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SPECIFICATION FOR FORMALDEHYDE SOLUTION

(First Revision)

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Indian Standard

SPECIFICATION FOR FORMALDEHYDE SOLUTION

(First Revision)

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Indian Standard

SPECIFICATION FOR FORMALDEHYDE SOLUTION

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 31 December 1973, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1965. The Sectional Committee responsible for the preparation of this standard reviewed it and decided to revise the standard and align the methods of test with ISO Recommendations. In this revised standard, the requirements and methods of test for heavy metals and *p*H value have been included and a chemical method for the determination of methanol content in addition to the graphical method has been added.

0.3 Formaldehyde, also known as formalin is used in the manufacture of synthetic resins, dyes, explosives and other chemicals. It is also used in embalming fluids, brewing, distilled liquors, inks, leather, waterproofing straw hats, waterproofing paper and as a hardening and preservative agent. It also finds wide application in rayon, textile processing, silver mirrors, metallurgy, rubber, in fertilizers and as an inhibitor in oil wells.

0.4 This standard contains clause **2.3** which calls for an agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard lays down the requirements and the methods of sampling and test for formaldehyde solution and also meets the requirements for processing of photosensitized materials.

*Rules for rounding off numerical values (*revised*).

1.1.1 This standard does not, however, apply to the material intended for medicinal purposes.

2. REQUIREMENTS

2.1 Description — The material shall be clear and nearly colourless (*see* Note), having a characteristic pungent and irritating odour. It shall consist essentially of a solution of formaldehyde (HCHO) in water, with methanol as a stabilizing agent. It shall be free from foreign matter and from precipitated polymer.

NOTE — On long standing the solution may become cloudy due to separation of paraformaldehyde, which will disappear when the solution is warmed.

2.2 The material shall comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR FORMALDEHYDE SOLUTION

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Acidity (as HCOOH), percent by mass, *Max*	0·05	A-2
ii)	Ash, percent by mass, *Max*	0·01	A-3
iii)	Aldehyde content (as HCHO), percent by mass (*m/m*)	37·0 ± 0·5	A-4
iv)	Iron (as Fe), parts per million, *Max*:		A-5
	a) In non-iron containers	2	
	b) In iron containers	10	
v)	Methanol content, percent by mass	4 to 6	A-6

2.3 The requirements for heavy metals (other than iron) and *p*H at 27 ± 1°C shall be subject to agreement between the purchaser and the supplier. The tests prescribed under **A-7** and **A-8** shall be followed.

3. PACKING AND MARKING

3.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in air-tight containers made of stainless steel, aluminium or glass or in steel drums suitably lined with acid-resistant material. The material may also be packed in HDPE barrels. It is desirable to store it in a place not below 17°C.

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and recognized trade-mark, if any;

c) Net mass of the material; and

d) Lot or batch number, in code or otherwise.

3.3 BIS Certification Marking

The product may also be marked with Standard Mark.

3.3.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

ANALYSIS OF FORMALDEHYDE SOLUTION

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

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

A-2. DETERMINATION OF ACIDITY

A-2.1 Reagents

A-2.1.1 *Standard Sodium Hydroxide Solution* — 0·1 N.

A-2.1.2 *Bromothymol Blue Indicator Solution* — Grind 0·4 g of powdered bromothymol blue in an agate mortar with 6·4 ml of the sodium hydroxide solution and dilute to 1 000 ml with freshly boiled and cooled distilled water.

*Specification for water, distilled quality (*revised*).

A-2.2 Procedure — Mix 100 ml of the material with 100 ml of freshly boiled and cooled distilled water in a 500-ml conical flask and titrate the mixture with standard sodium hydroxide solution, using four drops of bromothymol blue indicator.

A-2.3 Calculation

$$\text{Acidity (as HCOOH), percent by mass} = \frac{0{\cdot}046\ V N}{S}$$

where

V = volume in ml of standard sodium hydroxide solution used,

N = normality of standard sodium hydroxide solution, and

S = relative density of the material at room temperature.

A-3. DETERMINATION OF ASH

A-3.1 Procedure — Place 100 ml of the material in a 500-ml beaker on a hot plate and evaporate until the volume is reduced to 50 ml. Transfer the liquid quantitatively to a platinum or silica basin previously heated to 600 ± 30°C and cooled in a desiccator and weighed and evaporate to dryness on a boiling water-bath. Heat the basin and its contents gently at first to volatalize the paraformaldehyde and then at 600 ± 30°C in an electric furnace until the mass is constant. Cool in a desiccator and weigh.

A-3.2 Calculation

$$\text{Ash, percent by mass} = \frac{M}{S}$$

where

M = mass in g of the residue, and

S = relative density of the material at room temperature.

A-4. DETERMINATION OF ALDEHYDE CONTENT

A-4.0 General — The method is based on the quantitative liberation of sodium hydroxide when formaldehyde reacts with sodium sulphite to form the formaldehyde bisulphite addition product.

A-4.1 Reagents

A-4.1.1 *Sodium Sulphite Solution* — Dissolve 126 g of anhydrous sodium sulphite, or 252 g of the hydrated salt, in water and dilute to 1 000 ml.

A-4.1.2 *Thymolphthalein Indicator Solution* — Dissolve 0·1 g of thymolphthalein in 100 ml of 80 percent rectified spirit (conforming to IS : 323-1959*).

*Specification for rectified spirit (*revised*).

A-4.1.3 *Standard Hydrochloric Acid* — 0·1 N and 1 N.

A-4.1.4 *Sodium Hydroxide Solution* — 0·1 N.

A-4.2 Procedure

A-4.2.1 Weigh accurately a glass-stoppered conical flask containing 10 ml of distilled water. Add to it about 3 ml of the formaldehyde solution and reweigh. Add two drops of (approximately 0·1 ml) of the thymolphthalein indicator solution followed by 0·1 N sodium hydroxide solution, drop by drop until a faint blue colour is just perceptible.

A-4.2.2 Measure 75 ml of freshly prepared sodium sulphite solution into a second 250-ml conical flask. To it add two drops (approximately 0·1 ml) of the thymolphthalein indicator solution followed by 0·1 N hydrochloric acid solution until the blue colour just disappears. Add this solution to the neutralized formaldehyde solution previously prepared.

A-4.2.3 Mix the two solutions by swirling for two minutes and then titrate with 1 N hydrochloric acid until the blue colour just disappears.

A-4.3 Calculation

$$\text{Aldehyde content (as HCHO), percent by mass} = \frac{3{\cdot}003\ V\ N}{M}$$

where

V = volume in ml of standard hydrochloric acid required for the titration,

N = normality of standard hydrochloric acid used for titration, and

M = mass of the material taken for the test.

A-5. DETERMINATION OF IRON

A-5.1 Method A (Thioglycollic Acid Method)

A-5.1.1 *Reagents*

A-5.1.1.1 *Dilute sulphuric acid* — 20 percent (*v*/*v*).

A-5.1.1.2 *Thioglycollic acid* — 10 percent solution (*v*/*v*) in water.

A-5.1.1.3 *Hydrogen peroxide* — 15 percent (*m*/*m*).

A-5.1.1.4 *Standard iron solution* — Dissolve 7·022 g of ferrous ammonium sulphate in a mixture of 600 ml of water and 350 ml of dilute sulphuric acid, and dilute to 1 000 ml with water. Further dilute 10 ml of the solution so obtained to 1 000 ml with water. One millilitre of the diluted solution contains 0·01 mg of iron (as Fe).

A-5.1.2 Procedure

A-5.1.2.1 Place 50 ml of the material in the case of a bulk consignment or 10 ml of the material in the case of a small container, in a 400-ml beaker and add to it 10 ml of dilute sulphuric acid. Evaporate the mixture on a hot-plate until acid fumes are evolved. Allow to cool to room temperature. Add to the cold solution, in successive small portions, 10 ml of hydrogen peroxide and heat on a hot-plate until acid fumes are evolved. Allow to cool again to room temperature and transfer the solution to a 50-ml Nessler cylinder. Wash the beaker with a small quantity of water and transfer the washings to the cylinder. Add 1 ml of thioglycollic acid solution to the mixture and make slightly alkaline with ammonia solution. Mix thoroughly and dilute with water to the graduation mark.

A-5.1.2.2 To 40 ml of water in another 50-ml Nessler cylinder, add 1 ml of thioglycollic acid and after making the mixture slightly alkaline with ammonia solution, run in standard iron solution from a burette, shaking with each addition until the intensity of the colour in the two cylinders is identical when they are viewed along their axis. Record the volume of standard iron solution added.

A-5.1.2.3 At the same time carry out a blank test on the reagents alone, and determine, in the same manner as for the test, the amount of iron present.

A-5.1.3 *Calculation*

$$\text{Iron (as Fe), parts per million} = \frac{10\,(V_1 - V_2)}{VS}$$

where

V_1 = volume in ml of standard iron solution added to Nessler cylinder in the case of the sample,

V_2 = volume in ml of standard iron solution added to another Nessler cylinder in the case of the blank test,

V = volume in ml of formaldehyde solution taken for the test, and

S = relative density of the formaldehyde solution at room temperature.

A-5.2 Method B (2, 2'-Bipyridyl Method)

A-5.2.0 *General* — Ferrous iron gives with 2, 2'-bipyridyl a red coloured complex having maximum absorption at 550 nm. The molecular absorption coefficient of the complex is 8000. The iron is first reduced into ferrous state for complex formation.

A-5.2.1 *Reagents*

A-5.2.1.1 *Dilute sulphuric acid* — approximately 0·1 N solution.

A-5.2.1.2 *Hydrogen peroxide* — 15 percent (*m/m*).

A-5.2.1.3 *Hydroxyl ammonium chloride* — 10 percent solution in water.

A-5.2.1.4 *Ammonium acetate* — Dissolve 50 g of ammonium acetate in water and dilute to 100 ml.

A-5.2.1.5 2, 2'-*Bipyridyl solution* — Dissolve 0·5 g of 2, 2'-bipyridyl in 10 ml of approximately 1 N hydrochloric acid solution and dilute to 100 ml.

A-5.2.1.6 *Standard iron solution* — Dissolve 7·02 g ammonium ferrous sulphate [$(NH_4)_2SO_4.FeSO_4.6H_2O$] in water containing 10 ml of concentrated sulphuric and dilute with water to 1 000 ml. One millilitre of this solution contains 1·0 mg of iron (as Fe). It may be diluted suitably to contain 10, 25 or 100 μg of iron per millilitre.

A-5.2.2 *Procedure* — To several aliquots of the standard iron solution containing 20, 40, 70, 100, 150 and 200 μg of iron, add 10 ml of sulphuric acid and evaporate on a sand-bath until white fumes are fast evolved. Allow to cool to room temperature and add in successive small portions 10 ml of hydrogen peroxide solution and heat on the sand-bath again to evolve out white fumes of sulphuric acid. Again cool and transfer the contents quantitatively to 100 ml one-mark flask. To each flask, add 2 ml of hydroxylammonium chloride solution, mix and allow to stand for 2 minutes. Then add 30 ml of ammonium acetate solution and 5 ml of 2, 2'-bipyridyl solution, dilute to the mark and mix thoroughly.

Determine the absorbance against a reagent blank at 550 nm using a 1-cm cell. Plot absorbance against concentration of iron (in μg/ml).

A-5.2.2.1 Take an aliquot of clear solution of the material containing 0·01 to 0·02 mg of iron. Determine the absorbance of the test solution by treating an aliquot of the sample solution in the same manner as described above against a reagent blank using 1-cm cell. Read the concentration of iron (in μg/ml) from the calibration curve.

A-5.2.3 *Calculation*

$$\text{Iron (as Fe), percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the iron found in the sample solution, and

M_2 = mass in g of the material taken for the test.

A-6. DETERMINATION OF METHANOL CONTENT

A-6.0 Methods — Two methods, namely, Method A and Method B have been prescribed. In case of dispute Method B shall be used as a referee method.

A-6.1 Method A (Graphical Method)

A-6.1.1 *Procedure*

A-6.1.1.1 Determine the relative density of the material at 25/25°C by any suitable method capable of giving a result accurate to ± 0·001.

A-6.1.1.2 Read the value of methanol content percentage by mass from the graph in Fig. 1 in which the relationship between the formaldehyde content (as determined in **A-4**), relative density at 25/25°C and methanol content is plotted.

A-6.2 Method B (Chemical Method)

A-6.2.1 *Reagents*

A-6.2.1.1 *Concentrated sulphuric acid* — *See* IS : 266-1961*.

A-6.2.1.2 *Chromic oxide solution* — Dissolve 83·3 g of pure oxide of chromium (Cr_2O_3) in 688 ml of freshly prepared and cooled distilled water by filtering through asbestos and by adding carefully 412 ml of concentrated sulphuric acid.

A-6.2.1.3 *Sodium thiosulphate solution* — 0·1 N.

A-6.2.1.4 *Potassium iodide solution* — 40 percent.

A-6.2.1.5 *Starch indicator* — Triturate 1 g of starch with 10 ml of cold water and pour with constant stirring into 200 ml of boiling water. Allow to settle and use the clear supernatant liquid.

A-6.2.2 *Procedure* — Take 10 ml of the material into 1 000-ml measuring flask and fill up to the mark with distilled water. Measure 20 ml of this solution and add to it about 10 ml of chromic oxide solution contained in a round-bottom flask fitted with ground-glass stopper. Heat the above mixture under reflux on a boiling water-bath for half-an-hour. Cool the contents and transfer quantitatively to a 100-ml round-bottom flask and fill up to the mark with distilled water. Take 10 ml of this solution and mix with 5 ml of potassium iodide solution and titrate with sodium thiosulphate solution using starch as an indicator.

*Specification for sulphuric acid (*revised*).

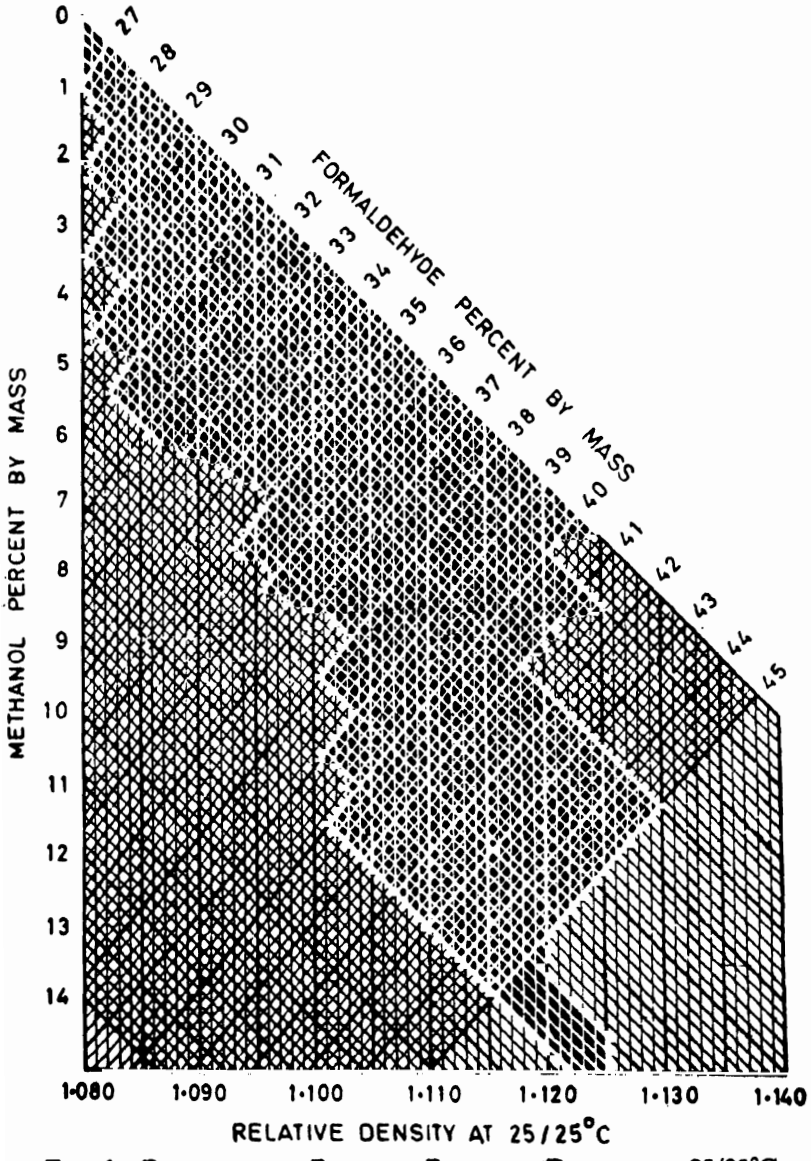

FIG. 1 RELATIONSHIP BETWEEN RELATIVE DENSITY AT 25/25°C AND COMPOSITION OF FORMALDEHYDE SOLUTIONS

A-6.2.3 *Calculation*

$$\text{Methanol, percent by mass} = \left[10 \times f - \left(\frac{v}{2} + \%CH_2O \times 0{\cdot}133\, d \right) \right] \times 5$$

where

f = factor of chromic acid solution,

v = volume in ml of 0·1 N sodium thiosulphate solution used in the titration, and

d = density in g/ml of formaldehyde solution.

A-6.2.3.1 *Determination of factor* — Pipette 10 ml of chromic acid solution into 20 ml of distilled water contained in a 100-ml round-bottom flask fitted with ground-glass stopper and reflux the contents on a boiling water-bath for about 30 minutes. Cool and transfer the contents into a 100-ml volumetric flask and fill up to the mark with distilled water. Mix 10 ml of this solution with 5 ml of potassium iodide solution and titrate against sodium thiosulphate solution using starch as an indicator.

$$\text{Then, factor } (f) = \frac{V}{20}$$

where

V = volume in ml of sodium thiosulphate solution used in the titration.

A-7. TEST FOR HEAVY METALS (OTHER THAN IRON)

A-7.1 Reagents

A-7.1.1 *Hydrochloric Acid* — approximately 1 N.

A-7.1.2 *Hydrogen Sulphide Solution* — saturate a quantity of water with hydrogen sulphide gas. This solution shall be prepared fresh.

A-7.1.3 *Standard Lead Solution* — Weigh to the nearest 0·2 mg, 0·016 g of lead nitrate. Place in a 1 000-ml one-mark volumetric flask and dissolve in water and fill up to the mark. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-7.2 Procedure — Take 20 ± 0·2 g of the material in a Nessler cylinder and add 2·5 ml of hydrochloric acid solution. Dilute the contents to 100 ml with water and saturate with hydrogen sulphide. To another Nessler cylinder containing 80 ml of water, add an agreed volume of standard lead solution followed by 2·5 ml of hydrochloric acid solution and dilute to 100 ml.

A-7.2.1 The material shall be taken to have passed the test if the colour produced with the material is not darker than that produced with the standard lead solution.

A-8. DETERMINATION OF *p*H

A-8.1 Apparatus

A-8.1.1 p*H Meter* — Use a standard laboratory *p*H meter conforming to IS : 2711-1966*.

A-8.2 Procedure — Take about 5 g of the material and dissolve in water. Allow 5 minutes time and measure the *p*H of the solution at 27 ± 1°C in accordance with IS : 4309-1967† using a standard *p*H meter.

APPENDIX
(*Clause* 4.1)

SAMPLING OF FORMALDEHYDE SOLUTION

B-1. GENERAL REQUIREMENTS FOR SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that an ullage of about 5 percent is left after pouring in the sample.

B-1.7 Each sample container shall be sealed air-tight after filling, and marked with the full details of sampling, the date of sampling and details given under **3.2**.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

*Specification for direct reading *p*H meters (*revised*).
†Methods of measurement on direct reading *p*H meters.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM LOTS OF DIFFERENT SIZES

LOT SIZE	SAMPLE SIZE
N	n
(1)	(2)
Up to 25	3
26 to 50	4
51 ,, 100	5
101 ,, 200	6
201 ,, 300	7
301 ,, 500	8
501 ,, 800	9
801 and above	10

B-2.3 In order to ensure the randomness of selection, random sampling procedure given in IS : 4905-1968* may be followed.

B-3. INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

B-3.1 From each of the containers selected according to **B-2.3**, a representative portion of the material, about 300 ml in volume, shall be drawn. These samples shall constitute individual samples.

B-3.2 From each of these individual portions (**B-3.1**), an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 600 ml in volume. The composite sample shall be divided into three equal parts, one for the purchaser, one for the supplier and the third for the referee.

B-3.3 Referee Sample — The referee sample consists of the composite sample marked for this purpose, and shall bear the seals of purchaser and the supplier. It shall be kept at a place agreed to between the two and shall be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of all the characteristics given in Table 1 shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For declaring the conformity of the lot to the requirements of all the characteristics, the test results on the composite sample shall meet the corresponding requirement specified.

*Methods for random sampling.

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(First Revision)

(*Page* 5, *clause* **A-1.1**, *line* 2) — Substitute 'IS 1070 : 1992*' *for* 'IS 1070 : 1960*'.

(*Page* 5, *foot-note with* '*' *mark*) — Substitute '*Reagent grade water (*third revision*)' for the existing matter.

(*Page* 10, *clause* **A-6.2.1.1**) — Substitute 'IS 266 : 1993*' *for* 'IS 266 : 1961*'.

(*Page* 10, *foot-note with* '*' *mark*) — Substitute '(*third revision*)' *for* '(*revised*)' at the end of the text.

(*Page* 10, *clause* **A-6.2.1.2**) — Substitute the following for the existing matter:

'**A-6.2.1.2** *Chromic oxide solution* — Mix 83.3 g of pure oxide of chromium (Cr_2O_3) with 688 ml of freshly prepared, distilled water and add slowly with stirring 412 ml of concentrated sulphuric acid to get a clear homogeneous solution. Filter through a Gooch crucible if necessary.'

(*Page* 13, *clause* **A-8.1.1**) — Substitute 'IS 2711 : 1979*' *for* 'IS 2711 : 1966*'.

(*Page* 13, *clause* **A-8.2**, *line* 3) — Substitute 'IS 4309 : 1979†' *for* 'IS 4309 : 1967†'.

(*Page* 13, *foot-note with* '*' *mark*) — Substitute '(*second revision*)' *for* '(*revised*)' at the end of the text.

(*Page* 13, *foot-note with* '†' *mark*) — Insert '(*first revision*)' at the end of the text.

(PCD 9)

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AMENDMENT NO. 2 AUGUST 2005
TO
IS 3321:1973 SPECIFICATION FOR FORMALDEHYDE SOLUTION

(First Revision)

(*Page* 10, *clause* **A-6.2.1.2**, *Chromic Oxide Solution*) — Insert the following note:

'NOTE — The oxide of chromium may be digested with alkaline hydrogen peroxide before dissolving in acidulated water to get a clear solution.'

(*Page* 10, *clauses* **A-6.2.3** *and* **A-6.2.3.1**, *Calculation*) — Substitute the following for the existing equation:

Methanol, percent by mass = $1.001\ 7 + 0.003\ F - D/0.002\ 53$

where

F = formaldehyde content, and
D = specific gravity.