IS 15557 (2005): Methods for preparation of buffer solutions and methods for determination of pH [CHD 1: Inorganic Chemicals]

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IS 15557 : 2005
(Superseding IS 3225 : 1965
and IS 5741 : 1970)

भारतीय मानक

बफर विलयन तैयार करने की पद्धतियाँ तथा *p*H ज्ञात करने की पद्धतियाँ

Indian Standard

METHODS FOR PREPARATION OF BUFFER SOLUTIONS AND METHODS FOR DETERMINATION OF *p*H

ICS 71.060.30

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Inorganic Chemicals and Photographic Materials Sectional Committee, CHD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

This standard has been formulated by amalgamating IS 3225 : 1965 'Methods for preparation of buffer solutions' and IS 5741 : 1970 'Methods for determination of *p*H'. IS 3225 was published in 1965 whereas IS 5741 was published in 1970. Since there are related standards, it was decided to amalgamate these two into one. Once, this standard will be published, the above two standards will be withdrawn.

In this standard, a series of buffer solution have been prescribed. It is expected that they will cover the common experimental conditions and be useful for control of *p*H measurement in industry. Different buffer solutions for the same *p*H range have been mentioned in order to offer a choice of a particular buffer for specific experiment.

The *p*H is given at different temperatures for different buffer solutions as data for a uniform temperature for these solutions were not available. It is intended to adopt a uniform temperature of 27°C for all of these solutions when the required data become available.

The determination of *p*H is the common and important control test in many chemical processing industries. Various methods of determination, ranging from simple indicator paper method to sophisticated *p*H meter methods, are followed depending upon the accuracy required. The determination of *p*H may be broadly classified into two classes, colorimetric and electrometric methods. The colorimetric method employs indicators which develop a range of colours at different *p*H. The electrometric method is based on the principle that the developed emf varies with *p*H.

Commercially a number of colorimetric comparators as well as the glass colour shades and indicator solutions are available. This standard prescribes a general method for determination of *p*H by colorimetry to permit the use of any commercially available comparator or device. Colorimetric determination of *p*H is recommended where approximate *p*H is required. For more accurate measurement of *p*H, electrometric procedures are to be followed. While it is true that the *p*H determination by electrometric procedures may be followed by using electrodes like hydrogen, quinhydrone, antimony and glass, this standard prescribes only glass-electrode direct-reading *p*H meter as it is used largely and has many advantages over others. With the availability of special glasses, glass electrode given very accurate results without being affected by oxidizing or reducing agents or organic substances. Moreover, glass electrode may be employed with unbuffered and small quantities of solution. It is also not poisoned easily.

There is no ISO specification for the methods for preparation of buffer solutions and methods for determination of *p*H.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

In reporting the result of a test or analysis made in accordance with this standard, if the final values, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR PREPARATION OF BUFFER SOLUTIONS AND METHODS FOR DETERMINATION OF *p*H

1 SCOPE

1.1 This standard prescribes the methods for preparation of buffer solutions used in analytical and control laboratories in industry and the methods for determination of *p*H by colorimetric and electrometric methods.

1.1.1 Primary and secondary standard buffer solutions for calibration of *p*H meters are also included in this standard.

1.1.2 This standard covers general methods of *p*H determination by colorimetric and glass-electrode direct-reading *p*H under electrometry.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
695 : 1986	Specification for acetic acid (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
2124 : 2000	Sodium bicarbonate — Specification (*second revision*)
2711 : 1979	Direct reading *p*H meters (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 *p*H — It is a measure of the hydrogen ion activity in aqueous media. It is represented by the equation:

$$pH = -\log_{10} \propto_{H} C_{H}$$

where

$\propto_{H}$ = activity coefficient of the hydrogen ion, and

C_{H} = concentration coefficient of the hydrogen ion.

3.2 Buffer Solution — A solution the hydrogen ion concentration of which is not appreciably affected by the addition of small amounts of acid or alkali.

3.3 Buffer Value, β (dm/d*p*H) — The moles of a strong acid or strong base when added to one litre of a solution to change its *p*H value by a unit.

3.4 Dilution Value (Δ *p*H½) — The change of *p*H units if a buffer solution is diluted with an equal volume of water.

3.5 Indicators — Substances that behave like weak acids or weak bases, and their dissociated or undissociated forms have different constitutions and colours.

3.6 Molality (m) (of a Solution) — The amount of solute in moles per 1 000 g of the solvent.

3.7 Molarity (M) (of a Solution) — The amount of solute in moles in one litre of the solution.

3.8 Temperature Coefficient $\left[{}^{\mathrm{d}pH}\!/_{\mathrm{dt}}\right]$ — The change of *p*H units per degree Celsius (Centrigrade) rise of temperature.

4 REAGENTS

4.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests. The reagents of the purity specified against each in **4.1.1** to **4.1.19** shall be used in the preparation of the buffer solutions. Freshly boiled and cooled distilled water shall be used for the preparation of all buffer solutions.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.1.1 *Acetic Acid* — (CH_3COOH; Molecular weight 60.05). Pure (*see* IS 695).

4.1.2 *Borax* — ($Na_2B_4O_7$. $10H_2O$; Molecular weight 381.43). It shall contain not less than 99.5 percent and not more than 100.5 percent of $Na_2B_4O_7$. $10H_2O$ and shall conform to the limits of impurities given below:

Sl No.	*Impurities*	*Percent by Weight, Max*
i)	Carbonate (as CO_3)	0.05
ii)	Chloride (as Cl)	0.001
iii)	Sulphate (as SO_4)	0.005
iv)	Calcium (as Ca)	0.01

NOTE — Before use the borax shall be gradually heated to 110°C and then its temperature raised to 400°C to dehydrate it completely.

4.1.3 *Boric Acid* — (H_3BO_3; Molecular weight 61.84). Analytical reagent grade. Before use the material shall be spread in a dry vacuum desiccator at room temperature preferably overnight.

4.1.4 *Calcium Hydroxide Solution* — [Ca $(OH)_2$, Molecular weight 74.09]. Calcium hydroxide shall be prepared from calcium carbonate of low alkali grade after it has been washed thoroughly. Heat the carbonate slowly to about 1 000°C and ignite at that temperature for at least 45 min. After cooling, add the resulting calcium oxide slowly to water with stirring. Heat the suspension to boiling, cool and filter on a G3 sintered glass funnel. Dry the solid in an oven and crush to a uniform, finely granular state for use.

4.1.4.1 To prepare a saturation solution, shake vigorously an excess of calcium hydroxide, prepared as in **4.1.4**, in a stoppered polyethylene bottle with water at room temperature. Allow the excess of solid to settle and remove the suspended material by filtration with suction on a G3 sintered glass funnel. It is advisable to determine the concentration of a saturated solution of a portion of calcium hydroxide prepared from each particular lot of calcium carbonate. If the concentration of the saturated solution at 25°C as determined alkalimetrically appears to exceed 0.0206 M, the presence of soluble alkalis is indicated. Calcium hydroxide solution shall be free from soluble alkalis.

4.1.5 *Disodium Hydrogen Phosphate* — (Na_2HPO_4; Molecular weight 141.97). It shall contain after drying for 2 h at 110 ± 2°C, not less than 99.5 percent and not more than 100.5 percent of Na_2HPO_4. It shall conform to the limits of impurities given below:

Sl No.	*Impurities*	*Percent by Weight, Max*
i)	Chloride (as Cl)	0.006
ii)	Sulphate (as SO_4)	0.03
iii)	Potassium (as K)	0.05
iv)	Loss on drying at 120 ± 2°C	0.2

4.1.6 *Glycine* — ($H_2N.CH_2COOH$; Molecular weight 75.05). It shall comply with the following requirements:

a) A solution of 2 g glycine in 20 ml of water shall be clear.

b) It shall give no precipitate with barium nitrate solution.

c) It shall show only the slightest opalescence with silver nitrate solution.

d) Sulphated ash obtained from 5 g of glycine shall not exceed 2 mg.

e) Nitrogen content (by Kjeldahl method) shall be 18.67 ± 0.10 percent by weight.

4.1.7 *Hydrochloric Acid* — 0.1 N and 0.2 N.

4.1.8 *Phosphoric Acid* — (H_3PO_4; Molecular weight 98.0). Analytical reagent grade.

4.1.9 *Potassium Hydrogen Phthalate* — ($HOOC.C_6H_4.COOK$; Molecular weight 204.22). It shall contain not less than 99.9 percent and not more than 100.1 percent of $HOOC.C_6H_4.COOK$ and shall conform to the limits of impurities given below:

Sl No.	*Impurities*	*Percent by Weight, Max*
i)	Chloride (as Cl)	0.001
ii)	Sulphate (as SO_4)	0.01
iii)	Sodium (as Na)	0.05
iv)	Moisture	0.1

4.1.10 *Potassium Chloride* — (KCl; Molecular weight 74.55). Analytical reagent grade, recrystallized several times from water and dried at 120 to 150°C.

4.1.11 *Potassium Dihydrogen Phosphate* — (KH_2PO_4; Molecular weight 136.09). The material, after drying at 110 ± 2°C, shall contain not less than 99.5 percent and not more than 100.5 percent of KH_2PO_4. It shall comply with the limits of impurities as given below:

Sl No.	*Impurities*	*Percent by Weight, Max*
i)	Chloride (as Cl)	0.000 5
ii)	Sulphate (as SO_4)	0.01
iii)	Ammonia (as NH_3)	0.001
iv)	Sodium (as Na)	0.05
v)	Loss on drying at 110 ± 2°C	0.1

4.1.12 *Potassium Hydrogen Tartrate* — ($KHC_4H_4O_6$; Molecular weight 188.18). The material, dried at 110 ± 2°C, shall contain not less than 99.8 percent of $KHC_4H_4O_6$. It shall comply with the limits of impurities as given below:

Sl No.	*Impurities*	*Percent by Weight, Max*
i)	Chloride (as Cl)	0.001
ii)	Sulphate (as SO_4)	0.01
iii)	Iron (as Fe)	0.001
iv)	Lead (as Pb)	0.001
v)	Moisture	0.1

4.1.13 *Potassium Tetra-oxalate* — [$KH_3(C_2O_4)_2.2H_2O$; Molecular weight 254.19]. The material shall contain not less than 99.9 percent and not more than 100.1 percent of $KH_3(C_2O_4)_2.2H_2O$. It shall comply with the limits of impurities given below:

Sl No.	*Impurities*	*Percent by Weight, Max*
i)	Chloride (as Cl)	0.002
ii)	Sulphate (as SO_4)	0.01
iii)	Calcium (as Ca)	0.005
iv)	Sodium (as Na)	0.05

4.1.14 *Sodium Acetate* — ($CH_3COONa.3H_2O$; Molecular weight 136.08). Analytical reagent grade.

4.1.15 *Sodium Bicarbonate* — ($NaHCO_3$; Molecular weight 84.01). Analytical reagent grade (*see* IS 2124).

4.1.16 *Sodium Chloride* — (NaCl; Molecular weight 58.45). Analytical reagent grade.

4.1.17 *Sodium Diethyl Barbiturate (Sodium Salt of Veronal)* — (Molecular weight 206.18). It shall contain, after drying, not less than 98.5 percent of $C_8H_{11}O_3N_2Na$. It shall conform to the limits of impurities given below:

Sl No.	*Impurities*	*Percent by Weight, Max*
i)	Loss on drying at 105°C	1.0
ii)	Neutral or basic substances	0.2
iii)	Lead (as Pb)	0.001

4.1.18 *Sodium Hydroxide* — (NaOH; Molecular weight 40.41). Carbonate — free. 0.1N and 0.2N; standarized against 0.1N and 0.2N phthalic acid solution using phenolphthalein as indicator.

4.1.19 *Tris (Hydroxymethyl) Amino Methane* — $[(HOCH_2)_3 = C(NH_2)]$.

5 WORKING BUFFER SOLUTIONS FOR GENERAL PURPOSES

In case of tartrates, citrates, oxalates and phthalates working buffer solutions either fresh solutions shall be prepared or a drop or two of thymol solution shall be added as they are liable to get fungus growth on storage.

5.1 Clark and Lubs Buffer Solutions (25°C)

5.1.1 p*H* 1.2 *to* 2.2 — Prepared by mixing hydrochloric acid (0.2N and potassium chloride solution) (0.2 N and diluting to 1 litre, with water).

Composition per litre of the solution:

Composition/Litre of the Solution		p*H*
Hydrochloric Acid ml	Potassium Chloride Solution ml	
(1)	(2)	(3)
425.0	+ 250	1.2
266.0	+ 250	1.4
162.0	+ 250	1.6
102.0	+ 250	1.8
65.0	+ 250	2.0
39.0	+ 250	2.2

5.1.2 p*H* 2.2 *to* 4.0 — Prepared by mixing hydrochloric acid (0.2 N) and potassium hydrogen phthalate solution (0.2 M) and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Hydrochloric Acid ml	Potassium Hydrogen Phthalate Solution ml	
(1)	(2)	(3)
247.5	+ 250	2.2
211.0	+ 250	2.4
177.0	+ 250	2.6
144.5	+ 250	2.8
111.5	+ 250	3.0
78.5	+ 250	3.2
52.0	+ 250	3.4
31.5	+ 250	3.6
14.5	+ 250	3.8
20.0	+ 250	4.0

5.1.3 p*H* 4.2 *to* 5.8 — Prepared by mixing sodium hydroxide solution (0.2 N) and potassium hydrogen phthalate solution (0.2 M) and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Sodium Hydroxide Solution ml	Potassium Hydrogen Phthalate Solution ml	
(1)	(2)	(3)
15.0	+ 250	4.2
33.0	+ 250	4.4
55.5	+ 250	4.6
82.5	+ 250	4.8
113.0	+ 250	5.0
144.0	+ 250	5.2
170.5	+ 250	5.4
194.0	+ 250	5.6
211.5	+ 250	5.8

5.1.4 p*H* 5.8 *to* 8.0 — Prepared by mixing sodium hydroxide solution (0.2 N) and potassium dihydrogen phosphate (0.2 M) and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Sodium Hydrochloric Solution ml	Potassium Dihydrogen Phosphate Solution ~~ml~~	
(1)	(2)	(3)
18.0	+ 250	5.8
28.0	+ 250	6.0
40.5	+ 250	6.2
58.0	+ 250	6.4
82.0	+ 250	6.6
112.0	+ 250	6.8
145.5	+ 250	7.0
173.5	+ 250	7.2
195.5	+ 250	7.4
212.0	+ 250	7.6
222.5	+ 250	7.8
230.5	+ 250	8.0

5.1.5 p*H* 8.0 *to* 10.0 — Prepared by mixing sodium hydroxide solution (0.2 N), and boric acid solution (0.2 M) in potassium chloride solution (0.2 M) and diluting to one litre with water. These solutions shall be checked to account for the influence of carbon dioxide.

Composition/Litre of the Solution		p*H*
Sodium Hydroxide Solution ml	Boric Acid-Potassium Chloride Solution ml	
(1)	(2)	(3)
19.5	+ 250	8.0
30.0	+ 250	8.2
43.0	+ 250	8.4
59.0	+ 250	8.6
79.0	+ 250	8.8
104.0	+ 250	9.0
132.0	+ 250	9.2
160.5	+ 250	9.4
184.5	+ 250	9.6
203.0	+ 250	9.8
218.5	+ 250	10.0

5.2 Sorensen Buffer Solution (18°C)

5.2.1 p*H* 1.04 *to* 3.68 — Prepared by mixing hydrochloric acid (0.1 N) and glycine solution (0.1 M) in sodium chloride solution (0.1 N).

Composition/Litre of the Solution		p*H*
Glycine-Sodium Chloride Solution ml	Hydrochloride Acid ml	
(1)	(2)	(3)
0.0	+ 100	1.04
10.0	+ 90	1.15
20.0	+ 80	1.25
30.0	+ 70	1.42
40.0	+ 60	1.64
50.0	+ 50	1.93
60.0	+ 40	2.28
70.5	+ 30	2.61
80.0	+ 20	2.92
90.0	+ 10	3.34
95.0	+ 5	3.68

5.2.2 p*H* 8.24 *to* 10.14 — Prepared by mixing sodium hydroxide solution (0.1 N), and glycine solution (0.1 M) in sodium chloride solution (0.1 N).

Composition/Litre of the Solution		p*H*
Glycine-Sodium Chloride Solution ml	Sodium Hydroxide Solution ml	
(1)	(2)	(3)
97.5	+ 2.5	8.24
95.0	+ 5.0	8.58
90.0	+ 10.0	8.93
80.0	+ 20.0	9.36
70.0	+ 30.0	9.71
60.0	+ 40.0	10.14

5.3 Michaells Buffer Solutions (25°C)

5.3.1 *Veronal — Sodium Hydroxide Buffer Solutions for Physiological Purposes (*p*H* 7.00 *to* 9.20*)* — Prepared with sodium salt of veronal solution (0.1 M) and hydrochloric acid (0.1 N).

Composition/Litre of the Solution		p*H*
Sodium Salt of Veronal Solution ml	Hydrochloric Acid ml	
(1)	(2)	(3)
53.6	+ 46.4	7.00
55.4	+ 44.6	7.20
58.1	+ 41.9	7.40
61.5	+ 38.5	7.60
66.2	+ 33.8	7.80
71.6	+ 28.4	8.00
76.9	+ 23.1	8.20
82.3	+ 17.7	8.40
87.1	+ 12.9	8.60
90.8	+ 9.2	8.80
93.6	+ 6.4	9.00
95.2	+ 4.8	9.20

5.3.2 *Veronal — Acetate Buffer Solutions (*p*H* 2.62 *to* 9.64*)* — Isotonic with blood at 25°C. The veronal-acetate solution shall contain 19.43 g of sodium acetate and 29.43 g of sodium salt of veronal in 1 litre of the solution made in carbon dioxide-free water.

5.3.2.1 To make 25 ml of buffer solution, take 5 ml of the veronal acetate solution (*see* **4.3.2**) and mix 2 ml of sodium chloride solution (8.5 percent *w/v*) and the following amounts of hydrochloric acid (0.1 N) and dilute to 25 ml with carbon dioxide-free water.

Volume of Hydrochloric Acid ml	p*H*
(1)	(2)
0.0	9.64
0.25	9.16
0.50	8.90
0.75	8.68
1.0	8.55
2.0	8.18
3.0	7.90
4.0	7.66
5.0	7.42
5.5	7.25
6.0	6.99
6.5	6.75
7.0	6.12
8.0	5.32
9.0	4.93
10.0	4.66
11.0	4.33
12.0	4.13

(1)	(2)
13.0	3.88
14.0	3.62
15.0	3.20
16.0	2.62

5.4 Britton and Robinson Universal Buffer Solutions (*p*H 1.81 to 11.98) — Prepared by adding the following volumes of sodium hydroxide solution (0.2 N) to 100 ml of a solution of mixed acid, being 0.04 M with respect to phosphoric acid, 0.04 N with respect to both acetic and boric acids.

Volume of Sodium Hydrochloric Solution ml (1)	*p*H (2)
0.0	1.81
2.5	1.89
5.0	1.98
7.5	2.09
10.0	2.21
12.5	2.36
15.0	2.56
17.5	2.87
20.0	3.29
22.5	3.78
25.0	4.10
27.5	4.35
30.0	4.56
32.5	4.78
35.0	5.02
37.5	5.33
40.0	5.72
42.5	6.09
45.0	6.37
47.5	6.59
50.0	6.80
52.5	7.00
55.0	7.24
57.5	7.54
60.0	7.96
62.5	8.36
65.0	8.69
67.5	8.95
70.0	9.15
72.5	9.37
75.0	9.62
77.5	9.91
80.0	10.38
82.5	10.88
85.0	11.20
87.5	11.40
90.0	11.58
92.5	11.70
95.0	11.82
97.5	11.92
100.0	11.98

5.5 Buffer Solution of *p*H 9.6 to 13.0 (25°C)

5.5.1 p*H* 9.6 *to* 11.0 — Prepared by mixing sodium bicarbonate solution (0.05 M) and sodium hydroxide solution (0.1 N) and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Sodium Hydroxide Solution ml (1)	Bicarbonate Solution ml (2)	(3)
50.0	+ 500	9.60
76.0	+ 500	9.80
107.0	+ 500	10.00
138.0	+ 500	10.20
165.0	+ 500	10.40
191.0	+ 500	10.60
212.0	+ 500	10.80
227.0	+ 500	11.00

5.5.2 p*H* 10.9 *to* 12.0 — Prepared by mixing disodium hydrogen phosphate solution (0.05 M) and sodium hydroxide solution (0.1 N) and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Sodium Hydroxide Solution ml (1)	Disodium Hydrogen Phosphate Solution ml (2)	(3)
33.0	+ 500	10.90
41.0	+ 500	11.00
63.0	+ 500	11.20
91.0	+ 500	11.40
135.0	+ 500	11.60
194.0	+ 500	11.80
269.0	+ 500	12.00

5.5.3 p*H* 12.0 *to* 13.0 — Prepared by mixing potassium chloride solution (0.2 M) and sodium hydroxide solution (0.2 N) and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Sodium Hydroxide Solution ml (1)	Potassium Chloride Solution ml (2)	(3)
60.0	+ 250	12.00
102.0	+ 250	12.20
162.0	+ 250	12.40
256.0	+ 250	12.60
412.0	+ 250	12.80
660.0	+ 250	13.00

5.6 Gomori Buffer Solutions (25°C) *p*H 7 to 9 for Physiological Purposes

Prepared by mixing 0.1 M tris (hydroxymethyl) aminomethane and 0.1 N hydrochloric acid and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Hydrochloric Acid ml	Tris Hydroxymethyl Aminomethane Solution ml	
(1)	(2)	(3)
466	+ 500	7.00
447	+ 500	7.20
420	+ 500	7.40
385	+ 500	7.60
345	+ 500	7.80
292	+ 500	8.00
229	+ 500	8.20
172	+ 500	8.40
124	+ 500	8.60
85	+ 500	8.80
57	+ 500	9.00

5.7 Walpole Buffer Solutions (18°C) *p*H 3.42 to 5.89

Prepared by mixing 0.2 N acetic acid and 0.2 M sodium acetate solution and diluting to one litre with water.

Composition/Litre of the Solution		p*H*
Acetic Acid ml	Sodium Acetate Solution ml	
(1)	(2)	(3)
950	+ 50	3.42
900	+ 100	3.72
800	+ 200	4.05
700	+ 300	4.27
600	+ 400	4.45
500	+ 500	4.63
400	+ 600	4.80
300	+ 700	4.99
200	+ 800	5.23
150	+ 850	5.37
100	+ 900	5.57
50	+ 950	5.89

6 PRIMARY AND SECONDARY BUFFER SOLUTION

6.1 For checking the *p*H of solutions prepared (*see* **4**), a *p*H meter shall be employed which shall be calibrated against primary and secondary buffer solutions given in Table 1. The solution given in a arranged in the table in the order of increasing *p*H values.

7 COLORIMETRIC METHODS OF DETERMINATION OF *p*H

7.1 Indicator Paper Method

7.1.1 *Materials*

7.1.1.1 *Indicator papers*

Supplied in booklets carrying the colour chart of twenty leaves measuring 64 mm × 12.5 mm and of range as follows:

Bromocresol green	Methyl red phenolphthalein
Bromothymol blue	Thymol blue
Chlorophenol red	Thymolphthalein
Methyl orange	Titan yellow (clayton yellow)

The paper may also be supplied in rolled tapes of about 5 m long and 10 m wide in a convenient plastic dispenser carrying clearly the printed *p*H colour chart.

7.1.2 *Procedure*

The leaf of the indicator paper or a strip cut from the dispenser is dipped in the test solution contained in a clean test tube. Compare the colour of the moistened indicator paper with those printed on the booklet or dispenser. The *p*H of the solution is designated as the

Table 1 Standard Primary and Secondary Buffer Solutions at 25°C

(*Clause* 6.1)

Sl No.	Buffer Solutions	Molality	Weight (in Air) of Buffer Substance at 25°C	*p*H at 25°C	Dilution Value (Δ *p*H½)	Buffer Value	Temperature Coefficient d*p*H/dt
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Potassium tetra-oxalate	0.05	12.61	1.679	+ 0.186	0.070	+ 0.001
ii)	Potassium hydrogen tartrate	0.034 (approx)	Saturated at 25°C	3.357	+ 0.049	0.027	– 0.001 4
iii)	Potassium hydrogen phthalate	0.05	10.12	4.008	+ 0.052	0.016	+ 0.001 2
iv)	Potassium dihydrogen phosphate PLUS	0.025	3.39	6.865	+ 0.080	0.029	– 0.002 3
	disodium hydrogen phosphate	0.025	3.53				
v)	Potassium dihydrogen phosphate PLUS	0.008 695	1.179	7.413	+0.07	0.016	– 0.002 8
	disodium hydrogen phosphate	0.030 43	4.30				
vi)	Borax	0.01	3.80	9.180	+0.01	0.020	– 0.008 2
vii)	Calcium hydroxide	0.020 3	Saturated at 25°C	12.454	-0.28	0.09	– 0.033

NOTE — Solution (ii) to (vi) are primary standard, and (i) and (vii) are secondary standard.

number written on a particular colour shade with which the colour of the moistened indicator paper matches. In case the colour is intermediate to two colour standards, express the *p*H to the one with which the moistened indicator paper matches more closely.

7.2 Comparator Method

7.2.0 *Outline of the Method*

The *p*H of an unknown solution is determined by testing it with universal indicator; solution and the approximate value is found. The *p*H value is then determined by selecting an indicator with a suitable colour-change interval.

7.2.1 *Apparatus*

7.2.1.1 *Comparator* — Any suitable comparator with ampoules of buffer solutions of known and accurate *p*H value to which the indicator is added. Alternatively, coloured glass slides may also be used.

NOTE — Any commercial comparator with indicator's kit or slides is suitable.

7.2.1.2 *Test tube,* of glass.

7.2.2 *Reagents*

7.2.2.1 *Indicators* — The common indicators used for the determination of *p*H are listing in Annex A.

7.2.2.2 *Universal indicator* — Dissolve in 100 ml of alcohol the following in the given order:

0.06 g of methyl yellow, 0.04 g of methyl red, 0.08 g of bromothymol blue, 0.10 g of thymol blue, and 0.02 of phenolphthalein.

Titrate the solution to yellow colour with 0.1 N sodium hydroxide solution. The indicator has the colour value as given below:

a) *p*H 1 Cherry-red
b) *p*H 2 Rose
c) *p*H 3 Red-orange
d) *p*H 4 Orange-red
e) *p*H 5 Orange
f) *p*H 6 Yellow
g) *p*H 7 Yellow-green
h) *p*H 8 Green
j) *p*H 9 Blue-green
k) *p*H 10 Blue

7.2.2.3 *Buffer solutions* — Buffer solutions are prepared from chemicals of analytical reagent grade. They should be stored in clean bottles, each bottle carrying the date and *p*H as they are liable to deteriorate with time. Commonly used buffer solutions are listed in Annex B.

The coloured buffer solutions are prepared by adding the correct quantity of the required indicator to the correct volume of the buffer and the solution sealed in an ampoule.

7.2.3 *Procedure*

To determine the *p*H of a solution, the indicator used shall be one which given a colour of the solution intermediate between those shown at the extremes of its *p*H range. Determine the approximate *p*H by adding to a small sample of the solution a little of universal indicator and find out the range. Measure out 10 ml of the solution whose *p*H has to be measured and transfer to a clean glass tube and add the correct quantity of the indicator. Normally 0.1 to 0.2 ml of the indicator solution is sufficient. The colour developed is compared with that of the buffer tubes of known *p*H value in a comparator as given in **7.2.3.1**.

7.2.3.1 Place three test tubes filled with liquids in spaces *A, B* and *C*, add the indicator to *B* only in the space *E* provided in front of it, place a test tube filled with distilled water. Place the buffer tubes containing the indicator in spaces *D* and *F* and change until a correct colour match is obtained when viewed. The solution is assigned the *p*H of the buffer with which it closely matches (*see* Fig. 1).

Alternatively, the *p*H of a solution may be determined using permanent colour glass standards,usually arranged as circles of suitable coloured glasses fixed in a circular disc, in such a way that selection of the appropriate colour.

7.3 Electrometric Method

Electrometric *p*H measurements are made with a suitable electrode system which develops a potential (emf) proportional to the *p*H of the solution.

7.3.1 *Apparatus*

7.3.1.1 *p*H *meter* — Direct reading type conforming to IS 2711.

7.3.1.2 *Calomel and glass electrodes* — Saturated calomel electrode shall be used in *p*H meter assembly. A few crystals of solid potassium chloride shall be present within the chamber surrounding the calomel at each temperature. The design of the electrode shall be such as to allow a fresh liquid junction between the solution of potassium chloride and the buffer or test solution.

The glass electrode shall be calibrated as prescribed in **7.3.2.1**. If the electrodes are used outside the electrode compartment of the assembly the leads shall be protected from the effects of body capacitance.

To attain equilibrium as quickly as possible when using a glass electrode, the electrode shall be kept under water when not in use so that it does not become dry externally.

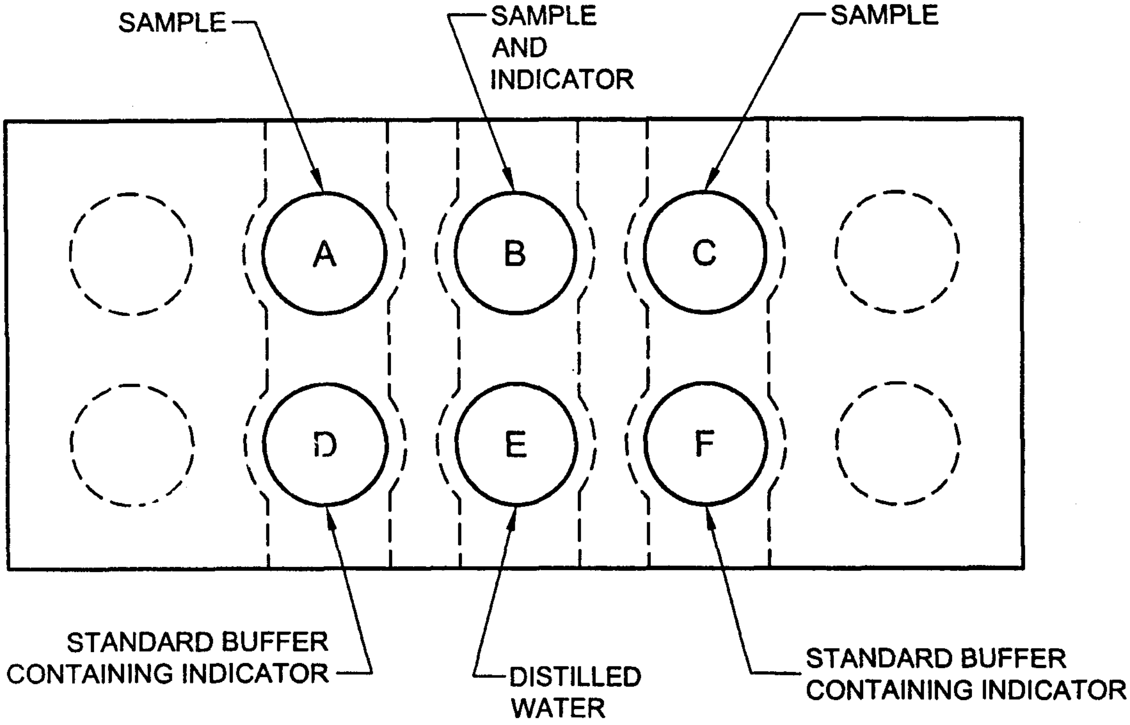

FIG. 1 ASSEMBLY DIAGRAM OF TUBES IN A COMPARATOR

7.3.2 *Procedure*

7.3.2.1 *Standardization*

The assembly shall be put on, allowed to warm up thoroughly and brought to electrical balance as per manufacturer's instructions. Wash the glass and calomel electrodes and the sample cup-thoroughly twice with distilled water and wipe dry with a clean absorbent paper. Note the temperature of the test solution and adjust the temperature dial of the meter to proper setting. Select two standard solutions from Table 1 as to bracket the anticipated *p*H of the test solution and the standard solutions shall be brought to the temperature of the test solution within 2°C. Fill the sample cup with the first standard solution and immerse the electrodes. Set the dial of the meter to the known *p*H or potential corresponding to the known *p*H of the standard solution at appropriate temperature. Bring the meter to the balance by engaging button and rotating asymmetry potential knob. Fill the sample cup repeatedly with additional portions of standard solution until the meter remains in balance within ± 0.02 *p*H unit for two successive portions without a change in the position of the asymmetry knob. To eliminate the effect of thermal and electrical hysteresis, the temperature of electrodes, standard solutions and wash water shall be kept very close to that of the test solution.

Wash the electrodes and sample cup thoroughly with water and form a fresh liquid junction. Place the second standard in the cup and adjust the meter to new balance point and read the *p*H. The setting on the asymmetry potential knob shall not be changed. Use additional portions of second standard solution until successive readings of *p*H agree within 0.05 units. The meter is deemed to work satisfactorily if the reading obtained for the second standard solution is within the permissible limits of error.

If the anticipated *p*H of the solution is greater than 10, disodium tetraborate and saturated calcium hydroxide solutions (*see* Table 2) shall be used for standardization. The meter, in this case, is considered to be working satisfactorily if the reading obtained for the second standard solution agreed with the assigned *p*H within 0.1 unit.

If the *p*H determinations are made only occasionally, the meter shall be standardized each time.

7.3.2.2 p*H of test solution*

Wash the electrodes and sample cup and wipe dry with a clean absorbent paper. Fill the cup with a portion of the test solution and form a fresh liquid junction. The meter is operated and *p*H directly read. In case of well buffered test solution, one to three portions will usually be sufficient to yield *p*H values reducible to ± 0.05 units. In case of slightly buffered solutions or distilled water six or more portions may be necessary.

7.3.2.3 Report the *p*H obtained for the test solution to the nearest 0.1 unit.

Table 2 Standard Solutions

(*Clause* 7.3.2.1)

Sl No.	Temperature °C	*p*H of Solution				
		0.05 M Potassium Tetroxalate	0.05 M Potassium Hydrogen Phthalate	0.025 M Potassium Dihydrogen Phosphate + 0.025 M Disodium Hydrogen Phosphate	0.01 M Disodium Tetraborate	Saturated Calcium Hydroxide
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	0	1.67	4.01	6.98	9.46	13.43
ii)	10	1.67	4.00	6.92	9.33	13.00
iii)	20	1.68	4.00	6.88	9.22	12.63
iv)	25	1.68	4.01	6.86	9.18	12.45
v)	30	1.69	4.01	6.85	9.14	12.30
vi)	35	1.69	4.02	6.84	9.10	12.14
vii)	40	1.70	4.03	6.84	9.07	11.99
viii)	50	1.71	4.06	6.83	9.01	11.58
ix)	60	1.73	4.09	6.84	8.96	11.45

ANNEX A

(*Clause* 7.2.2.1)

INDICATORS FOR DETERMINATION OF *p*H

A-1 PREPARATION

The indicators shall be prepared as shown in Table 3. The water used for preparation shall be carbon dioxide-free. Similarly ethyl alcohol used should also be neutral. When the indicator solution is to be prepared with 0.02N sodium hydroxide solution, tritrate well the quantity of the indicator material with the volume of 0.02 N sodium hydroxide solution, in an agate mortar, and dilute with carbon dioxide-free water to the volume indicated.

NOTE — The commercially available, ready to use, indicator solutions may also be used for determination of *p*H.

Table 3 Preparation of Indicator Solution

(*Clause* A-1.1)

Sl No.	Indicator	Range	Colour		Preparation
			Acid	Base	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Thymol blue	1.2 to 2.8	Red	Yellow	Dissove 0.10 g in 10.75 ml of 0.02 N sodium hydroxide and dilute to 250 ml with water
ii)	Methyl yellow	2.9 to 4.0	Red	Yellow	Dissolve 0.10 g in 100 ml of alcohol
iii)	Methyl orange	3.1 to 4.4	Red	Orange-Yellow	Dissolve 0.2 g in 100 ml of hot water
iv)	Bromophenol blue	3.0 to 4.6	Yellow	Blue-Violet	Dissolve 0.10 g in 7.45 ml of 0.02 N sodium hydroxide and dilute to 250 ml with water
v)	Bromocresol green	3.8 to 5.4	Yellow	Blue	Dissolve 0.10 g in 7.15 ml of 0.02 N sodium hydroxide and dilute to 250 ml with water
vi)	Methyl red	4.2 to 6.3	Red	Yellow	Dissolve 0.10 g in 18.6 ml of 0.02 N sodium hydroxide and dilute to 250 ml with water
vii)	Bromothymol blue	6.0 to 7.6	Yellow	Blue	Dissolve 0.10 g in 8.0 ml of 0.02 N sodium hydroxide and dilute to 250 ml with water
viii)	Phenol red	6.4 to 8.0	Yellow	Red	Dissolve 0.10 g in 14.20 ml of 0.02 N sodium hydroxide and dilute to 250 ml with water
ix)	Metacresol purple	7.4 to 9.0	Yellow	Purple	Dissolve 0.10 g in 13.1 ml of 0.02 N sodium hydroxide and dilute to 250 ml with water
x)	Thymol blue	8.0 to 9.6	Yellow	Blue	Dissolve 0.10 g in 10.75 ml of 0.02N sodium hydroxide and dilute to 250 ml with water
xi)	Phenolphthalein	8.2 to 9.8	Colour-less	Red-Violet	Dissolve 0.10 g in 100 ml of 80 percent alcohol
xii)	Thymolphthalein	9.3 to 10.5	Colour-less	Blue	Dissove 0.10 g in 100 ml of 80 percent alcohol
xiii)	Alizarine yellow	10.1 to 12.0	Yellow	Violet	Dissolve 0.10 g in 100 ml of 50 percent alcohol
xiv)	Nitramine	11.0 to 13.0	Yellow	Orange-Brown	Dissolve 0.10 g in 100 ml of 70 percent alcohol
xv)	1, 3, 5-Trinitrobenzene	11.5 to 14.0	Colour-less	Yellow	Dissolve 0.10 g in 100 ml of alcohol

ANNEX B

(*Clause* 7.2.2.3)

BUFFER SOLUTION

B-1 Buffer solutions shall be prepared by diluting the specified quantity of solution given below to 100 ml:

p*H* *at* 20°*C*	*Solution*						
	M/5 Potassium Chloride ml	M/5 Hydrochloric Acid ml	M/10 Potassium Hydrogen Phthalate ml	M/10 Hydrochloric Acid ml	M/10 Sodium Hydroxide ml	M/10 Potassium Dihydrogen Orthophosphate ml	M/10 Boric Acid and M/10 Potassium Chloride ml
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
1.0	25	48.5	–	–	–	–	–
1.2	25	32.25	–	–	–	–	–
1.4	25	20.75	–	–	–	–	–
1.6	25	13.15	–	–	–	–	–
1.8	25	8.3	–	–	–	–	–
2.0	25	5.3	–	–	–	–	–
2.2	25	3.35	(50)	(46.70)	–	–	–
2.4	–	–	50	39.60	–	–	–
2.6	–	–	50	32.95	–	–	–
2.8	–	–	50	26.42	–	–	–
3.0	–	–	50	20.32	–	–	–
3.2	–	–	50	14.70	–	–	–
3.4	–	–	50	9.90	–	–	–
3.6	–	–	50	5.97	–	–	–
3.8	–	–	50	2.63	–	–	–
4.0	–	–	50	–	0.40	–	–
4.2	–	–	50	–	3.70	–	–
4.4	–	–	50	–	7.50	–	–
4.6	–	–	50	–	12.15	–	–
4.8	–	–	50	–	17.70	–	–
5.0	–	–	50	–	23.85	–	–
5.2	–	–	50	–	29.95	–	–
5.4	–	–	50	–	35.45	–	–
5.6	–	–	50	–	39.85	–	–
5.8	–	–	50	–	43.00	–	–
6.0	–	–	50	–	45.45		–
					(5.70)	(50)	
6.2	–	–	–	–	8.60	50	–
6.4	–	–	–	–	12.60	50	–
6.6	–	–	–	–	17.80	50	–
6.8	–	–	–	–	23.65	50	–
7.0	–	–	–	–	29.63	50	–
7.2	–	–	–	–	35.00	50	–
7.4	–	–	–	–	39.50	50	–
7.6	–	–	–	–	42.80	50	–
7.8	–	–	–	–	45.20	50	(50)
					(2.61)		

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
8.0	–	–	–	–	46.80 (3.97)	50	(50)
8.2	–	–	–	–	5.90	–	50
8.4	–	–	–	–	8.50	–	50
8.6	–	–	–	–	12.00	–	50
8.8	–	–	–	–	16.30	–	50
9.0	–	–	–	–	21.30	–	50
9.2	–	–	–	–	26.70	–	50
9.4	–	–	–	–	–	–	50
9.6	–	–	–	–	36.85	–	50
9.8	–	–	–	–	40.80	–	50
10.0	–	–	–	–	43.90	–	50

NOTE — Figures in parentheses belong to the same set.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

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