IS 11673 (1992): Sodium Hypochlorite Solution [CHD 1: Chemical]

IS 11673 : 1992
(Reaffirmed 2003)

भारतीय मानक

सोडियम हाइपोक्लोराइट की विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

SODIUM HYPOCHLORITE SOLUTION — SPECIFICATION

(First Revision)

First Reprint FEBRUARY 1996

UDC 661·833·322·2 : 54 — 145

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April 1992

Price Group 5

Acids, Alkalies and Halides Sectional Committee, CHD 02

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finanlized by the Acids, Alkalies and Halides Sectional Committee had been approved by the Chemical Division Council.

Sodium hypochlorite solution is used in textile and paper bleaching, laundry trade, sterilization of swimming pools, disinfection of drinking water, treatment of cyanide wastes of electroplating industry and treatment of sewage effluent. It is also used as a sanitizer and deodorizer for floors, sinks and toilets, as a therapeutic adjunct in the treatment of certain skin diseases and as a safe antiseptic.

Commercial sodium hypochlorite solution usually contains 10 to 15 percent of available chlorine at ambient temperatures. The stability of hypochlorite solution is greatly affected by heat, light, *p*H and presence of heavy metal cations (which are present in caustic soda). The optimum stability is attained at a *p*H close to 11 and with the heavy metal cations content not more than 5 ppm. Storage temperatures should not exceed 32°C, above which the rate of decomposition becomes too high and the available chlorine content is rapidly depleted. At temperatures above 38°C, sodium chlorate formation becomes appreciable resulting in loss of sodium hypochlorite.

This standard was originally published in 1986. In this revision, the formulae for various requirements have been suitably corrected. The method of test for determination of sodium chlorate has been modified.

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 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SODIUM HYPOCHLORITE SOLUTION — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium hypochlorite solution.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1987	Hydrochloric acid (*third revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
2124 : 1974	Sodium bicarbonate (*first revision*)
4905 : 1968	Methods for random sampling.

3 TERMINOLOGY

3.1 Available Chlorine

This is the measure of the oxidizing power of the chlorine present as hypochlorite. It is expressed in terms of chlorine with a gram equivalent mass of 35·46.

4 GRADES

The material shall be of the following two grades:

Grade 1 — For household use, and

Grade 2 — For industrial use.

5 REQUIREMENTS

5.1 Description

The hypochlorite solution shall be a pale, yellowish green, clear liquid and shall not produce any sludge or turbidity when mixed with water.

5.2 Keeping Quality

The material for both the grades shall comply with the minimum available chlorine content for not less than 30 days from the date of packing. After the period of more than 30 days, the minimum available chlorine for both grades shall be as agreed to between the purchaser and the supplier.

NOTE — As a guide the following table provides a general guidance on the stability of the material.

Available Chlorine Trade, Percent	*Chlorine g/l*	*Half Life Days, 25°C*
3	30	1 700
6	60	700
9	90	250
12	120	180
15	150	100
18	180	60

5.3 The material, when tested according to the methods prescribed in Annex A, shall comply with the requirements given in Table 1. Reference to the relevant clauses of Annex A is given in col 5 of Table 1.

6 PACKING, MARKING AND STORING

6.1 Packing

The material shall be packed in air-tight plastic containers or as agreed to between the purchaser and the supplier. The containers used shall be dry and free from grease, dirt or other foreign matter likely to cause decomposition of the material.

6.2 Marking

Each package shall bear legibly and indelibly the following information:

a) Name and grade of the material;
b) Indication of the source of manufacture;
c) Gross and net mass;
d) Date of packing; and
e) Lot number.

6.3 Storing

The material shall be stored in a cool and dark place. While shipping, the material shall be stored away from boilers or any other source emanating heat and light.

7 SAMPLING AND CRITERIA FOR CONFORMITY

The method of drawing representative samples of the material and their criteria for conformity to the requirements of this standard shall be as prescribed in Annex B.

Table 1 Requirements for Sodium Hypochlorite Solution

(*Clause* 5.3)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Annex A)
		Grade 1	*Grade* 2	
(1)	(2)	(3)	(4)	(5)
i)	Relative density (at 25°/25° C)	1·07 to 1·18	1·20 *Min*	A-2
ii)	Available chlorine (as Cl), percent by mass by volume	4·0 to 6·0	12·5 to 15·0	A-3
iii)	Total chlorine, percent by volume (as Cl)	4·0 to 6·0	12·5 to 15·0	A-4
iv)	Free alkali (as NaOH), g/l, *Min*	1·0	5·0	A-5
v)	Free sodium carbonate (as Na_2CO_3), g/l, *Max*	0·5	0·5	A-6
vi)	Iron (as Fe), ppm, *Max*	0·4	1·0	A-7
vii)	Sodium chlorate	Traces	Traces	A-8

ANNEX A

(*Clause* 5.3)

METHODS OF TEST FOR SODIUM HYPOCHLORITE SOLUTION

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

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

A-2 DETERMINATION OF RELATIVE DENSITY

A-2.0 General

Relative density shall be determined by a capillary-stoppered relative density bottle, but subject to agreement between the purchaser and the supplier, a Twaddell or Baume hydrometer may also be used.

A-2.1 Procedure

Clean the bottle with a saturated solution of chromic acid in concentrated sulphuric acid followed by washing with alcohol. Dry the bottle, cool it to room temperature and weigh. Fill the weighed bottle with water and place it in the constant temperature bath maintained at 25·0 ± 0·5°C for an hour. Then insert the capillary stopper which has also been brought to 25·0 ± 0·5°C. Wipe excess liquid from the top of the stopper, remove the bottle from the bath, wipe the outside to dryness, bring to room temperature and weigh. The difference between the mass of the filled and empty bottle gives the water equivalent, that is, the mass in air of the water contained in the bottle at 25·0 ± 0·5 °C. Empty the bottle, rinse several times with alcohol and finally with ether. Remove the ether fumes with the aid of an air blast and permit the bottle to dry thoroughly. Fill the bottle with the material, warming both the material and bottle to assist filling and removal of air bubbles, and then bring the bottle and contents to 25·0 ± 0·5 °C. When the temperature is constant, insert the capillary stopper which has also been brought to 25·0 ± 0·5 °C and remove excess liquid from the top of the stopper. Remove the bottle from the bath, wipe the outside dry, bring to room temperature and weigh.

A-2.2 Calculation

$$\text{Relative density (at 25°/25°C)} = \frac{M_3 - M_1}{M_2 - M_1}$$

where

M_1 = mass in g of the empty relative density bottle;

M_2 = mass in g of water at 25°C and the relative density bottle; and

M_3 = mass in g of sodium hypochlorite solution at 25°C and the relative density bottle.

A-3 AVAILABLE CHLORINE

A-3.0 General

The sample is added to an acidified solution of potassium iodide and the released iodine is titrated with standard sodium thiosulphate solution to the usual starch end point.

A-3.1 Reagents

A-3.1.1 *Glacial Acetic Acid*

A-3.1.2 *Standard Potassium Iodate Solution* — 0·1 N.

A-3.1.3 *Starch Indicator Solution* — 0·5 percent.

Mix 0·5 g of soluble starch with 5 ml of cold water and add 95 ml of boiling water. Mix, cool and store in a glass bottle. Replace frequently or add 0·1 percent salicylic acid to the starch solution to minimize deterioration.

A-3.1.4 *Potassium Iodide* — iodate-free.

A-3.1.5 *Standard Sodium Thiosulphate Solution (Hypo)* — 0·1 N.

Dissolve 25 g of sodium thiosulphate ($Na_2S_2O_3 \cdot 5H_2O$) crystals in freshly boiled and cooled water, and dilute to 1 000 ml.

NOTE — The solution is more stable if the glassware is cleaned with sulphuric or chromic acids and thoroughly rinsed with water.

A-3.1.5.1 *Standardization of sodium thiosulphate solution*

Weigh accurately 3·567 g of dry potassium iodate (KIO_3) and transfer to a 1 000 ml volumetric flask. Dissolve in water, mix thoroughly and make up to the mark. This solution will be exactly 0·1 N. To standardize the sodium thiosulphate solution, carefully pipette out a 50 ml aliquot of the potassium iodate solution into a 250-ml conical flask and dilute to 100 ml with water. Add 1 g of potassium iodide crystals. When it is dissolved, add 15 ml of 0·1 N hydrochloric acid and titrate immediately with the sodium thiosulphate solution. When the solution becomes light yellow, add 1 ml of starch indicator solution and complete the titration to the disappearance of the blue colour. Standardize at least monthly. Calculate the normality of the sodium thiosulphate solution as follows:

$$\text{Normality} = \frac{(50 \times 0{\cdot}1)}{A}$$

where *A* is the volume, in ml, of standard sodium thiosulphate solution required for titration.

A-3.2 Procedure

A-3.2.1 Dissolve 2 to 3 g of potassium iodide crystals in 50 ml of water in a 250-ml conical flask. Add 10 ml of acetic acid, then pipette out the aliquot of sample into the solution, keeping the tip of the pipette beneath the surface of the solution until drained. Titrate at once with 0·1 N standard sodium thiosulphate solution until the iodine colour is nearly gone, then add 1 ml of starch indicator solution and complete the titration to the disappearance of the blue colour.

A-3.3 Calculation

$$\text{Available chlorine (as Cl), percent mass by volume} = \frac{(AN \times 0{\cdot}035\,46) \times 100}{V}$$

A-3.3.1 Sodium hypochlorite (as NaOCl), percent mass by volume

$$= \frac{(AN \times 0{\cdot}037\,22) \times 100}{V}$$

where

A = volume in ml of standard sodium thiosulphate solution required for titration of the sample;

N = normality of the standard sodium thiosulphate solution; and

V = volume in ml of original sample in aliquot used.

A-4 TOTAL CHLORINE

A-4.0 General

All hypochlorites and chlorates present are reduced to chloride by sodium metabisulphite in the presence of nitric acid. The total chlorine is then determined by Volhard method.

A-4.1 Reagents

A-4.1.1 *Iron Indicator Solution*

Dissolve 6·25 g of ferric ammonium sulphate [$Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$] in 50 ml of water and add 45 ml of nitric acid.

A-4.1.2 *Concentrated Nitric Acid — See* IS 264 : 1976.

A-4.1.3 *Standard Sodium Chloride Solution* — 0·05 N.

Dissolve 2·923 g of pure sodium chloride (NaCl) in water and dilute to 1 000 ml.

A-4.1.4 *Standard Potassium Thiocyanate Solution* — 0·05 N.

Prepare a 0·05 N solution of potassium thiocyanate (KCNS) and standardize against 0·05 N standard silver nitrate (**A-4.1.5**).

A-4.1.5 *Standard Silver Nitrate Solution* — 0·05 N.

Dissolve 8·5 g of silver nitrate ($AgNO_3$) in water and dilute to 1 000 ml. Store in an amber-coloured glass-stoppered bottle. Standardize the solution as follows:

'Pipette out 25 ml of the standard 0·05 N sodium chloride solution (*see* **A-4.1.3**) into a 250-ml conical flask resting on a white porcelain tile, and add 1 ml of potassium chromate indicator (*see* **A-4.1.7**). Add the silver nitrate solution slowly from a burette, swirling the liquid constantly until the red colour formed by the addition of each drop begins to disappear slowly; this is an indication that most of the chloride has been precipitated. Continue the addition dropwise until a faint but distinct reddish-brown colour persists after vigorous shaking.

Determine the indicator blank correction by adding 1 ml of the indicator to a volume of water equal to the final volume in the titration and then silver nitrate solution until the colour of the blank matches to that of the solution titrated. The indicator blank correction should not amount to more than 0·03 to 0·1 ml of silver nitrate solution. Repeat the titration with two futther 25-ml portions of the sodium chloride solution. The titration readings should agree within 0·1 ml.

A-4.1.6 *Sodium Metabisulphite* ($Na_2S_2O_5$) — Powder.

A-4.1.7 *Potassium Chromate* — 5 percent (*m/v*).

A-4.2 Procedure

To a 250-ml beaker add 50 ml of water and about 0·5 g of sodium metabisulphite powder. Then pipette into the mixture a sample aliquot of the same size as used for available chlorine and chlorate. Add about 10 drops of nitric acid to acidify the solution and boil until all the sulphur dioxide has been expelled. Cool to room temperature and add 5 ml of iron indicator solution. From a burette add 0·5 ml of 0·05 N potassium thiocyanate solution (*see* Note 1). Then titrate to complete decolourization with 0·05 N silver nitrate solution. Filter off the precipitate by suction and wash three times with water (*see* Note 2). Finally, back titrate the filtrate and washings with 0·05 N potassium thiocyanate solution until a faint reddish colour persists.

NOTES

1 The small amount of potassium thiocyanate solution serves as an indicator to show when an excess of standard silver nitrate solution has been added. The back titration is continued from the same burette and the total volume of potassium thiocyanate solution used is noted and used in the calculation.

2 For less accurate work the filtration may be avoided by adding 1 ml of nitrobenzene to coagulate the suspension before back-titrating the excess silver nitrate solution.

A-4.3 Calculation

$$\text{Total chlorine (as Cl), percent by volume} = \frac{(CN_1 - DN_2) \times 3{\cdot}546}{V}$$

where

C = volume in ml of standard silver nitrate solution required for titration of the sample;

D = volume in ml of standard potassium thiocyanate solution required for back-titration;

N_1 = normality of standard silver nitrate solution;

N_2 = normality of standard potassium thiocyanate solution; and

V = volume in ml of original sample in aliquot used.

A-5 FREE ALKALI

A-5.0 General

The sample is added to a neutralized, mixed solution of barium chloride and hydrogen peroxide, which precipitates any carbonate and reduces the hypochlorite to chloride. The free alkali is then titrated with standard hydrochloric acid using phenolphthalein indicator.

A-5.1 Reagents

A-5.1.1 *Barium Chloride Solution* — 10 percent (*m/v*).

Dissolve 100 g of barium chloride ($BaCl_2{\cdot}2H_2O$) in water and dilute to 1 000 ml. Filter, if the solution is turbid.

A-5.1.2 *Standard Hydrochloric Acid* — 0·1 N.

A-5.1.3 *Hydrogen Peroxide Solution* — 3 percent (*v/v*).

A-5.1.4 *Phenolphthalein Indicator Solution* — 0·5 percent (*m/v*).

Dissolve 0·5 g of phenolphthalein in 60 ml of 95 percent ethyl alcohol and dilute to 100 ml with water.

A-5.1.5 *Sodium Hydroxide Solution* — 0·4 percent (*m/v*).

A-5.2 Procedure

Place 50 ml of barium chloride solution and 30 ml of hydrogen peroxide solution in a 250-ml conical flask, add 10 drops of phenolphthalein indicator solution and neutralize with caustic soda solution. Introduce into this neutral mixture 10 ml of the liquid bleach, shake or stir vigorously for 1 minute, and titrate caustic soda solution with 0·1 N hydrochloric acid until the pink colour disappears.

A-5.3 Calculation

$$\text{From alkali (NaOH), g/l} = \frac{(V_1 N \times 40)}{V}$$

where

V_1 = volume in ml of standard hydrochloric acid solution required for titration of sample;

N = normality of standard hydrochloric solution acid; and

V = volume in ml of original sample solution in aliquot used.

A-6 FREE SODIUM CARBONATE

A-6.0 General

For determining free sodium carbonate, a number of determinations like total alkalinity, free sodium hydroxide and sodium bicarbonate are required. Finally, free carbonate is estimated from the data obtained.

A-6.1 Total Alkalinity (as Sodium Monoxide)

A-6.1.1 *Reagents*

A-6.1.1.1 *Standard hydrochloric acid* — 0·1 N.

A-6.1.1.2 *Dilute hydrogen peroxide solution* — 10 percent.

A-6.1.1.3 *Standard sodium hydroxide solution* — 0·1 N.

A-6.1.1.4 *Mixed indicator*

Mixture of 3 parts of 2 percent of methyl red in alcohol and 1 part of 0·1 percent of bromocresol green in alcohol.

A-6.1.2 *Procedure*

Use a volume of sample solution to require at least 10 ml of 0·1 N standard hydrochloric acid for titration. Add the sample to three times its volume of hydrogen peroxide solution, previously neutralized with 0·1 N sodium hydroxide solution, using methyl red-bromocresol mixed indicator solution. Add a few drops more of the indicator solution and titrate to the end point with 0·1 N hydrochloric acid.

A-6.1.3 *Calculation*

$$\text{Total alkalinity (as sodium monoxide), g/l} = \frac{V_1 \times N \times 31}{V}$$

where

V_1 = volume in ml of standard hydrochloric acid;

N = normality of hydrochloric acid; and

V = volume in ml of original sample solution in aliquot used.

A-6.2 Free Sodium Hydroxide

A-6.2.1 *Reagents*

A-6.2.1.1 *Dilute barium chloride solution* — 10 percent (*m/v*).

A-6.2.2 *Apparatus*

A-6.2.2.1 p*H meter*

A-6.2.3 *Procedure*

Place 50 ml of barium chloride solution and 30 ml of hydrogen peroxide solution in a 250-ml beaker and, using a *p*H meter, bring the *p*H to 7·5 with 0·1 N sodium hydroxide solution. Introduce into this solution 10 ml of the sample solution, stir vigorously for 1 min, and titrate with 0·1 N hydrochloric acid to a *p*H of 7·5 while continuing to stir the solution.

A-6.2.4 *Calculation*

$$\text{Sodium hydroxide (as NaOH), g/l} = \frac{V_1 \times N \times 40}{V}$$

where

V_1 = volume in ml of standard hydrochloric acid;

N = normality of hydrochloric acid; and

V = volume in ml of original sample solution in aliquot used.

A-6.3 Sodium Bicarbonate

If no free sodium hydroxide is found, sodium bicarbonate may be present and can be determined as follows.

A-6.3.1 Place in a 250-ml conical flask a volume of 0·1 N sodium hydroxide solution equal to the volume of 0·1 N hydrochloric acid required for the determination of total alkalinity. Into this solution, add by pipette the same volume of sample solution as used for the determination of total alkalinity. In a 250-ml beaker, place 50 ml of barium chloride solution, 30 ml of hydrogen peroxide, and 10 drops of phenolphthalein indicator solution, neutralize with 0·1 N sodium hydroxide solution. Add this neutralized solution to the prepared sample solution and shake vigorously for 1 min. Titrate the excess sodium hydroxide with 0·1 N hydrochloric acid to the disappearance of the pink colour.

A-6.3.2 *Calculation*

$$\text{Sodium bicarbonate (as } NaHCO_3 \text{) g/l} = \frac{(V_1N_1 - V_2N_2) \times 84}{V}$$

where

V_1 = volume in ml of sodium hydroxide solution;

N_1 = normality of sodium hydroxide solution;

V_2 = volume in ml of hydrochloric acid;

N_2 = normality of hydrochloric acid; and

V = volume in ml of original sample solution in aliquot used.

A-6.4 Free Sodium Carbonate

A-6.4.1 If sodium hydroxide is present, calculate free sodium carbonate as follows:

Free sodium carbonate (as Na_2CO_3), g/l = [Total alkalinity — 0·775 (sodium hydroxide)] × 1·709

A-6.4.2 If sodium bicarbonate is present, calculate free sodium carbonate as follows:

Free sodium carbonate (as Na_2CO_3), g/l = [Total alkalinity — 0·369 (sodium bicarbonate)] × 1·709.

A-7 IRON

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50-ml capacity.

A-7.2 Reagents

A-7.2.1 *Ammonium Persulphate*

A-7.2.2 *Butanolic Potassium Thiocyanate Solution*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously till the solution is clear.

A-7.2.3 *Standard Iron Solution A*

Dissolve 0·702 2 g of ferrous ammonium sulphate [$FeSO_4·(NH_4)_2SO_4·6H_2O$] in 100 ml of water, add 5 ml of 1 : 5 (*v*/*v*) sulphuric acid and run in cautiously a dilute solution of potassium permanganate (0·2 percent, *m*/*v*) until a slight pink coloration remains after stirring well. Dilute with water to 1 000 ml and mix thoroughly. One millilitre of this solution contains 0·1 mg of iron as Fe.

A-7.2.4 *Standard Iron Solution B*

Take 100 ml of the standard iron Solution A (*see* **A-7.2.3**) and dilute to 1 000 ml with water in a 1 000-ml volumetric flask. This dilute solution should be prepared fresh. One millilitre of this solution contains 0·01 mg of iron (as Fe).

A-7.3 Procedure

A-7.3.1 *For Grade 1*

Weigh 50·0 g of the material and evaporate it almost to dryness. Dilute it to 30 ml, add about 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for about 30 seconds and allow the layers to separate. Carry out a control test in another Nessler cylinder using 2 ml of standard iron Solution B (*see* **A-7.2.4**). Compare the intensity of the colour produced in the butanol layers in the two cylinders.

A-7.3.2 *For Grade 2*

Carry out the test as given for Grade 1 in **A-7.3.1**, using 5 ml of the standard iron Solution B (*see* **A-7.2.4**) for the control test.

A-7.3.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-8 SODIUM CHLORATE

A-8.0 General

Sodium chlorate is reduced with sodium bromide in 8 N hydrochloric acid. After dilution and addition of potassium iodide, the released iodine (equivalent to the hypochlorate plus chlorate) is titrated with standard sodium thiosulphate solution and starch indicator.

A-8.1 Apparatus

The apparatus (*see* Fig. 1) consists of 1 000-ml wide-mouthed reaction bottle (*A*), fitted with a double hole rubber stopper carrying a separating funnel *B*, conveniently graduated or marked at the 10, 20 and 100 ml levels, and a delivery tube leading to a 50-ml test tube gas trap *C*, which is fitted with rubber tubing and a glass mouthpiece, *D*.

A-8.2 Reagents

A-8.2.1 *Concentrated Hydrochloric Acid* — *See* IS 265 : 1987.

A-8.2.2 *Sodium Bromide Solution* — 10 percent (*m*/*v*).

A-8.2.3 *Potassium Iodide Solution* — 10 percent (*m*/*v*).

A-8.2.4 *Standard Sodium Thiosulphate Solution* — 0·05 N.

A-8.2.5 *Starch Indicator Solution* — 0·5 percent (*m*/*v*).

A-8.2.6 *Sodium Bicarbonate* — pure grade (*see* IS 2124 : 1974).

A-8.3 Procedure

Pipette out an aliquot of the sample (same amount as used for available chlorine determination) into the reaction bottle (*A*), add 1 ml of concentrated hydrochlotic acid and 0·3 g of pure sodium bicarbonate to expel all the air from the vessel through the long test tube (*C*), containing saturated solution of sodium bicarbonate. Then add 20 ml of sodium bromide solution followed by 80 ml of concentrated hydrochloric acid. Stopper the bottle and shake well. Allow to stand for 10 minutes. Add 20 ml of 10 percent potassium iodide solution through the separating funnel carefully and titrate the liberated iodine against 0·05 N sodium thiosulphate solution using a few drops of starch indicator solution.

Run a blank with all the reagent except the sample by proceeding in the same manner as that of the test.

A-8.4 Calculation

$$\text{Sodium chlorate (as } NaClO_3\text{), g/l} = \frac{(V_2 - V_1) \times N \times 17 \cdot 75}{V}$$

where

V_2 = volume in ml of sodium thiosulphate, solution used for the test;

V_1 = volume in ml of sodium thiosulphate solution used for the blank;

N = normality of sodium thiosulphate solution; and

V = volume in ml of original sample solution in aliquot used.

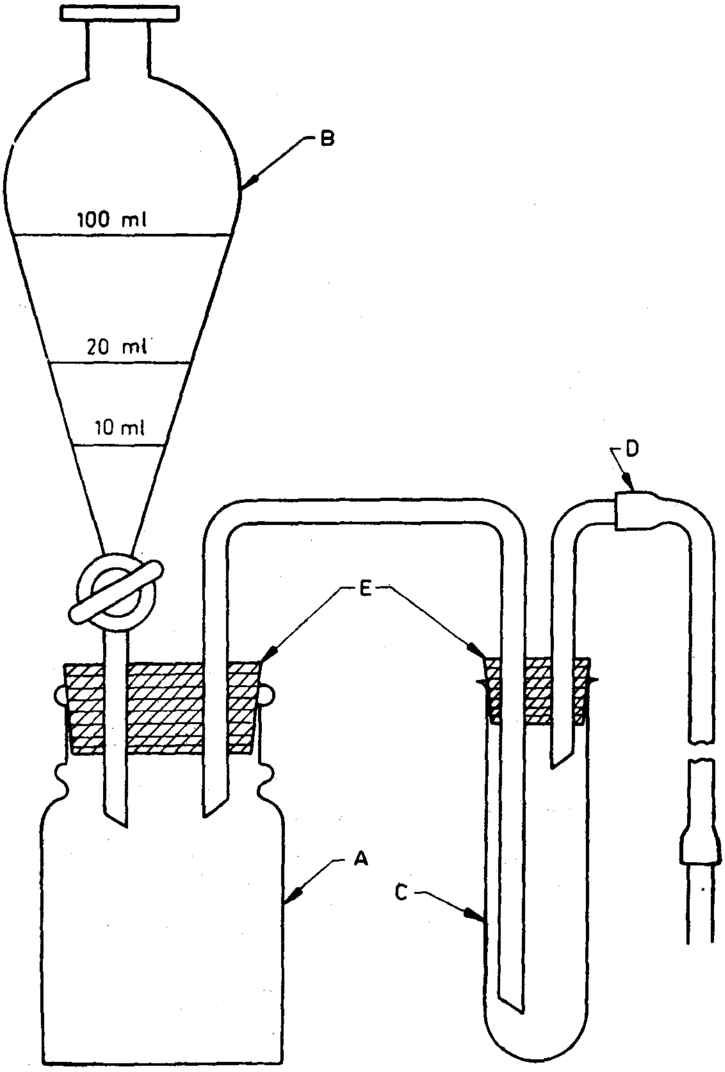


FIG. 1 APPARATUS FOR DETERMINATION OF SODIUM CHLORATE

ANNEX B

(*Clause* 7)

SAMPLING OF SODIUM HYPOCHLORITE SOLUTION

B-1 GENERAL REQUIREMENTS

B-1.1 Samples shall not be taken out in an exposed place.

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

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 Precautions shall be taken to protect the samples, material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

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

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling and the month and year of manufacture of the material.

B-1.8 Samples shall be stored in a cool and dry place.

B-2 SAMPLING INSTRUMENT

B-2.1 Sampling Tube

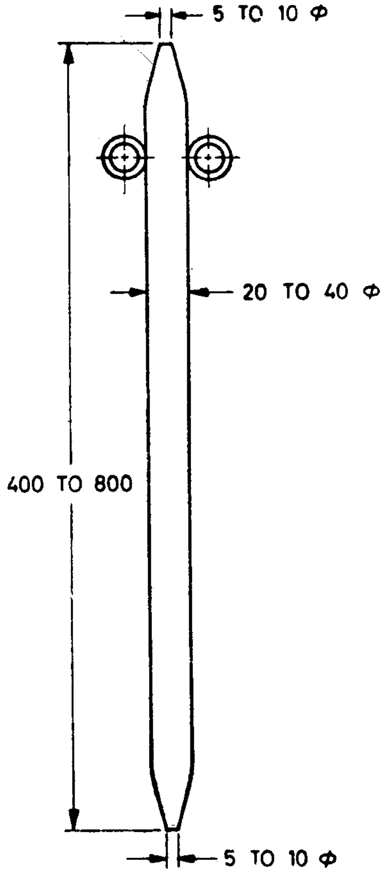
It is made of thick glass and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

B-2.1.1 For small containers, the size of the sampling tube may be altered suitably.

B-3 SCALE OF SAMPLING

B-3.1 Lot

In any consignment, all the containers of the same size belonging to the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture of different sizes of containers, the containers belonging to the same batch and same size shall be grouped together and each such group shall constitute a separate lot.



All dimensions in millimetres.

FIG. 2 SAMPLING TUBE

B-3.2 For ascertaining the conformity of the material in a lot to the requirement of this specification, test shall be carried out for each lot separately. For this purpose the number of containers to be selected from a lot shall be in accordace with Table 2.

Table 2 Scale of Sampling

(*Clause* B-3.2)

Lot Size (*N*)	No. of Containers to be Selected (*n*)
Up to 15	2
16 to 50	3
51 ,, 100	4
101 ,, 300	5
301 and above	6

B-3.2.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, random sampling procedure as given in IS 4905 : 1968 shall be followed.

B-4 PREPARATION OF SAMPLE

B-4.1 Draw with an appropriate sampling instrument (*see* B-2) equal portion of material from each container selected in **B-3.2** and mix them thoroughly together to constitute a single composite sample. Divide this composite sample into three parts, each sufficient for carrying out the intended tests and transfer them to thoroughly cleaned and dry sample containers. Send each one of these to the purchaser and the supplier. Reserve the third composite sample as referee sample. The referee sample shall be kept at a place and under conditions agreed to between the purchaser and the supplier. The referee sample shall be used in case of a dispute.

B-5 CRITERIA FOR CONFORMITY

B-5.1 The material in any lot shall be accepted as conforming to the requirements of this specification if the composite sample meets the requirements given in Table 1 when tested as per the procedure laid down in Annex A.

Bureau of Indian Standards

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This Indian Standard has been developed from **Doc : No** CHD 02 (0094)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Printograph, New Delhi-5 (INDIA

AMENDMENT NO. 1 JULY 1996
TO
IS 11673 : 1992 SODIUM HYPOCHLORITE SOLUTION — SPECIFICATION

(*First Revision*)

[*Page* 2, *Table* 1, *Sl No.* (v), *col* 2] — Substitute '*Min*' *for* '*Max*'.

(*Page* 6, *clause* A-8, *line* 4) — Substitute 'hypochlorite' *for* 'hypochlorate'.

(CHD 02)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JANUARY 2000
TO
IS 11673 : 1992 SODIUM HYPOCHLORITE SOLUTION — SPECIFICATION

(*First Revision*)

[*Page* 1, *clause* **6.2**(e)] — Insert (f) after (e):

'f) Available chlorine (to be declared by the manufacturer).'

[*Page* 2, *Table* 1 , *Sl No.* (iv), *col* 3] — Substitute '1.0' *for* '1 0'.

[*Page* 2, *Table* 1, *Sl No.* (vii), *col* 2]] — Substitute 'Sodium chlorate, percent by mass, Max' *for* 'Sodium chlorate'.

[*Page* 2, *Table* 1, *Sl No.* (vii), *col* 3] — Substitute '0.05' *for* 'traces'.

[*Page* 2, *Table* 1, *Sl No.* (vii), *col* 4] — Substitute '0.3' *for* 'Traces'.

(CHD 2)

.Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 MARCH 2002
TO
IS 11673 : 1992 SODIUM HYPOCHLORITE SOLUTION — SPECIFICATION

(First Revision)

(*Foreword, para* 4) — Insert the following para after para 4:

'Considerable assistance has been derived from ASTM D 2022:1989 Standard test methods of sampling and chemical analysis of chlorine containing bleaches.'

(*Page* 1, *clause* **2**) — Substitute the following for the existing:

NORMATIVE REFERENCES

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

IS No.	*Title*
264 : 1976	Nitric acid *(second revision)*
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Water for general laboratory use (*third revision*)
4905 : 1968	Methods for random sampling

(*Page* 2, *clause* **A-1.1,** *line* 2) — Substitute 'IS 1070:1992' *for* 'IS 1070 : 1977'.

(*Page* 3, *clause* **A-3.2**) — Insert the following new clause and renumber '**A-3.2.1**' as '**A-3.2.2**':

'**A-3.2.1** *Preparation of Sample Solution* — Strong solutions of bleach shall be accurately diluted and aliquots taken for determination of available chlorine (**A-3**), total chlorine (**A-4**), free sodium carbonate (**A-6**) and sodium chlorate (**A-8**). The size of aliquots shall be such that approximately 40 ml of the 0.1 N reagent is required.'

(*Page* 6, *clause* **A-8**) — Substitute the following for the existing:

A-8 SODIUM CHLORATE

A-8.0 General

Sodium chlorate is reduced with sodium bromide in 8 N hydrochloric acid. After dilution and addition of potassium iodide, the released iodine (equivalent to the hypochlorite plus chlorate) is titrated with standard sodium thiosulphate solution and starch indicator.

A-8.1 Apparatus

The apparatus (*see* Fig. 1) consists of 1 000-ml wide-mouthed reaction bottle (*A*), fitted with a double hole rubber stopper carrying a separating funnel *B*, conveniently graduated or marked at the 10, 20 and 100 ml levels, and a delivery tube leading to a 50 ml test tube gas trap *C*, which is fitted with rubber tubing and a glass mouth piece, *D*.

A-8.2 Reagents

A-8.2.1 *Concentrated Hydrochloric Acid* — *See* IS 265 : 1993.

A-8.2.2 *Sodium Bromide Solution* — 10 percent (*m/v*).

A-8.2.3 *Potassium Iodide Solution* — 10 percent (*m/v*).

Prepare a 10 percent solution of potassium iodide (KI). Decolourize with $Na_2S_2O_3$, when necessary.

A-8.2.4 *Standard Sodium Thiosulphate Solution* — 0.1 N (*see* **A-3.1.5**).

A-8.2.5 *Starch Indicator Solution* — 0.5 percent (*m/v*).

A-8.3 Procedure

Pipette an aliquot of the sample (same amount as used for available chlorine determination (*see* **A-3.2.2**) into the reaction vessel. Assemble the apparatus and put 25 ml of potassium iodide solution in the gas trap. Close the funnel stopcock. Pour 20 ml of sodium bromide solution into the funnel. Open the stopcock, and with gentle suction on the mouthpiece, draw the sodium bromide solution into the sample. Close the stopcock and pour 100 ml of hydrochloric acid into the funnel. Open the stopcock and allow the acid to drain into the sample. Draw in the last drops with the suction. Close the stopcock. Swirl the vessel to mix the acid, and let stand exactly for 5 minutes (use time clock). There will be a tendency for a vacuum to form and draw potassium iodide solution from the trap back into the sample. This must be avoided by filling the funnel with water and relieving the vacuum by opening the stopcock and adding a small amount of water.

After 5 minutes open the stopcock and allow the water to drain into the sample swirling to dilute the acid. Add water through the funnel sufficient to dilute the sample to about 700 ml. Close the stopcock, and add 10 ml of potassium iodide solution to the funnel. Apply pressure at the mouthpiece to blow the contents of the trap back into the vessel, opening the stopcock to allow the necessary amount of gas to escape through the funnel. Rinse the trap twice with water each time blowing the contents into the vessel as above. Finally allow the contents of the funnel to drain into the vessel. Rinse down the funnel and stopper and thoroughly mix the contents of the vessel. Titrate at once with 0.1 N sodium thiosulphate solution. When the colour of the solution becomes pale yellow add 5 ml of starch indicator solution and complete the titration to the disappearance of the blue colour.

A-8.4 Calculation

$$\text{Sodium chlorate (NaClO}_3\text{), g/l} = \frac{[(B-A)N \times 17.74]}{V}$$

where

A = volume in ml of sodium thiosulphate solution required for titration for available chlorine (*see* **A-3.3.1**);

B = volume in ml of sodium thiosulphate solution required for titration for sodium chlorate;

N = normality of the sodium thiosulphate solution; and

V = original sample in aliquot used.

(CHD 2)

AMENDMENT NO 4 MAY 2013
TO
IS 11673 : 1992 SODIUM HYPOCHLORITE SOLUTION — SPECIFICATION

(First Revision)

(*Page* 1, *clause* **6.2**) — Insert the following new clauses after **6.2**:

'**6.2.1** *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

6.2.1.1 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.'

(CHD 1)

Reprography Unit, BIS, New Delhi, India