effect, as in this pH region the sodium error of glass electrodes is negligible.

- b) For testing the response to increasing concentrations of Na⁺ at constant pH (Bj into BjNa0, BjNa1, BjNa2, where j = 6, 7, 9, 11, 12, 14).

A.2 Reagents

A.2.1 Hydrochloric acid. Volumetric reagent solutions are available commercially at 1 mol/l. These may contain a small amount of Hg(II) which is not detrimental for the purposes of this British Standard.

A.2.2 Tetramethylammonium hydroxide (TMAH) is available commercially as a 25 ± 1.5 % (m/m) aqueous solution; 25 % (m/m) corresponds to 2.792 mol/l. This solution should be titrated with 0.1 mol/l hydrochloric acid (HCl) to an endpoint of pH 3.5 to 4.5 and the masses given in A.4.1 divided by the factor [(determined molarity)/2.792]. Analysis shows typically 5 × 10⁻⁴ mol/l of Na⁺ and 1 × 10⁻⁴ mol/l of K⁺ in buffer solution B12 prepared as described in A.4.1. Sulphate, bromide and iodide may also be present, but are not detrimental in small amounts.

A.2.3 Succinic acid is available in recognized analytical reagent grade. Dry at 80 °C.

A.2.4 Glycine is available in recognized analytical reagent grade. Dry at 80 °C.

A.2.5 Bis-Tris. 2,2 Bis(hydroxymethyl)aminotris-(hydroxethyl)methane (molecular weight = 209.25). Dry at 80 °C.

A.2.6 Tris. Tris(hydroxymethyl)aminomethane (molecular weight = 121.05). Dry at 80 °C.

A.2.7 Piperidine is a volatile liquid commercially available at greater than 98 % purity which, from a freshly opened bottle, can be used without further purification. Lower grade material should be distilled (boiling point = 106 °C) in the presence of sodium hydroxide pellets and a middle fraction collected.

A.2.8 Ethanolamine is a liquid commercially available at greater than 99 % purity which can be used without further purification. Lower grade material should be distilled (boiling point = 171 °C) and a middle fraction collected.

A.2.9 Sodium hydroxide is available commercially as a 4 mol/l aqueous solution "carbonate-free".

A.2.10 Sodium chloride is available in recognized analytical reagent grade. Dry at 110 °C.

A.2.11 Sodium perchlorate is available as monohydrate.

CAUTION: Do not dry. Analysis shows that variation from monohydrate composition is insufficient to affect sodium ion concentration significantly.

A.3 Apparatus

A.3.1 Glassware. Open beakers are suitable for containing solutions for short-term tests but for extensive long-term tests more elaborate closed vessels with necks to support the electrodes are necessary.

A.3.2 Pipettes 10 ml, 20 ml, 25 ml, 50 ml and 100 ml (class B), complying with the requirements of BS 1583.

A.3.3 One mark volumetric flasks 200 ml, 250 ml, 500 ml and 1 000 ml (class B), complying with the requirements of BS 1792.

A.3.4 Reference electrodes. Silver/silver chloride electrodes are available commercially.

Alternatively, these electrodes may be prepared as described below. The standard potential is unimportant, but if desired it can be determined in buffer solution B1 against a hydrogen gas electrode taking 0.796 as the value for the mean ionic activity coefficient of hydrochloric acid (HCl) at 0.1 mol/l at 25 °C.

One reference electrode only is needed if this is transferred together with the glass electrode between solutions. Using this procedure, however, it is not possible to ascribe a slow response solely to the glass electrode unless preliminary tests have been made on the time-response of the reference electrode. It is preferable to use reference electrodes for each buffer solution, allowing them time to reach equilibrium before the glass electrode transfers are commenced. However, silver/silver chloride electrodes should not be left in tetramethylammonium hydroxide (TMAH) solutions for lengthy periods, because of their possible iodide content.

If not available, commercially, silver/silver chloride electrodes may be prepared by one of the following three methods.

a) *Electrolytic type.* Anodize pure silver wire suitably sealed into glass tube supports, in 0.1 mol/l potassium chloride or hydrochloric acid for 1 h at a current density of 1 mA/cm². Use platinum wire for the cathode.

b) *Bi-electrolytic type.* Cathodize pure platinum wire (1 mm) suitably sealed into glass tube supports, in 1 % potassium argentocyanide solution for 24 h at a current density of 1 mA/cm². Wash thoroughly in distilled water. Convert part of the deposited silver to silver chloride by anodizing in 0.1 mol/l potassium chloride solution or hydrochloric acid for 1 h at a current density of 1 mA/cm². Use platinum wire as the other electrode in both electrolyses.

c) *Thermal-electrolytic type.* Dissolve 28 g of silver nitrate in 250 ml distilled water and heat to 50 °C to 60 °C. Add, drop by drop with vigorous stirring, a solution of 6 g sodium hydroxide in 25 ml of distilled water. Cool and filter the precipitated silver oxide using a sintered glass funnel. Wash very thoroughly with distilled water, preferably by shaking with 20 changes of 100 ml of distilled water in a stoppered bottle. Filter but retain as a thick paste.

Wind 50 mm lengths of 1 mm platinum wire into helices 2 mm in diameter and 5 mm long and seal a protruding length into glass tube supports. Apply silver oxide paste to each helix and heat in a small tube furnace at 450 °C until white. Add further coats as necessary until no platinum wire in the helix remains exposed. Convert part of the silver to silver chloride as for type b).

Electrodes should be stored in fresh 0.1 mol/l potassium chloride solution for 24 h before use. Bias (intercomparison) potentials then do not usually exceed ± 0.1 mV.

Method a) may not yield electrodes of this quality because of strains present in drawn wire.

NOTE Further details on preparation can be found in Ives and Janz, *Reference Electrodes*, Academic Press N.Y. 1961.

A.3.5 Measuring equipment. Measurements may be made with a pH meter on its mV scale to avoid complications arising from slope factor variation and temperature compensation.

Since $0.01 \text{ pH} = 0.6 \text{ mV}$, a scale interval of 1 mV or representation unit of 0.1 mV is desirable (see BS 3145), even though reproducibility at this level is not achievable.

A.4 Procedure

A.4.1 Preparation of buffer solutions. Select buffer solutions of the desired pH value and sodium ion concentration (if required). Make up solutions in freshly distilled water having a conductivity of less than 0.2 mS m^{-1} at 20°C . All these buffer solutions (except B14) may be stored in closed borosilicate flasks for periods of up to 1 month. Sodium ion-free solutions can be stored for longer periods. B14 buffer solutions should be stored in polyethylene bottles. All buffer solutions from B6 to B14 will absorb carbon dioxide if left exposed to the atmosphere, but rigorous precautions to exclude it need not be adopted. Avoid use of rubber bungs and tubing in dispensing vessels. Prepare the primary range of solutions in the following way.

B1. Dilute 100 ml of 1 mol/l hydrochloric acid to 1 000 ml with the distilled water.

B2. Dissolve 7.507 g of glycine in water, add 50 ml of 1 mol/l hydrochloric acid and make up to 1 000 ml with the distilled water.

B4. Dissolve 4.724 g of succinic acid and 5.844 g of sodium chloride in a little water, add 20 ml of 1 mol/l sodium hydroxide solution (NaOH) and make up to 1 000 ml with the distilled water.

B5. Dissolve 4.724 g of succinic acid and 5.844 g of sodium chloride in a little water, add 60 ml of 1 mol/l sodium hydroxide solution and make up to 1 000 ml with water.

B6. Dissolve 20.924 g of Bis-Tris in a little water, add 50 ml of 1 mol/l hydrochloric acid and make up to 1 000 ml with water.

B7. Dissolve 18.171 g of Tris in a little water, add 100 ml of 1 mol/l hydrochloric acid and make up to 1 000 ml with water.

B9. Mix 3.054 g of ethanolamine with 30 ml of 1 mol/l hydrochloric acid, dilute with some water and make up to 1 000 ml with water. Take care in weighing: ethanolamine is a volatile liquid and fumes in the presence of hydrogen chloride.

B11. Mix 10.644 g of piperidine with 50 ml of 1 mol/l hydrochloric acid, dilute with water and make up to 1 000 ml with water.

B12. Add 100 ml of 1 mol/l hydrochloric acid to 54.7 g of 25 % TMAH¹⁾ solution and make up to 1 000 ml with water.

B14. Add 100 ml of 1 mol/l hydrochloric acid to 328.1 g of 25 % TMAH¹⁾ and make up to 1 000 ml with water.

Solutions BjNa2 where $j = 6, 7, 9$ and 11.

Take 0.351 g of sodium perchlorate dissolved in buffer solution Bj, and make up to 250 ml.

CAUTION: Solutions containing perchlorates should not be left to form solid residues. Such residues are likely to be highly explosive.

Solutions BjNa1 where $j = 6, 7, 9$ and 11.

Take 3.511 g of sodium perchlorate (see caution) and make up to 250 ml with buffer Bj.

Solutions BjNa0 where $j = 6, 7, 9$ and 11.

Take 35.11 g of sodium perchlorate (see caution) and make up to 250 ml with buffer Bj.

B12Na2. Dissolve 0.146 g of sodium chloride (NaCl) in 25 ml of 1 mol/l hydrochloric acid. Add 13.67 g of 25 % TMAH¹⁾ and make up to 250 ml with water.

B12Na1. Dissolve 1.461 g of sodium chloride in a little water, add 4.56 g of 25 % TMAH¹⁾ and make up to 250 ml with water.

B12Na0. Mix 30 ml of 1 mol/l sodium hydroxide solution, 20 ml of 1 mol/l hydrochloric acid and some water. Add 23.877 g of sodium perchlorate (see caution) and make up to 200 ml with water.

B14Na2. Dissolve 0.146 g of sodium chloride in 25 ml of 1 mol/l hydrochloric acid. Add 82.03 g of 25 % TMAH¹⁾ and make up to 250 ml with water.

B14Na1. Dissolve 1.461 g of sodium chloride in a little water, add 72.91 g of 25 % TMAH¹⁾ and make up to 250 ml with water.

B14Na0. Mix 125 ml of 4 mol/l sodium hydroxide solution, 50 ml of 1 mol/l hydrochloric acid and some water and make up to 500 ml with water.

A.4.2 Preparation of pH glass electrodes under test and testing. Condition the glass electrode before use as stated by the manufacturer (see 6.1). In transferring a glass electrode between solutions (and the reference electrode, if transferred) the procedure of washing with a little of the solution into which the electrode is to be transferred is the most satisfactory. Swabbing with tissue may lead to carryover from one solution to the next unless an intermediate wash with distilled water is employed, but introducing this wash may have an adverse effect on the time response of the electrode.

¹⁾ Mass requires adjustment if TMAH is not 25 %. [Divide required mass by (determined molarity/2.792).]

A.4.3 Treatment of results. Determine the error of the glass electrode on transferring between any two solutions by subtracting the observed potentials from the appropriate value derived from Table 3 or Table 4.

Example 1. A glass electrode was transferred between buffer solutions B6 and B12 yielding an e.m.f. difference of 332.6 mV. Comparing this with the entry (6th row, 11th column in Table 3) of 340.8 mV, then the error is 8.2 mV (equivalent to a pH too low by 0.14) at a pH value of 12.6.

Example 2. A glass electrode was transferred between buffer solutions B9 and B9Na0 yielding an e.m.f. difference of 18.0 mV. Comparing this with the entry (3rd row, 4th column in Table 4) of 20.8 mV, then the error is 2.8 mV (equivalent to a pH too low by 0.047) at a pH value of 9.4.

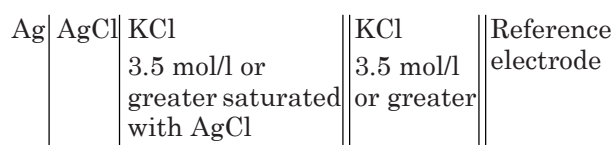
NOTE 1 The error can only be ascribed to the second solution if the electrode is known (or assumed) to be error-free in the first buffer.

NOTE 2 Values in Table 3 and Table 4 refer to a temperature of 25 °C, but no serious inaccuracy is caused if tests are made in a draught-free atmosphere at temperatures between 23 °C and 27 °C, provided that all the solutions are at the same temperature.

Appendix B Performance tests for reference electrodes

B.1 Measurement of the electrode potential of a reference electrode

Using a digital voltmeter, potentiometer or a pH meter on its millivolt scale, measure the potential difference at 25.0 ± 0.5 °C between the reference electrode and a silver-silver chloride electrode immersed in concentrated potassium chloride solution saturated with silver chloride, according to the cell scheme:



The double vertical bars indicate liquid junctions at ceramic plugs, frits or otherwise; and “reference electrode” refers to the electrode under test complete with its filling solution, and, in the case of double junction electrodes, with its bridge solution changed to concentrated potassium chloride solution (3.5 mol/l or greater). The stability of the potential of a reference electrode is more important than its actual value.

NOTE 1 Silver/silver chloride | KCl (concentrated) reference electrodes are available commercially, the potentials of which are not likely to vary by more than ± 1 mV between unused electrodes. Variation between mercury-calomel | KCl (concentrated) electrodes may be greater than this.

A new calomel reference electrode with liquid junction should show a potential difference, as determined above, of 45 ± 10 mV. Deviations after usage indicate one of the effects described in note 1 of 9.3.

NOTE 2 The inner element of a silver/silver chloride | KCl (concentrated) reference electrode can be constructed by one of the methods described in A.3.4. Ceramic plugs and frits are available commercially. Adequate coating with silver chloride and, additionally, saturation of potassium chloride with silver chloride is essential because of the increased solubility of the latter in concentrated chloride solutions.

B.2 Methods of assessing satisfactory design and performance of reference electrodes

B.2.1 Approximate constancy of liquid junction potential. Repeat the measurement described in B.1, replacing the concentrated potassium chloride solution between the electrodes firstly with a), below, and secondly with b):

- a) a buffer solution of pH 3 to pH 5 (e.g. 0.05 mol/l potassium hydrogen phthalate);
- b) a buffer solution of pH 7 to pH 10 (e.g. 0.05 mol/l sodium tetraborate).

The three values should not differ by more than 2 mV. Deviations greater than this indicate an unsatisfactory new electrode or contamination of a used electrode.

B.2.2 Ingress of water. After immersing the reference electrode in at least 100 ml of water at ambient temperature for several days with the liquid junction formed 10 mm below the water surface, repeat the measurement described in B.1. For an electrode of good design, deviation from the value measured as described in B.1 should not exceed ± 2 mV after immersion for 2 weeks.

B.2.3 Egress of electrolyte (from chloride-filled electrodes). Mount a reference electrode so that it dips 10 mm below its junction in a 10 ml sample of distilled water (see A.4.1). Measure the head of the filling solution. After a suitable period of time, e.g. 3 days, estimate the chloride lost from the electrode by either of the following methods:

- a) potentiometric titration of the water sample with 0.01 mol/l silver nitrate and a silver indicating electrode;
- b) conductance measurement of the water sample and comparison with diluted potassium chloride solutions of known strength.

Similar tests can be devised for electrodes with other filling solutions and double junction electrodes with non-chloride containing bridge solutions by choosing an appropriate analytical method.

NOTE For a cylindrical electrode with a transparent stem, an approximate indication of the filling solution leak rate can be obtained by measuring the drop in height of the surface of the filling solution over a period of days or weeks. This is a measure of the volume loss and is not necessarily equivalent to the salt loss.

B.2.4 *Ingress of ions from test solutions*

a) *Non-destructive test* (unsealed electrodes only). Immerse the electrode in 0.1 mol/l sodium fluoride solution. After 48 h to 60 h, remove the filling solution (or the bridge solution for double liquid junction electrodes) with a syringe attached to catheter tubing. Using the cell:



determine the fluoride concentration by comparison with measurements on suitable test solutions made by adding known amounts of 0.1 mol/l sodium fluoride solution to portions of the filling or bridge solution. Calculate the ingress rate as micromoles of fluoride per 24 h.

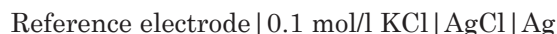
b) *Destructive test* (sealed and unsealed electrodes). Mount the reference electrode in a closed vessel containing 100 g/l solution of ammonium sulphide. After 2 weeks, remove and rinse the electrode with distilled water. Repeat the measurement described in **B.1**.

NOTE A change in potential difference of more than ± 6 mV (equivalent to a difference in pH value of 0.1) is indicative of a reference electrode which would be prone to poisoning if exposed continuously in some flow analysis applications.

B.3 **Electrical resistance**

Carry out either of the procedures described in **B.3.1** or **B.3.2**.

B.3.1 Place the reference electrode in 0.1 mol/l potassium chloride solution with a silver/silver chloride electrode (see **A.2.5**) according to the cell:



Measure the potential difference:

- directly;
- after 10 s, while passing a current of 0.1 μA through the cell;
- after 10 s, while passing a current of 0.1 μA in the reverse direction through the cell.

NOTE 1 A change in potential of ± 10 mV corresponds to a resistance of less than 100 k Ω .

NOTE 2 A suitable source of current would be a 1.5 V dry battery in series with a 15 M Ω resistor.

B.3.2 Place the reference electrode in a solution of 4.0 mol/l potassium chloride saturated with silver chloride and introduce a silver/silver chloride electrode (see **A.3.2**). Apply an a.c. voltage (not exceeding 5 V at 50 Hz), and measure the a.c. current. Calculate the resistance of the electrode from the ratio of the r.m.s. voltage to the r.m.s. current.

NOTE 1 A commercially available conductivity or resistance meter may be used provided it applies the specified a.c. voltage.

NOTE 2 For reference electrodes having very small surface areas, use the method described in **B.3.2**. Polarization may occur in the method described in **B.3.1**.

Publications referred to

BS 1583, *One-mark pipettes*.

BS 1647, *pH scale*²⁾.

BS 1792, *One-mark volumetric flasks*.

BS 3041, *Radio-frequency connectors*.

BS 3041-2, *Specification for coaxial unmatched connector*.

BS 3145, *Laboratory pH meters*.

²⁾ In course of revision.

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