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1984

*Incorporating*
*Amendment No. 1*

# pH measurement —

## Part 2: Specification for reference value standard solutions and operational reference standard solutions

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# Committees responsible for this British Standard

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# Contents

# Foreword

This revision, in two Parts, of BS 1647 has been prepared under the direction of the Laboratory Apparatus Standards Committee. This Part together with Part 1, supersedes BS 1647:1961 which is withdrawn.

Part 1 of this standard explains the development of the standard and specifies the pH scale.

**Summary of pages**

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 16, 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.

## 0 Introduction

As specified in Part 1 of this standard, the reference value standard solution is an aqueous solution of potassium hydrogen phthalate with a molality of 0.5 mol/kg.

Operational pH standards are those assigned pH values at various temperatures based on the operational cell measurements specified in clause **4**, where the operational reference standard is solution *X* and the reference value standard is solution *S*. Values assigned by this method for 15 operational reference standards are given in Table 1 for various temperatures in the range 0 °C to 60 °C. Above 60 °C values have been obtained by extrapolation of the smoothing equations given in Table 2.

NOTE 1 pH values of operational standards are based on the work reported elsewhere [1, 2], using liquid junctions formed under highly reproducible conditions.

NOTE 2 Within the pH range 3 to 9, pH values assigned to operational reference standard solutions using the cell without a liquid junction (the method described in Part 1 of this standard for assigning the pH to the reference value standard solution) are within ± 0.02 of those given in Table 1 because the residual liquid junction errors are small. Some useful values are tabulated in Table 3. Outside the pH range 3 to 9, differences may be appreciable and operationally-derived values are preferred. For work of low accuracy class, many of the values relating to solutions with specialist application found in the literature could be employed [3].

## 1 Scope

This Part of BS 1647 specifies the preparation of the reference value standard solution used as the basis of the pH scale specified in Part 1 of this standard and requirements for operational reference standard solutions suitable for calibration of pH meters against this pH scale. It also specifies accuracy classes for pH measurements.

NOTE 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 definitions given in Part 1 of this standard apply.

## 3 Preparation of the reference value standard solution

The purity of the material used in the preparation of the reference value standard solution shall be such that the pH value of the solution, when determined by the method described in Appendix A, is 4.005 ± 0.003 at 25 °C.

The reference value standard solution shall be prepared as described in Appendix B.

NOTE Methods of independently checking the purity of the reference value standard material are described in Appendix C and Appendix D.

## 4 Operational reference standard solution

The pH values of the operational reference standard solutions at temperatures between 0 °C and 60 °C shall be in accordance with the following equation:

$$\mathrm{pH}(X) = \frac{E(S) - E(X)}{k} + \mathrm{pH}(S) \qquad (1)$$

where

*k* is the slope factor;

pH(*S*) is the pH value of the reference value standard solution;

*E(S)* and *E(X)* are the electromotive forces of the operational cells

Reference electrode $||\mathrm{X}|\mathrm{H}_2|\mathrm{Pt}$

Reference electrode $||\mathrm{S}|\mathrm{H}_2|\mathrm{Pt}$

The electromotive forces *E(X)* and *E(S)* of the operational cells shall be measured consecutively, both cells being at the same temperature throughout, and the same reference electrode shall be used in each cell.

NOTE 1 The hydrogen electrodes in both cells may be replaced by a single glass electrode transferred between the cells, subject to limitations of the glass electrode (see **B.2** in BS 1647-1:1984).

NOTE 2 The pH difference, pH(*X*) – pH(*S*), is calculated.

The pH values of the operational reference standard solutions between 60 °C and 95 °C shall be in accordance with the following equation:

$$\mathrm{pH}(X) = A + B(T - 298.15) + C(T - 298.15)^2 + D(T - 298.15)^3 \qquad (2)$$

where the constants *A, B, C* and *D* are given in Table 2.

NOTE 3 Values of pH at various temperatures are given in Table 1.

NOTE 4 The two cells both involve liquid junctions because the reference electrode consists of a calomel or silver/silver chloride electrode in contact with a solution of potassium chloride of fixed concentration. Provided that the filling solution of a single junction reference electrode has a high concentration of potassium chloride (not less than 3.5 mol/L KCl), or the bridge solution of a double junction reference electrode has a high concentration of potassium nitrate (not less than 1 mol/L), the actual concentration has a negligible effect on the determined value of pH(*X*). For work of the highest accuracy, it is essential that the junction be formed in a reproducible fashion within a capillary tube as described in **F.2.4**.

NOTE 5 If the test solution is chemically incompatible with the reference electrode filling solution, it is necessary to use a bridge solution in the form of a double junction reference electrode.

NOTE 6 Factors pertinent to the choice of reference electrode are described in BS 2586.

NOTE 7 The preparation of the operational standard reference solutions listed in Table 2 is described in Appendix E.

### Table 1 — pH values of operational reference standard solutions

| Operational reference standard solution | Temperature (°C) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 10 | 20 | 25 | 30 | 37 | 40 | 50 | 60 | 70 | 80 | 90 | 95 |
| 0.1 mol/kg potassium tetraoxalate | — | — | 1.475 | 1.479 | 1.483 | 1.490 | 1.493 | 1.503 | 1.513 | 1.52 | 1.53 | 1.53 | 1.53 |
| 0.05 mol/kg potassium tetraoxalate | — | 1.638 | 1.644 | 1.646 | 1.648 | 1.649 | 1.650 | 1.653 | 1.660 | 1.671[a] | 1.689[a] | 1.72 | 1.73 |
| 0.05 mol/kg sodium hydrogen diglycolate | — | 3.470 | 3.484 | 3.492 | 3.502 | 3.519 | 3.527 | 3.558 | 3.595 | — | — | — | — |
| Saturated potassium hydrogen tartrate | — | — | — | 3.556 | 3.549 | 3.544 | 3.542 | 3.544 | 3.553 | 3.570[a] | 3.596[a] | 3.627[a] | 3.649[a] |
| 0.05 mol/kg potassium hydrogen phthalate | 4.000 | 3.997 | 4.000 | 4.005 | 4.011 | 4.022 | 4.027 | 4.050 | 4.080 | 4.116 | 4.159 | 4.208 | 4.235 |
| 0.1 mol/L acetic acid + 0.1 mol/L sodium acetate | 4.664 | 4.652 | 4.645 | 4.644 | 4.643 | 4.647 | 4.650 | 4.663 | 4.684 | 4.713 | 4.75 | 4.80 | 4.83 |
| 0.01 mol/L acetic acid + 0.01 mol/L sodium acetate | 4.729 | 4.717 | 4.712 | 4.713 | 4.715 | 4.722 | 4.726 | 4.743 | 4.768 | 4.800 | 4.839[a] | 4.88 | 4.91 |
| 0.02 mol/kg piperazine orthophosphate | — | 6.419 | 6.310 | 6.259 | 6.209 | 6.143 | 6.116 | 6.030 | 5.952 | — | — | — | — |
| 0.025 mol/kg *di*sodium hydrogen orthophosphate + 0.025 mol/kg potassium dihydrogen orthophosphate | 6.961 | 6.912 | 6.873 | 6.857 | 6.843 | 6.828 | 6.823 | 6.814 | 6.817 | 6.830 | 6.85 | 6.90 | 6.92 |
| 0.03043 mol/kg *di*sodium hydrogen orthophosphate + 0.08695 mol/kg potassium dihydrogen orthophosphate[c] | 7.506 | 7.460 | 7.423 | 7.406 | 7.390 | 7.369 | — | — | — | — | — | — | — |
| 0.04 mol/kg *di*sodium hydrogen orthophosphate + 0.01 mol/kg potassium dihydrogen orthophosphate[d] | — | 7.488 | 7.445 | 7.428 | 7.414 | 7.404 | — | — | — | — | — | — | — |
| 0.05 mol/kg Tris hydrochloride + 0.01667 mol/kg Tris[b] | 8.399 | 8.083 | 7.788 | 7.648 | 7.513 | 7.332 | 7.257 | 7.018 | 6.794 | — | — | — | — |
| 0.05 mol/kg *di*sodium tetraborate (borax) | 9.475 | 9.347 | 9.233 | 9.182 | 9.134 | 9.074 | 9.051 | 8.983 | 8.932 | 8.898 | 8.88 | 8.84 | 8.89 |
| 0.01 mol/kg *di*sodium tetraborate | 9.451 | 9.329 | 9.225 | 9.179 | 9.138 | 9.088 | 9.066 | 9.009 | 8.965 | 8.932 | 8.91 | 8.90 | 8.89 |
| 0.025 mol/kg sodium hydrogen carbonate + 0.025 mol/kg sodium carbonate | 10.27 | 10.154 | 10.045 | 9.995 | 9.948 | 9.889 | 9.866 | 9.800 | 9.753 | 9.728[a] | 9.725[a] | 9.75 | 9.77 |
| Saturated calcium hydroxide | 13.360 | 12.965 | 12.602 | 12.431 | 12.267 | 12.049 | 11.959 | 11.678 | 11.423 | 11.192[a] | 10.984[a] | 10.80 | 10.71 |

[a] The last decimal figure is not significant but is included to facilitate smooth interpolation.

[b] NOTE Tris = Tris(hydroxymethyl) aminomethane.

[c] = ratio of 3.5 : 1.

[d] = ratio of 4 : 1.

## Table 2 — Values of the constants for calculating pH values of operational reference standard solutions

$\mathrm{pH}(X) = A + B(T - 298.15) + C(T - 298.15)^2 + D(T - 298.15)^3$

| Operational reference standard solution | Constant | | | |
|---|---|---|---|---|
| | $A$ | $10^4B$ | $10^6C$ | $10^7D$ |
| 0.1 mol/kg potassium tetraoxalate | 1.4787 | 8.708 | 7.756 | – 1.266 |
| 0.05 mol/kg potassium tetraoxalate | 1.6462 | 3.184 | – 8.733 | 3.136 |
| 0.05 mol/kg sodium hydrogen diglycolate | 3.4924 | 18.871 | 27.983 | 0.4838 |
| Saturated potassium hydrogen tartrate | 3.5555 | – 14.752 | 40.281 | – 0.0301 |
| 0.05 mol/kg potassium hydrogen phthalate | 4.0048 | 10.007 | 32.745 | 0 |
| 0.1 mol/L acetic acid + 0.1 mol/L sodium acetate | 4.6435 | – 0.7529 | 31.991 | 0.9828 |
| 0.01 mol/L acetic acid + 0.01 mol/L sodium acetate | 4.7130 | 2.910 | 37.174 | – 0.1575 |
| 0.02 mol/kg piperazine orthophosphate | 6.2589 | – 100.99 | 39.601 | – 0.4978 |
| 0.025 mol/kg *di*sodium hydrogen orthophosphate + 0.025 mol/kg potassium dihydrogen orthophosphate | 6.8567 | – 29.777 | 49.338 | 0.8568 |
| 0.03043 mol/kg *di*sodium hydrogen orthophosphate + 0.08695 mol/kg potassium dihydrogen orthophosphate | 7.4060 | – 32.435 | 17.178 | – 5.439 |
| 0.04 mol/kg *di*sodium hydrogen orthophosphate + 0.01 mol/kg potassium dihydrogen orthophosphate | 7.4277 | – 31.767 | 80.661 | 14.713 |
| 0.05 mol/kg Tris hydrochloride + 0.01667 mol/kg Tris[a] | 7.6484 | – 274.70 | 96.190 | – 2.493 |
| 0.05 mol/kg *di*sodium tetraborate (borax) | 9.1818 | – 98.9233 | 75.587 | 0.9281 |
| 0.01 mol/kg *di*sodium tetraborate | 9.1793 | – 87.021 | 81.179 | – 2.189 |
| 0.025 mol/kg sodium hydrogen carbonate + 0.025 mol/kg sodium carbonate | 9.9947 | – 96.703 | 67.187 | 3.571 |
| Saturated calcium hydroxide | 12.4305 | – 334.88 | 141.78 | – 2.037 |

[a] Tris = Tris(hydroxymethyl) aminomethane.

## Table 3 — pH values of useful buffer solutions

| **Buffer solutions** | **Temperature (°C)** | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | **0** | **5** | **10** | **15** | **20** | **25** | **30** | **35** | **37** | **40** | **45** | **50** |
| 0.01 mol/L HCl + 0.09 mol/L KCl | — | — | — | — | — | 2.07 | — | — | 3.08 | — | — | — |
| 0.05 mol/L potassium dihydrogen citrate | 3.86 | 3.84 | 3.82 | 3.80 | 3.79 | 3.78 | 3.78 | 3.76 | 3.76 | 3.75 | 3.75 | 3.75 |
| 0.01 mol/L phenylacetic acid + 0.01 mol/L sodium phenylacetate | 4.27 | — | 4.27 | — | 4.28 | 4.29 | 4.30 | — | 4.32 | 4.33 | — | 4.38 |
| 0.02 mol/L Bis-Tris + 0.02 mol/L Bis-Tris HCl (1) | 7.00 | 6.91 | 6.81 | 6.72 | 6.64 | 6.55 | 6.47 | 6.39 | 6.36 | 6.31 | 6.24 | 6.17 |
| 0.06 mol/L tricine + 0.02 mol/L sodium tricinate (2) | — | 8.02 | 7.92 | 7.81 | 7.71 | 7.62 | 7.53 | 7.43 | 7.41 | 7.36 | 7.28 | 7.20 |
| 0.05 mol/L tricine + 0.05 mol/L sodium tricinate (2) | — | 8.49 | 8.38 | 8.27 | 8.18 | 8.08 | 7.99 | 7.90 | 7.86 | 7.82 | 7.74 | 7.66 |

NOTE 1 Bis (hydroxymethyl) aminotris (hydroxyethyl) methane; dry at 80 °C.
NOTE 2 Tris (hydroxymethyl) methylglycine.

NOTE 8 The reference value standard solution specified in clause **3** is also suitable for use as an operational reference standard solution and is therefore included in Table 1. No distinction need be made between the reference value standard solutions and operational reference standard solutions when choosing the best one to employ for a particular pH measurement.

## 5 Operational reference standard materials

The pH value of a solution of an operational reference standard material prepared by the appropriate method described in Appendix E and tested as described in Appendix F shall differ by no more than ± 0.003 from the values given in Table 1 at the test temperature which shall be either 20 °C or 25 °C. If certified operational reference standard material is not available, or not available in sufficient quantity, the procedures described in Appendix D and Appendix F shall be employed to ensure that the selected recognized analytical grade material satisfies the above requirement if the accuracy class of the desired measurements warrants this.

Operational reference standard materials supplied in prepackaged form for the preparation of a stated volume of operational reference standard solution shall state on the package the tolerance on the pH value the user may expect when the solution is prepared at 20 °C in grade B glassware as specified in BS 1792.

## 6 Accuracy class

The accuracy necessary for pH measurements shall be classified as given in Table 4.

**Table 4 — 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 |

The accuracy class shall be 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).

## Appendix A Test method for the verification of samples of potassium hydrogen phthalate (KHPh) as the reference value standard material

NOTE to Appendix A Considerable practical experience in the construction and use of electrochemical cells is required for the successful performance of these determinations.

### A.1 Principle

The method is based ([4], [5] and [6]) on measurements of the e.m.f. of the cell

$\mathrm{Pt(Pd)} \mid \mathrm{H_2} \mid \mathrm{KHPh}\ (0.05\ \mathrm{mol/kg},\ \mathrm{KCl}(m) \mid \mathrm{AgCl} \mid \mathrm{Ag}$

at 25 ± 0.01 °C, where $m$ = 0.01 mol/kg, 0.015 mol/kg, 0.02 mol/kg, 0.03 mol/kg, 0.04 mol/kg and 0.05 mol/kg.

The e.m.f. is given by the equation

$$E = E^{\circ} - k \lg \frac{m_{\mathrm{H}}\gamma_{\mathrm{H}} m_{\mathrm{Cl}}\gamma_{\mathrm{Cl}}}{m_{\mathrm{o}}^{2}} \qquad (3)$$

where

$k$ is the slope factor;

$m$ signifies molalities of the subscripted species;

$\gamma$ signifies the activity coefficients of the subscripted species;

$E^{\circ}$ is the standard e.m.f.;

$m_{\mathrm{o}}$ = 1 mol/kg.

Equation (3) can be rearranged to

$$-\lg m_{\mathrm{H}}\gamma_{\mathrm{H}}\gamma_{\mathrm{Cl}}/m_{\mathrm{o}} = (E - E^{\circ})/k + \lg m_{\mathrm{Cl}}/m_{\mathrm{o}} \qquad (4)$$

The standard e.m.f. is found from measurements of the cell

$\mathrm{Pt, H2} \mid \mathrm{HCl}\ (0.01\ \mathrm{mol/kg}) \mid \mathrm{AgCl} \mid \mathrm{Ag}$

at 25 ± 0.01 °C and calculated from equation (3) with $\gamma_{\mathrm{H}}\gamma_{\mathrm{Cl}} = 0.817$ and should lie within the range 222.4 ± 0.2 mV. The quantity $-\lg m_{\mathrm{H}}\gamma_{\mathrm{H}}\gamma_{\mathrm{Cl}}/m_{\mathrm{o}}$ is then calculated from (4) for all six chloride concentrations.

It is then plotted against $m = m_{\mathrm{Cl}}$ and extrapolated to $m_{\mathrm{Cl}} = 0$. The pH value can be calculated from

$$\mathrm{pH} = \lg m_{\mathrm{H}}\gamma_{\mathrm{H}}\gamma_{\mathrm{Cl}}/m_{\circ} - 0.0877 \qquad (5)$$

where the last term ($\lg\gamma_{\mathrm{Cl}} = -0.0877$) has been calculated from the Bates-Guggenheim convention (equation (1) in Part 1) for the single ion activity at 0.0534 mol/kg which is the actual ionic strength of 0.05 mol/kg potassium hydrogen phthalate solution.

NOTE For measurements at temperatures other than 25 °C, the appropriate values of $E^{\circ}$ (or $\gamma_{\mathrm{H}}\gamma_{\mathrm{Cl}}$ and $\gamma_{\mathrm{Cl}}$ can be derived from reference [2].

### A.2 Apparatus and reagents

**A.2.1** *Electrodes.* Prepare silver/silver chloride electrodes by the thermal electrolytic method described in BS 2586. Reduction of phthalate at a platinized platinum hydrogen gas electrode leads to drifting potentials and, to avoid this, it is necessary to use palladized platinum electrodes. Cathodize platinum foil electrodes (1 $\mathrm{cm^2}$), welded to platinum wire and sealed into glass support tubes mounted on extended 19/26 cones, for 10 min at a current density of 10 $\mathrm{mA\ cm^{-2}}$ in a solution of 2 g of palladium (II) chloride in 100 mL of 1 mol/L HCl, using a platinum wire anode.

**A.2.2** *Potassium chloride.* Dry potassium chloride (KCl) (recognized analytical reagent grade) at 110 °C.

**A.2.3** *Cell vessels.* A suitable cell vessel made from borosilicate glass is shown on the left of Figure 1 and consists of two electrode compartments, each fitted with 19/26 ground glass sockets (see BS 572); the one on the right for the hydrogen gas electrode is connected by a capillary tube (2 mm i.d.) to the one on the left for the silver/silver chloride electrode. Before reaching the hydrogen electrode compartment, hydrogen gas[1] passes through a three stage bubbler, containing the cell solution, with a sintered glass disc (porosity grade P10 or P16 as defined in BS 1752) in the second stage. Each electrode compartment has an additional 7/16 ground glass socket. The socket on the silver/silver chloride electrode compartment is used to fill the cell (with the electrodes already in position) and is closed with a 7/16 stopper after the filling process. The one on the hydrogen gas electrode compartment serves as an exit for the hydrogen gas and may be taken to a solution seal device to prevent ingress of air. Filling is carried out from suitable solution-dispensing flasks under nitrogen at positive gas pressure. Two fillings of the cell are used to wash the electrodes and are displaced into the bubbler compartment, which has double the volume of the electrode compartments. Total cell vessel volume is 150 mL.

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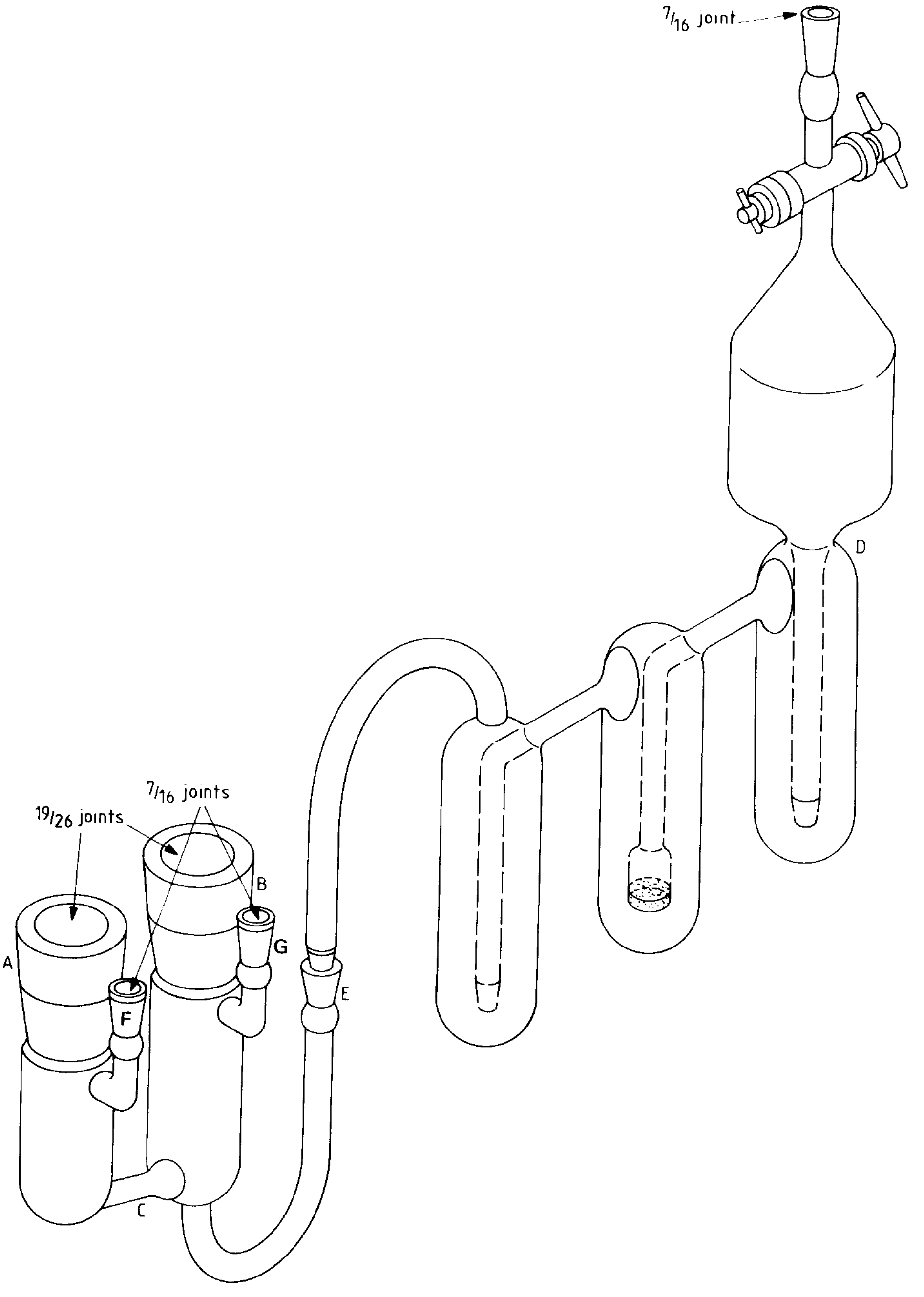
[1] Hydrogen to BS 3906, type 2 is suitable.



Explanatory notes to figure 1:

The cell vessel has two electrode compartments (A,B) of capacity 25 cm$^3$ each, with 19/26 sockets, one for the reference electrode (A) and the other for the hydrogen gas electrode (B), connected by a capillary tube (C). Before reaching the cell, the hydrogen gas passes through a three-stage bubbler (D) with a sintered glass frit in the middle position. The bubbler is attached by a 17/16 joint (E) to the hydrogen electrode compartment in the same line, so that the whole set-up can be fixed to a metal holder with the aid of two clamps and placed in a water thermostat.

Compartments (A) and (B) have outlets with 7/16 sockets. In the reference electrode compartment (F) this is closed with a stopper after the filling process. In the other compartment (G) there is a hydrogen gas exit device to prevent contact with atmospheric carbon dioxide. Filling is from a dispensing flask by means of nitrogen gas pressure through a three-way tap at (F), where solution or nitrogen gas can enter the cell. Alternatively, the first two fillings wash the electrodes and are pushed to the bubbler (D).

This design is based on one from NBS* but utilizes vessels with twice the volume. It permits filling under carbon dioxide-free conditions and, with all taps closed, there is no contamination of the electrolyte by atmospheric carbon dioxide.

*R. Gary, R.G. Bates and R.A. Robinson *J. Phys. Chem.*, 1964, **68**, 1186.

**Figure 1 — Cell vessel for certification of samples of potassium hydrogen phthalate as the reference value standard material**

### A.3 Procedure

Prepare the six solutions referred to in **A.1** by adding the requisite amounts of solid potassium chloride to 200 mL portions of 0.05 mol/kg potassium hydrogen phthalate solution prepared according to the method detailed in **B.2**.

Assemble and fill six cell vessels as described in **A.2.3** and mount them in a water bath, thermostatically controlled at 25.00 ± 0.01 °C. Distribute hydrogen gas of the highest available purity from the cylinder to each cell vessel at two bubbles per second using a manifold of needle valves. Allow 1 h for hydrogen gas saturation and electrode equilibration before taking potential difference readings at 10 min intervals with a potentiometer or a digital voltmeter with 0.05 mV discrimination or better. Record the steady values reached, and check that they remain constant for at least 1 h. Determine the prevailing atmospheric pressure and correct the measured values to 1 atmosphere[2)] partial pressure of hydrogen gas by subtracting the vapour pressure of water at 25 °C (see [3] for a table of corrections). Use the corrected values as values of $E$ in equation (4). The measurement of standard e.m.f. ($E°$) may, if desired, be made simultaneously in a separate cell vessel containing 0.01 mol/kg hydrochloric acid. Calculate the six values of $-\lg m_H\ \gamma_H\gamma_{Cl}/m_o$. Perform a linear regression on the values against $m$ (see **A.1**).

### A.4 Expression of results

Report the intercept value and its estimated standard deviation.

The extrapolated value of $-\lg m_H\,\gamma_H\gamma_{Cl}$ should lie within the range 4.093 ± 0.003 corresponding to pH = 4.005 ± 0.003.

## Appendix B Preparation of the reference value standard solution

### B.1 Reference value standard material

Potassium hydrogen phthalate certified as reference value standard material shall have been tested in accordance with Appendix A. Its pH value at 25 °C shall differ by no more than ± 0.003 from the value of 4.005. Values at other temperatures shall be in accordance with the values listed in Table 2 with error limits of ± 0.005 between 5 °C and 20 °C, ± 0.01 between 30 °C and 65 °C and ± 0.02 above 65 °C.

NOTE Samples of potassium hydrogen phthalate sold or used in this country for the preparation of the reference value standard solution may, by arrangement, be tested at the National Physical Laboratory in accordance with this British Standard, and suitable samples will be certified. For particulars, including the fees charged, application should be made to the Director, National Physical Laboratory, Teddington, Middlesex, TW11 0LW.

If certified reference value standard material is not available, or not available in sufficient quantity, the procedures described in Appendix D, Appendix E and Appendix F shall be employed to ensure that the selected recognized analytical grade reagent is of sufficient purity to satisfy the above requirements.

### B.2 Preparation of reference value standard solution

NOTE 1 Buoyancy corrections to weighings of potassium hydrogen phthalate are negligible and need not be made.

Dry the material at 110 °C for 2 h before use. Use water of the quality specified in Appendix G. The required solution contains 0.05 moles (10.211 g) potassium hydrogen phthalate per kilogram of water and shall be prepared on a mass basis for the most precise work.

NOTE 2 This solution can be prepared on a volume basis by dissolving 10.125 g of potassium hydrogen phthalate and making up to 1 L with water at 25 °C (this solution is 0.04958 mol/L with a density at 25 °C of 1.00168 g/cm$^3$) or by dissolving 10.138 g and making up to 1 L with water at 20 °C (this solution is 0.04964 mol/L with a density at 20 °C of 1.00300 g/cm$^3$). The pH of a solution of 0.05 mol/L at 25 °C is less than that of the 0.05 mol/kg solution by about 0.001.

## Appendix C Method for estimating the phthalic acid content of potassium hydrogen phthalate

### C.1 Introduction

The purity of a sample of potassium hydrogen phthalate to be used as a reference value standard material may be inferred from a cell measurement (Appendix A). However, an independent purity check is possible [7, 8]. The principal impurity in commercial samples of KHPh is the parent acid and the following method is suitable for its extraction and estimation from dried samples. Phthalic acid is soluble in ether (solubility $2 \times 10^{-2}$ mol/L) whereas KHPh is insoluble.

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[2)] 1 atmosphere = 101.3 kPa.

### C.2 Samples

The amount of sample to be taken depends on the suspected level of impurity. Free acid contents of 0.05 % *m*/*m* corresponding to a pH error of – 0.002, are not uncommon. The method is capable of detecting 0.003 % *m*/*m* phthalic acid with a reproducibility of ± 10 % between samples. At such low levels, all glass-ware and cells should be washed with ether to remove adsorbed phthalic acid.

Do not grind the sample. The impurity acid appears to be on the crystal surfaces and is extracted rapidly. Grinding and vigorous shaking causes an increase in surface area and readsorption of material.

### C.3 Procedure

Dry a sample of potassium hydrogen phthalate for 2 h at 120 °C. Transfer a weighed amount (1 g to 10 g) of sample to a small glass stoppered flask or tube and add 10 mL of sodium-dried diethyl ether. Shake by hand for 15 min or place in an ultrasonic vibrator for 5 min. Decant the supernatant solvent into a centrifuge tube, stopper it and centrifuge at 2 500 r/min for 5 min to remove any fine particles. Ascertain the position of the peak maximum by scanning the spectrum in the range 220 nm to 350 nm. Measure the absorbance of the ether extract at the peak maximum (275 nm) in a 10 mm stoppered silica cell against ether in the reference cell.

Alternatively, and particularly when the ambient temperature and ether volatility is high, remove the ether by evaporation and dissolve the residue in 10 mL of 1,1,1,3,3,3-hexafluoropropan-2-ol (spectroscopic grade reagent) by placing in an ultrasonic vibrator for 5 min. Measure the absorbance at the peak maximum (285 nm).

### C.4 Expression of results

Obtain the mass percentage of phthalic acid impurity from

$$\% \ m/m \text{ phthalic acid} = 0.15\, A_1/m$$

where

$A_1$ is the absorbance in the ether at the peak maximum for 10 mm cells;

$m$ is the mass of the sample (in g).

or from

$$\% \ m/m \text{ phthalic acid} = 0.069\, A_2/m$$

where

$A_2$ is the absorbance in the hexafluoropropanol at the peak maximum for 10 mm cells.

as appropriate.

NOTE Samples showing no extraction of phthalic acid may contain dipotassium phthalate and should be recrystallized from water (see Appendix E) if its presence is suspected.

### C.5 Notes of procedure

The peak maximum of 275 nm in ether is chosen in preference to that at about 217 nm because, even though the molar absorbance is lower, the spectrum is more reproducible. In hexafluoropropanol, the maximum is more clearly defined at 283 nm and its molar absorbance is greater by a factor of 2 than that of the 275 nm band in ether, where there is an additional band at 282 nm. Change of ambient temperature has little effect on absorbance except when volatile loss of ether occurs.

## Appendix D Simple method for establishing the identity of the pH of samples of potassium hydrogen phthalate

NOTE to Appendix D This method can also be used to compare samples of operational standard reference materials.

### D.1 General

The use of the cell without liquid junction (Appendix A) to establish the identity of the pH of samples of KHPh is a specialized and time-consuming procedure. If a small quantity of certified reference value standard material is available, a simpler method based on the cell

$$\text{Pt} \mid \text{H}_2 \mid \text{Sample A} \mid \mid \text{Sample B} \mid \text{H}_2 \mid \text{Pt}$$

devised by Baucke [9] should be used. The cell vessel should be a simple H-shape with the two samples forming a liquid junction at a sintered glass disc of porosity grade 4 as specified in BS 1752. As the samples will differ only slightly, no appreciable liquid junction potential is involved and the measured potential difference indicates a difference in hydrogen ion concentration between samples. The technique is simpler than that using a cell with a concentrated potassium chloride bridge solution between the samples.

It is, however, essential that the hydrogen gas leaving each hydrogen electrode compartment is taken to a common exit tube to ensure that the pressures are identical.

If no certified reference value standard material is available, the procedure described in **D.2** is suitable.

### D.2 Procedure

Carry out the procedure described in Appendix C to estimate the phthalic acid content of the sample. If this is greater than 0.025 % *m*/*m*, and the corresponding error in pH of 0.001 is not acceptable, recrystallize the sample from aqueous solution held at 40 °C. Dry at 120 °C for 2 h and compare the original and recrystallized material in the H-shaped cell referred to in **F.1** as 0.05 mol/kg solutions. Alternatively or additionally, repeat the phthalic acid impurity determination. Continue until the phthalic acid content falls to less than 0.025 % *m*/*m*, or until the e.m.f. of the H-shaped cell falls below 0.1 mV after successive recrystallization of the sample.

## Appendix E Preparation of operational reference standard solutions

NOTE 1 If there is doubt as to whether the quality of the analytical reagent grade materials is adequate, they should be recrystallized until no further change in pH of their solutions is detectable on successive recrystallizations.

NOTE 2 Buoyancy corrections to weighings of standard reference materials are negligible and need not be made.

NOTE 3 Information on densities and molarities at 20 °C, and tolerances on masses for the preparation of some operational standard reference solutions is given in Table 5.

NOTE 4 Use water of the quality specified in Appendix G.

### E.1 Potassium tetraoxalate ($KH_3C_4O_8.2H_2O$): 0.05 mol/kg and 0.1 mol/kg

Dry the material at 50 °C to constant mass. Prepare the 0.05 mol/kg solution by dissolving 12.7096 g in 998.2 g of water, or alternatively, prepare the solution by dissolving 12.62 g in water and make up to 1 L at 20 °C. Prepare the 0.1 mol/kg solution by dissolving 25.4191 g in 996.4 g water or, alternatively, by dissolving 25.10 g in water and making up to 1 L with water at 20 °C.

### E.2 Sodium hydrogen diglycolate (2,2 sodium oxydiacetate) ($NaOOC.CH_2.O.CH_2COOH$): 0.05 mol/kg

Dry material to constant mass at 110 °C, Prepare the solution by dissolving 7.923 g in 1 kg of water. If the salt is not available, half neutralize the parent acid with sodium hydroxide. Crystallize and filter on a sintered glass funnel.

NOTE The free acid and the disodium salt are more soluble than the monosodium salt and remain in solution.

### E.3 Potassium hydrogen tartrate ($KHC_4H_4O_6$): saturated at 25 °C

Prepare a saturated solution (about 6.4 g/L) at 25 °C.

### E.4 Sodium acetate + acetic acid: 0.01 mol/L and 0.1 mol/L (equal concentrations of each component)

NOTE These two solutions cannot be prepared from the sodium salt because the latter is hygroscopic. They are prepared by half neutralization of acetic acid with sodium hydroxide. Analytical standard volumetric acetic acid is commercially available.

Prepare the two solutions on a volume basis at 20 °C to give molalities of 0.0100 mol/kg and 0.1015 mol/kg respectively.

### E.5 Piperazine orthophosphate monohydrate $2.H_2O$): 0.02 mol/kg

Recrystallize from water.

NOTE 1 The material should be air dried to constant mass as decomposition of the monohydrate occurs above 70 °C.

Prepare the solution by dissolving 4.043 g in 1 kg of water.

NOTE 2 Anomalously large liquid junction potentials are observed with this solution.

### E.6 *Di*sodium hydrogen orthophosphate ($Na_2HPO_4$) + potassium dihydrogen orthophosphate ($KH_2PO_4$): 0.025 mol/kg of each component

Dry the salts to constant mass at 120 °C and 110 °C respectively. Prepare the solution by dissolving 3.5490 g of $Na_2HPO_4$ and 3.4021 g of $KH_2PO_4$ in 1 kg of water.

NOTE The solution can also be prepared by dissolving 3.54 g and 3.39 g respectively in water and making up to 1 L with water at 20 °C.

### E.7 *Di*sodium hydrogen orthophosphate ($Na_2HPO_4$) + potassium dihydrogen orthophosphate ($KH_2PO_4$): (3.5 : 1)

Dry the salts to constant mass at 120 °C and 110 °C respectively. Prepare the solution by dissolving 4.320 g of $Na_2HPO_4$ and 1.183 g of $K_2HPO_4$ in 1 kg of water, or alternatively 4.302 g and 1.179 g respectively and make up to 1 L with water at 25 °C.

### E.8 *Di*sodium hydrogen orthophosphate ($Na_2HPO_4$) + potassium dihydrogen orthophosphate ($KH_2PO_4$): (4 : 1)

Dry the salts to constant mass at 120 °C and 110 °C respectively. Prepare the solution by dissolving 5.679 g of $Na_2HPO_4$ and 1.361 g of $KH_2PO_4$ in 1 kg of water.

### Table 5 — Useful data on some operational reference standard solutions

| Operational standard reference material | Molecular formula | Molality | Relative molar mass | Density at 20 °C | Molarity at 20 °C | Mass of 1 L at 20 °C | Mass tolerance for ± 0.001 pH[a] | Mass tolerance expressed as a percentage |
|---|---|---|---|---|---|---|---|---|
| | | mol/kg | | g/cm$^3$ | mol/L | g | g | |
| Potassium tetraoxalate | $KH_3C_4O_8.2H_2O$ | 0.1 | 254.1913 | 1.0091 | 0.09875 | 25.1017 | 0.07 | 0.27 |
| Potassium tetraoxalate | $KH_3C_4O_8.2H_2O$ | 0.05 | 254.1913 | 1.0038 | 0.04965 | 12.6202 | 0.034 | 0.26 |
| *D*isodium hydrogen orthophosphate | $Na_2PO_4$ | 0.025 | 141.9588 | 1.0038 | 0.02492 | 3.5379 | 0.02 | 0.56 |
| Potassium dihydrogen orthophosphate | $KH_2PO_4$ | 0.025 | 136.0852 | | | 3.3912 | 0.02 | 0.58 |
| Disodium tetraborate | $Na_2B_4O_7.10H_2O$ | 0.05 | 381.367 | 1.0075 | 0.04985 | 19.0117 | 0.9 | 4.73 |
| Disodium tetraborate | $Na_2B_4O_7.10H_2O$ | 0.01 | 381.367 | 1.0001 | 0.009981 | 3.8064 | 0.19 | 0.49 |
| Sodium carbonate | $Na_2CO_3$ | 0.025 | 105.9887 | 1.0021 | 0.02494 | 2.6428 | 0.017 | 0.64 |
| Sodium hydrogen carbonate | $NaHCO_3$ | 0.025 | 84.0069 | | | 2.0947 | 0.013 | 0.62 |

[a] Calculated from known dilution value of solution.

### E.9 Tris (hydroxymethyl) aminomethane[3] ($NH_2C(CH_2OH)_3$) + HCl: 0.01667 mol/kg Tris + 0.05 mol/kg Tris hydrochloride

Dry the materials to constant mass at 80 °C and 40 °C respectively. Prepare the solution by dissolving 1.999 g of Tris and 7.800 g of Tris hydrochloride in water and make up to 1 L at 25 °C. Alternatively, prepare the solution by dissolving 8.008 g of Tris and 50 mL of 1 mol/L HCl in water and make up to 1 kg with water.

NOTE Anomalously large liquid junction potentials are observed with this solution, particularly with fibre types of junction.

### E.10 *Di*sodium tetraborate ($Na_2B_4O_7.10H_2O$): 0.05 mol/kg and 0.01 mol/kg

NOTE Recrystallization of recognized analytical reagent grade material is essential for precise work as the pH value of solutions of the unrecrystallized material may be lower by 0.005.

Recrystallize a solution, containing about 300 g/L, below 55 °C. Wash the crystals successively with ice-cold water, then twice with ethanol and twice with ether, and store over aqueous saturated sodium bromide solution [10]. Prepare the 0.05 mol/kg solution either by dissolving 19.0684 g in 991.0 g of water or by dissolving 19.01 g in water and making up to 1 L with water at 20 °C. Prepare the 0.01 mol/kg solution either by dissolving 3.8137 g in 998.2 g of water or by dissolving 3.80 g in water and making up to 1 L with water at 20 °C.

### E.11 Sodium hydrogen carbonate + sodium carbonate: 0.025 mol/kg of each component

Dry sodium carbonate at 250 °C and cool over calcium chloride. Dry sodium hydrogen carbonate at 50 °C. Prepare the solution either by dissolving 2.1002 g of $NaHCO_3$ and 2.6497 g of $Na_2CO_3$ in 1 kg of carbon dioxide-free water or by taking 2.095 g and 2.643 g respectively and making up to 1 L with carbon dioxide-free water at 20 °C.

### E.12 Calcium hydroxide: saturated at 20 °C

Heat well-washed calcium carbonate at 1 000 °C for 45 min. After cooling, add slowly to water without stirring. Heat the suspension to boiling. Cool and filter on a sintered glass funnel. Dry the solid, crush to a fine granular state and store in a sealed container. Prepare a saturated solution (about 1.6 g/L) at 20 °C. Use carbon dioxide-free water unless excess solid is allowed to be present.

## Appendix F Determination of pH values of operational reference standard solutions

NOTE Considerable practical experience in the construction of electrochemical cells is required for the successful performance of these determinations and they should not be carried out by inexperienced persons.

### F.1 Principle

The determination of pH values of operational reference standard solutions is based on measurements of the operational cell where the operational reference standard solution is *X* and the reference value standard solution, potassium hydrogen phthalate 0.05 mol/kg, is solution *S*. The preparation of the reference value standard solution is described in Appendix B and the preparation of 15 operational reference standard solutions is described in Appendix E. The method uses free diffusion liquid junctions formed in cylindrical tubes of 1 mm internal diameter (see references [1] and [2]).

### F.2 Apparatus

**F.2.1** *Reference electrode.* Measurements are made of the two cells described in clause **4** in combination, either with or without the use of a reference electrode

$$\mathrm{Pt}(Pd)\,|\,\mathrm{H_2}\,|\,\mathrm{S}\,|\,|\,\mathrm{KCl}\ (m > 3.5\ \mathrm{mol/L})\,|\,|\,X\,|\,\mathrm{H_2}\,|\,\mathrm{Pt}$$

but it is often convenient to incorporate a reference electrode complying with the requirements of BS 2586 in a third electrode compartment in order to determine, in the event of unsteady readings, which of the two hydrogen gas electrodes is responsible for a drifting potential. If the measurements are to be made separately, a reference electrode of high reproducibility is necessary; commercial calomel electrodes are not adequate for the purpose. The preparation of suitable calomel electrodes is described in reference [10] but alternatively, and preferably, prepare silver/silver chloride electrodes as described in BS 2586 and use in contact with potassium chloride solution (3.5 mol/L to 4.2 mol/L) saturated with silver chloride.

**F.2.2** *Hydrogen electrodes*

NOTE Platinized platinum, hydrogen gas electrodes may be used in the operational standard reference solutions unless, as in the case of the reference value standard, potassium hydrogen phthalate, there is a risk of catalytic hydrogenation of the buffer substance.

Prepare platinized platinum electrodes as described in **A.2.1** but with platinum (II) chloride substituted for palladium chloride.

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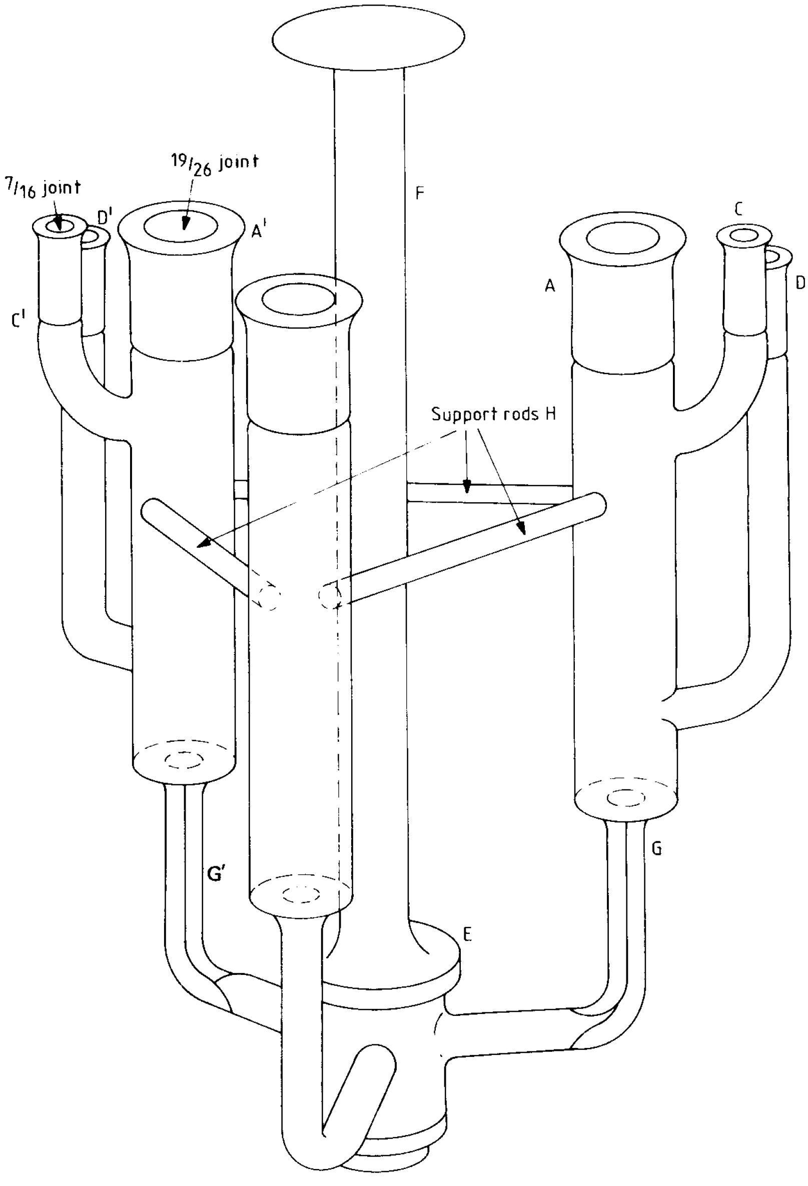
[3] Known as “Tris”; the systematic chemical name is 2-amino-2(hydroxylmethyl)propane 1,3-diol.



Explanatory notes to figure 2.

In the cell vessel, G and G′ are each made from a 50 mm length of 1 mm internal diameter capillary tubing which, at one end, joins flush with the base of a measuring electrode compartment (A, A′) of capacity 25 $cm^3$. At its other end, the capillary is joined to a length of 6 mm internal diameter tubing leading to a hollow three-way (60 °) stopcock (Y-form tap) (E), which is used in making the liquid junction. The Y-form tap is fitted with an elongated hollow handle (F). The three compartments are linked by support rods (H) to strengthen the cell vessel against breakage. (B) is the bridge solution compartment of capacity approximately 25 $cm^3$.

The Y-form tap is greased only at its top and bottom so that, when closed, it leaves a central conducting path of bridge solution.

To form the liquid junction, the cell is used in the following manner. The bridge solution in (B) is allowed, by tilting the vessel and turning the tap to the appropriate position, to flow half-way up the capillary of one of the measuring electrode compartments, and the tap is then closed. The cell vessel is rotated so that the other electrode compartment is in the upper position, the tap is turned to connect with it, and the same procedure is followed. The buffer solutions are introduced into the capillary tubes with long needle hypodermic syringes, which are also used to remove any air bubbles trapped between the bridge solution and the buffer solutions. More of each of the buffer solutions is then added to each measuring compartment to bring the liquid level to a predetermined mark. This mark, and a similar one in the KCl reservoir are positioned, to give equal hydrostatic heads in all three compartments to minimise any tendency for flow through the incompletely greased tap resulting in the liquid junction moving out of the capillary tubes.

After filling the measuring compartments, the palladised-platinum electrode or platinised electrodes are rinsed with the corresponding buffer solutions, and the hydrogen flow is started at a rate of two bubbles per second. The hydrogen is passed through a presaturator containing some of the same solution, entering the cell through (C) and (C′) and leaving through (D) and (D′). To equalise the pressure in the two measuring electrode compartments, these are led to a Y-tube and a common exit.

**Figure 2 — Cell vessel for determination of pH values of operational reference standard solutions**

**F.2.3** *Solutions*. Prepare the reference value standard solution in accordance with **B.2** and the chosen operational standard reference solution(s) in accordance with Appendix E.

Prepare potassium chloride solution (3.5 mol/L or saturated at 20 °C, i.e. 300 g/L) from recognized analytical reagent grade material, previously dried at 110 °C.

**F.2.4** *Cell vessels*. A suitable cell vessel made from borosilicate glass is shown in Figure 2 and consists of three electrode compartments (of volume 25 mL each) with 19/26 ground glass sockets as specified in BS 572, connected by a three-way (60°) hollow glass stopcock, lightly greased at top and bottom only, which permits any two compartments to be interconnected. This stopcock is used to form the liquid junction. Two of the compartments contain entry ports for hydrogen gas from suitable hydrogen gas presaturators, by way of 7/16 joints, as illustrated in Figure 2.

Additional 7/16 sockets on each gas electrode compartment provide for the exit of hydrogen gas, preferably to a common tube to ensure that the exit pressures are identical. At the base of each of these two gas electrode compartments is a 60 mm length of 1 mm i.d. capillary tubing connected to the tap. The potassium chloride solution is introduced into the reference compartment and is allowed, by turning the tap to the appropriate position, to flow half way up the capillary tube of one of the measuring electrode compartments. The tap is then turned to connect with the other electrode compartment and the procedure is repeated. The reference value standard reference solution is introduced into one electrode compartment with a long needle hypodermic syringe which is also used to remove any air bubbles trapped between the two solutions. More of the solution is added to bring the solution level to a predetermined mark. The procedure is repeated with a selected operational standard reference solution. The three solution levels are adjusted to give equal hydrostatic heads to ensure no solution flow, which would result in movement of the liquid junctions out of the capillary tubes, takes place on turning the tap.

### F.3 Procedure

Fill the cell vessel following the procedure described in **F.2.4** and insert the electrodes. Carefully transfer the cell to a thermostatic water bath maintained at 25.00 ± 0.01 °C, or other appropriate temperature (see Table 2). Avoid disturbing the liquid junction during the transfer by positioning the tap so that none of the compartments interconnect. Hydrogen gas of the highest available purity[4] is distributed from the cylinder to both hydrogen gas electrode compartments at a rate of two bubbles per second using a manifold of needle valves. Allow 1 h for hydrogen gas saturation and electrode equilibration. Turn the tap to interconnect the hydrogen gas electrode compartments.

NOTE This may not be necessary if the tap is greased top and bottom lightly and there is an annular film of solution in the tap.

Take potential difference readings at 10 min intervals with a potential or digital voltmeter with 0.05 mV discrimination or better.

### F.4 Expression of results

Record the steady values reached and substitute, with appropriate sign, for $E(S) - E(X)$ in equation (1) to calculate pH($X$) from the pH($S$) value in Table 2.

NOTE It is unnecessary to correct to 1 atmosphere partial pressure of hydrogen gas unless separate measurements are made against a reproducible reference electrode.

## Appendix G Water quality for the preparation of reference standard solutions

**G.1** For pH measurements the essential requirements are low concentrations of ionic impurities (determined by conductance) and low concentrations of dissolved carbon dioxide (determined by pH measurement). Both are more likely to be affected by storage conditions than by the quality of the water immediately after preparation either by distillation or ion-exchange.

High purity water, suitable for the accuracy classes for the pH measurements specified in clause **6**, is described in Table 6.

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[4] Hydrogen to BS 3906, type 2 is suitable.

**G.2** Carry out conducance measurements on flowing samples (see BS 2690-9).

NOTE Measurements on static samples will yield higher conductances.

**G.3** Prepare borate and carbonate solutions with carbon dioxide-free water.

**Table 6 — Water for the preparation of standard solutions**

| pH measurement accuracy class | Maximum conductivity at 20 °C | Storage |
|---|---|---|
| | mS/m | |
| 0.02 | 0.1 | Temporary only[a] |
| 0.1 | 0.2 | Borosilicate glass or polyethylene |
| 0.3<br>1.5 | 0.5 | Glass or plastics |

[a] Out of contact with atmosphere (pass a stream of nitrogen to deoxygenate and protect from carbon dioxide).

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# Publications referred to

BS 572, *Interchangeable conical ground glass joints.*

BS 1647, *pH measurement.*

BS 1647-1, *Specification for pH scale.*

BS 1752, *Specification for laboratory sintered or fritted filters including porosity grading.*

BS 1792, *Specification for one-mark volumetric flasks.*

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

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

BS 2690-9, *Appearance (colour and turbidity), odour, suspended and dissolved solids and electrical conductivity.*

BS 3906, *Electrolytic compressed hydrogen.*

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