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मानक

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“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 252 (1991): Caustic soda, pure and technical [CHD 1: Inorganic Chemicals]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 252 - 1991

(Reaffirmed 2010)

भारतीय मानक

कास्टिक सोडा, शुद्ध और तकनीकी — विशिष्ट

(तीसरा पुनरीक्षण)

Indian Standard

CAUSTIC SODA, PURE AND TECHNICAL —
SPECIFICATION

(*Third Revision*)

Third Reprint MAY 2007
(Including Amendment 1)

UDC 661.322.1

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BUREAU OF INDIAN STANDARDS
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FOREWORD

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

The specifications for caustic soda, technical (IS 252) and caustic soda, pure (IS 1021) were first issued in 1950 and 1956, respectively. These specifications were later revised in 1962 and 1964, respectively modifying the limits for iron, chlorides, sulphates, silicates and manganese. During the second revision in 1973, IS 1021 was amalgamated with IS 252 and requirements for caustic soda lye and caustic soda solid were also included. The method of sampling and tests were also modified during the course of the second revision.

In the present revision, requirements for chlorates, perchlorates and matter insoluble in water along with the relevant test methods have been incorporated for pure grade of caustic soda. Methods of test for determination of silica, carbonates, sodium hydroxide, chlorides, iron and copper have been suitably 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.

**AMENDMENT NO. 1 FEBRUARY 1995
TO
IS 252 : 1991 CAUSTIC SODA, PURE AND TECHNICAL
— SPECIFICATION**

(Third Revision)

[Page 2, Table 1, Sl No. (x). col 5 and 6] — Substitute '0.2' for '0.02'.

(CHD 002)

Indian Standard

CAUSTIC SODA, PURE AND TECHNICAL — SPECIFICATION

(Third Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for caustic soda, pure and technical.

1.1.1 It covers the material in the solid and lye form.

2 REFERENCES

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

<i>IS No.</i>	<i>Title</i>
265 : 1987	Hydrochloric acid (<i>third revision</i>)
296 : 1986	Sodium carbonate, anhydrous (<i>third revision</i>)
323 : 1959	Rectified spirit (<i>revised</i>)
1070 : 1977	Water for general laboratory use (<i>second revision</i>)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods (<i>first revision</i>)
4264 : 1967	Code of safety for caustic soda

3 GRADES

There shall be two grades of the material, namely:

- a) Pure — suitable for use in rayon and cosmetic industries, and
- b) Technical — suitable for use in textile, soap, vegetable oil refining paper and other industries.

4 REQUIREMENTS**4.1 Form and Description**

The material shall be supplied in the form of lye or solid (including flakes, blocks, sticks and pellets).

4.1.1 The material shall be free from foreign matter, dirt or other visible impurities.

4.2 Relative Density of Caustic Soda Lye

The relative density of caustic soda lye shall be subject to an agreement between the purchaser and the supplier.

4.3 The material shall comply on dry basis with the requirements given in Table 1, when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 7 of the table.

4.4 For Technical Grade

Chlorides (as NaCl) and sulphates (as Na₂SO₄ together shall be 3.5 percent. The value for sulphate content shall also be reported.

4.5 Calculation of Results on Dry Basis

In **A-3** to **A-12**, results have been calculated on the basis of the material as received. To calculate the results on the dry basis as specified in Table 1, the method given in **A-13** shall be adopted.

5 PRECAUTION IN HANDLING AND STORING

The precautions in handling and storing as given in IS 4264 : 1967 shall be observed.

6 PACKING AND MARKING**6.1 Packing**

The material shall be packed in polyethylene bags or polyethylene lined gunny bags or steel drums, or as agreed to between the purchaser and the supplier. The solution shall be supplied in tankers or tank cars.

6.2 Marking

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

- a) Name and grade of the material;
- b) Indication of the source of manufacture;
- c) Net mass of the material; and
- d) Lot or batch number, in code or otherwise.

Table 1 Requirements for Caustic Soda, Pure and Technical
(Clause 4.3)

SI No.	Characteristic	Requirement on Dry Basis				Method of Test Ref To Cl. No. In Annex A
		Pure		Technical		
		Lye 3	Solid 4	Lye 5	Solid 6	
1	2					7
i)	Sodium carbonate (as Na ₂ CO ₃), percent by mass, <i>Max</i>	0.40	0.40	2.00	2.00	A-3
ii)	Sodium hydroxide (as NaOH), percent by mass, <i>Min</i>	99.50	99.50	95.00	95.00	A-4
iii)	Chlorides (as NaCl), percent by mass, <i>Max</i>	0.10	0.10			A-5
iv)	Sulphates (as Na ₂ SO ₄), percent by mass, <i>Max</i>	0.10	0.10	3.50	3.50	A-6
v)	Silicates (as SiO ₂), percent by mass, <i>Max</i>	0.02	0.02	—	—	A-7
vi)	Iron (as Fe), ppm, <i>Max</i>	20	20	350	350	A-8
vii)	Copper (as Cu), ppm, <i>Max</i>	2	2	—	—	A-9
viii)	Manganese (as Mn), ppm, <i>Max</i>	1	1	—	—	A-10
ix)	Chlorates and per-chlorates (as NaClO ₃), ppm, <i>Max</i>	10	10	—	—	A-11
x)	Matter insoluble in water, percent by mass, <i>Max</i>	0.05	0.05	0.02	0.02	A-12

6.2.1 The containers shall be marked with the following caution note:

'CORROSIVE - HANDLE WITH CARE'

and appropriate label to indicate the possible corrosion hazards [see IS 1260 (Part 1) : 1973].

7 SAMPLING

The procedure for drawing representative samples of the material and their criteria for conformity shall be as prescribed in Annex B.

ANNEX A

(Clauses 4.3, 4.4, 4.5 and Table 1)

METHODS OF TEST FOR CAUSTIC SODA

A-1 QUALITY OF REAGENTS

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

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

A-2 PREPARED SAMPLE SOLUTION

A-2.1 In a weighing bottle with a ground-glass stopper, weigh to the nearest 0.01 g, of the material (solid or liquid), equivalent to a little less than 50 g, of caustic soda.

A-2.1.1 Solid Material

Dissolve the material (see A-2.1) in approximately 200 ml of water and cool it to room temperature; transfer the solution quantitatively to a 500-ml one-mark volumetric flask and dilute nearly to the mark, recool, then dilute to the mark and mix thoroughly. The solution thus prepared shall be used for the subsequent tests.

A-2.1.2 Liquid Material

Transfer the material (see A-2.1) directly to a 500-ml one-mark volumetric flask and dilute nearly to the mark; cool to room temperature, then dilute to the mark and mix thoroughly.

A-3 DETERMINATION OF CARBONATES

A-3.0 Two methods, namely, Method *A* Double Indicator Method and Method *B* Gaseometric Method have been prescribed. Method *A* shall be used for routine analysis and Method *B* shall be used as a referee method.

A-3.1 Method A (Double Indicator Method)

A-3.1.1 Reagents

A-3.1.1.1 Standard hydrochloric acid — 1 *N*.

A-3.1.1.2 Standard hydrochloric acid — 0.1 *N*.

A-3.1.1.3 Methyl orange indicator solution — Dissolve 0.1 g of methyl orange in 100 ml of water.

A-3.1.1.4 Phenolphthalein indicator solution — Dissolve 0.1 g of phenolphthalein powder in 60 ml of rectified spirit (*sec* IS 323 : 1959) and dilute with water to 100 ml.

A-3.1.2 Procedure

Pipette out 25 ml of the prepared sample solution (*see* A-2) into a conical flask and add 2 to 3 drops of phenolphthalein indicator. Titrate it against standard hydrochloric acid solution (1 *N*) up to a little before the end point. Take this reading as *A*. Further titrate it against standard hydrochloric acid (0.1 *N*) till the pink colour just disappears. Take this reading as *B*. Then add 2 to 3 drops of methyl orange indicator and continue titration against standard hydrochloric acid (0.1 *N*) to a reddish orange colour. Take this reading as *C*.

A-3.1.3 Calculation

Carbonates (as Na_2CO_3),

$$\text{Percent by mass, } A = \frac{212 (C - B) \times N}{M}$$

where

N = normality of standard hydrochloric acid (0.1*N*) and,

M = mass of the material taken for test.

A-3.2 Method B (Gaseometric Method)

A-3.2.0 Principle

Measurement of the volume of carbon dioxide evolved from a portion of the test sample by reaction with a hydrochloric acid.

A-3.2.1 Reagents

A-3.2.1.1 Distilled water, or water of equivalent purity—Free from carbon dioxide at room temperature. Eliminate any carbon dioxide present in water either by boiling for 10 minutes and

cooling it in the absence of atmospheric carbon dioxide or, more simply, by bubbling air free from carbon dioxide through it for 15 minutes. The air is freed from carbon dioxide by passing it through a column containing pellets of sodium hydroxide. Store it in the absence of atmospheric carbon dioxide.

A-3.2.1.2 Hydrochloric acid — 12 *N*.

A-3.2.1.3 Sodium chloride, coloured acid solution

Dissolve 26 g of sodium chloride in water. Add 5 ml of sulphuric acid solution ($d=1.83$). Dilute it to 1 000 ml, add a small amount of 0.05 percent methyl orange indicator solution and mix thoroughly.

A-3.2.1.4 Sodium hydroxide solution — 6 *N*.

A-3.2.2 Apparatus — Apparatus as assembled is shown in Fig. 1.

A-3.2.3 Procedure

A-3.2.3.1 Weigh, to the nearest 0.01 g, the test sample (solid or lye) corresponding to approximately 10 g of caustic soda.

A-3.2.3.2 Assembly of the apparatus

Fill burette *B* of the apparatus with the coloured acid solution through leveling bottle *F*. Pour into absorber *C* some of the sodium hydroxide solution. (Renew this solution after 100 determination.) With burette *B* and absorber *C* filled up to cock *R*₁ and graduation mark *a*, respectively, and cocks *R*₁ and *R*₂ closed, place the test portion in flask *A* and, in the case of solid material, dissolve it in approximately 30 ml of water. Dilute to approximately 40 ml so as to reduce the dead space to a volume slightly greater than 100 ml (volume above the level of the liquid in flask *A* plus the volume of condenser tube *D* up to cock *R*). Place in the flask three porcelain or glass balls, about 2 mm in diameter, and a few pieces of pumice having a total volume approximately equal to that of the balls. Stopper the flask and close cock *R*. Connect flask *A* with burette *B* through cock *R*₁ and lower the leveling bottle *F*. Check the tightness of the apparatus by appropriately handling the cocks and the leveling bottle.

A-3.2.3.3 Evolution and measurement of carbon dioxide

By means of a separating funnel, pour 35 ml of hydrochloric acid into flask *A* taking care to avoid loss of gas. The acidity of the solution in the flask is thus approximately 2 *N* after the evolution of carbon dioxide. Heat the flask and maintain the solution at boiling point for

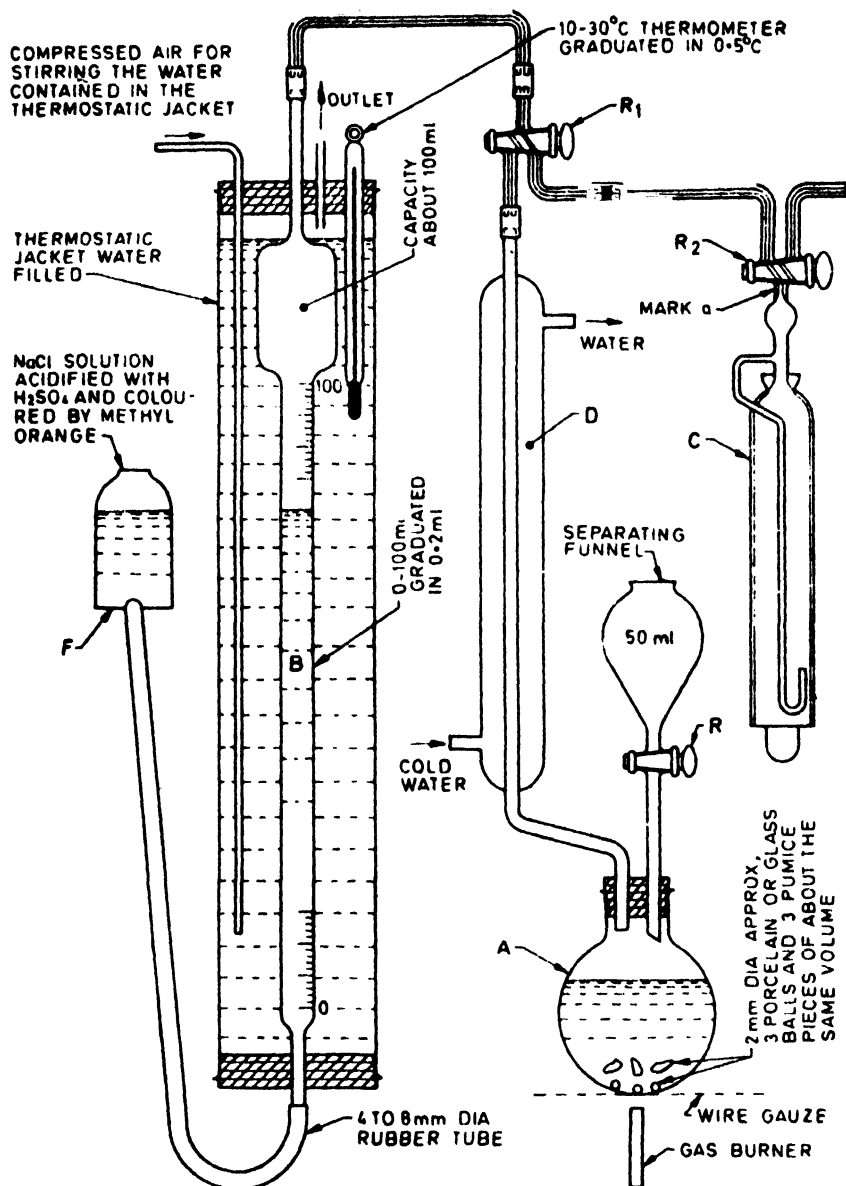


FIG 1 ASSEMBLY OF APPARATUS FOR DETERMINATION OF CARBONATES

5 minutes while running cold water through the condenser. Then stop heating and add more of the coloured acid solution by means of the separating funnel, lowering the leveling bottle *F* still further to make the solution in the flask *A* rise in the condenser tube up to cock *R*₁. Then close the latter and wait for 5 minutes to allow the gas to reach the temperature of the water jacket. Measure the volume of gas *V* at atmospheric pressure *P* and at the temperature *t* of the water in the jacket. For this purpose move the leveling bottle *F* to bring the coloured acid solution to the same level both in the flask and

in the burette *B*; read the volume of the latter. Adjust the cocks *R*₁ and *R*₂ so that burette *B* and absorber *C* are connected, then raise the leveling bottle *F* so that the gas is transferred to the absorber where the carbon dioxide is absorbed. Then transfer the non-absorbed gas back to burette *B* by lowering the leveling bottle *F* and, after adjusting the level of the hydroxide solution to the gauge mark *a* on absorber *C*, close cock *R*₂ and read the volume of the residual gas. Repeat these operations until a constant volume *V*₁ is obtained. The difference in the volume, *V* - *V*₁, represents

the carbon dioxide contained in the test portion, measured at atmospheric pressure and at the temperature of the water jacket.

A-3.2.4 Calculation

Carbonates (as Na_2CO_3), percent
by mass, $A = 0170 \frac{P-p}{273+t} \times \frac{V-V_1}{M}$

where

P = atmospheric pressure in mm, Hg at the time of determination,

p = vapour pressure, in mm, Hg of the coloured acid solution at the temperature of the water contained in the jacket,

t = temperature in degrees centigrade of the water contained in the water jacket,

V = volume, in ml, of gas before absorption of carbon dioxide,

V_1 = volume, in ml, of gas after absorption of carbon dioxide, and

M = mass, in g of the material taken for the test.

A-4 DETERMINATION OF SODIUM HYDROXIDE

A-4.1 Reagents

A-4.1.1 Standard Hydrochloric Acid — 1 N.

A-4.1.2 Methyl Orange Indicator Solution — Dissolve 0.1 g of methyl orange in 100 ml of water.

A-4.2 Procedure

Transfer exactly 20 ml of the sample solution (see A-2) to a 500-ml conical flask. Add approximately 80 ml of water and 5 drops of methyl orange indicator solution and titrate it against standard hydrochloric acid until the colour of the indicator changes from yellow to orange.

A-4.3 Calculation

Total alkalinity (as NaOH),
percent by mass $B = \frac{V \times N}{M} \times 99.9925$

where

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

N = normality of standard hydrochloric acid, and

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

Sodium hydroxide (as NaOH), percent by mass = $B - (A \times 40/53)$

where

B = total alkalinity (as NaOH), and

A = carbonate content (as Na_2CO_3) as calculated in A-3.3 or A-3.2.4.

A-5 DETERMINATION OF CHLORIDES

A-5.1 Reagents

A-5.1.1 Concentrated Nitric Acid

A-5.1.2 Standard Silver Nitrate Solution — 0.1.

A-5.1.3 Nitrobenzene

A-5.1.4 Standard Ammonium Thiocyanate Solution — 0.1 N.

A-5.1.5 Ferric Ammonium Sulphate Indicator — saturated solution.

A-5.1.6 Diluted Sample for Testing Chlorides in Technical Grade

Pipette out 50 ml of the sample solution (see A-2.1.1) to a 250-ml standard measuring flask and make up the volume to 250 ml mark with water. This solution shall also be used for the determination of iron content.

A-5.2 Procedure

A-5.2.1 For Caustic Soda, Solid

Transfer exactly 50 ml of the sample (see A-5.1.6) solution to a conical flask, neutralize it with concentrated nitric acid and then add 5 ml of the acid in excess. Add 5 ml of nitrobenzene and shake vigorously; pipette out into this 20 ml of standard silver nitrate solution. Titrate it against standard ammonium thiocyanate solution using ferric ammonium sulphate indicator.

A-5.2.2 For Caustic Soda, Lye

Weigh accurately a quantity of the material containing not more than 0.05 g of chlorides (as NaCl) and carry out the test as prescribed in A-5.2.1.

A-5.2.3 For Caustic Soda, Pure Grade

Weigh accurately about 5 g of the material, dissolve it in about 50 ml distilled water and carry out the test as prescribed in A-5.2.1.

A-5.3 Calculation

A-5.3.1 For Caustic Soda, (Solid Technical)

Chlorides (as NaCl),

percent by mass = $\frac{292.3 (20 N_1 - V N_2)}{M}$

where

N_1 = normality of standard silver nitrate solution,

V = volume in ml of standard ammonium thiocyanate solution used in A-5.2.1,

N_2 = normality of standard ammonium thiocyanate solution, and

M = mass in g of the material taken in A.2.1.

A-5.3.2 For Caustic Soda, Lye

Chlorides (as NaCl),

$$\text{percent by mass} = \frac{5.845 (20 N_1 - V N_2)}{M}$$

where

N_1 and N_2 are the same as specified in A.5.3.1.

V = volume in ml of standard ammonium thiocyanate solution used in A.5.2.2, and

M = mass in g of the material taken for the test in A-5.2.2.

A-5.3.3 For Caustic Soda, Pure Grade

Chlorides (as NaCl).

$$\text{percent by mass} = \frac{5.845 (20 N_1 - V_1 N_2)}{M}$$

where

N_1 and N_2 , are the same as specified in A.5.3.1

V_1 = volume in ml of standard ammonium thiocyanate solution used in A-5.2.3, and

M = mass in g of the material taken for the test in A-5.2.3.

A-6 DETERMINATION OF SULPHATES

A-6.1 Reagents

A-6.1.1 *Concentrated Hydrochloric Acid* — see IS 265 : 1987.

A-6.1.2 *Barium Chloride Solution* — 10 percent.

A-6.2 Procedure

Weigh accurately about 10 g of the material and dissolve it in 100 ml of water. Add sufficient quantity of concentrated hydrochloric acid to neutralize and leave an excess of acid in the solution. Boil it to decompose the carbonates. Filter the resulting solution through a folded filter paper thoroughly, collecting both the filtrate and washing in a 500-ml beaker.

Dilute it to 250 ml, boil and add 10 ml of hot barium chloride solution to the boiling solution. Boil it again for 2 minutes; let it stand for 4 hours, and then filter through a tared sintered glass crucible (G No. 4) or a tared Gooch crucible. Wash the precipitate to free it from chlorides, and dry to constant mass at 105 to 110°C.

A-6.2.1 Excess barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by the addition of barium chloride in a slow stream with stirring minimizes the mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

A-6.3 Calculation

Sulphates (as Na_2SO_4),

$$\text{percent by mass} = \frac{60.86 M_1}{M}$$

where

M_1 = mass in g of the precipitate, and

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

A-7 DETERMINATION OF SILICATES

A-7.0 Outline of the Method

Silica is determined colorimetrically by visual comparison or by spectrophotometer as molybdenum blue.

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 100-ml capacity.

A-7.2 Reagents

A-7.2.1 *Dilute Sulphuric Acid* — approximately

A-7.2.2 *Ammonium Molybdate Solution* — Dissolve 10 g of ammonium molybdate in 100 ml of dilute sulphuric acid (1 N).

A-7.2.3 *Sodium Citrate Solution* — Dissolve 8 g of sodium citrate (as $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7, 2\text{H}_2\text{O}$) in 100 ml of water.

A-7.2.4 *Reducing Solution* — Dissolve 7 g of anhydrous sodium sulphite in 100 ml of water and add 1.5 g of 1-amino-2-naphthol-4-sulphonic acid. When this is dissolved, add to this solution, a solution containing 90 g of sodium bisulphite in 800 ml of water, and dilute to 1 000 ml.

A-7.2.5 *Sodium Carbonate, Anhydrous* — see IS 296 : 1986

A-7.2.6 Standard Silicate Solution — Fuse 0.1 g of pure precipitated silica with anhydrous sodium carbonate in a platinum crucible and leach the melt with water, neutralise with dilute sulphuric acid and dilute to 1 000 ml. Dilute 10 ml of this solution to 100 ml. One ml of the diluted solution contains 0.01 mg of silica (as SiO_2). Store the solution in a polyethylene bottle.

A-7.3 Procedure

Dissolve 5 g of the sample (10 g in case of caustic soda in lye form) in water in a polyethylene beaker and make upto 250 ml in a standard flask. Transfer 10 ml of the solution into a polyethylene beaker, neutralise with dilute sulphuric acid and add excess of acid to bring the *pH* of the solution to 1.5 to 1.6. Transfer to a 100-ml Nessler cylinder, add 2 ml of ammonium molybdate solution and allow to stand for 5 minutes. Then add 5 ml of sodium citrate solution, followed by 2 ml of reducing solution (*see* A-7.2.4). Dilute to 100 ml and stir well.

Carry out a control test in another Nessler cylinder, using 4 ml of standard silica solution in place of the sample and the same quantities of other reagents in the same total volume of reaction mixture. Allow the two Nessler cylinders to stand for 30 minutes and compare the colour developed in the two cylinders.

A-7.3.1 The limit prescribed for silicates shall be taken as not having been exceeded if any blue colour produced in the test with the material is not deeper than that produced in the control test.

A-7.4 The colour produced can also be measured on a spectrophotometer by measuring the absorbance at 820 nm and compared with a standard graph drawn as given below:

A standard graph is drawn by measuring the absorbance of 2 ml, 4 ml, 6 ml, 8ml and 10 ml of standard silica solution treated with the above reagents in the same way and plotting the absorbance against the silica content.

AS DETERMINATION OF IRON

A-8.1 Apparatus

A-8.1.1 Nessler Cylinders — 100-ml capacity.

A-8.2 Reagents

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

A-8.2.2 Ammonium Persulphate

A-8.2.3 Potassium Thiocyanate Solution — approximately 5 percent.

OR

Ammonium Thiocyanate Solution — approximately 5 percent.

A-8.2.4 Standard Iron Solution

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 100 ml of distilled water and 10 ml of concentrated sulphuric acid, add dilute potassium permanganate solution dropwise (0.2 percent, *m/v*) until a slight pink colour persists after stirring, and then dilute with water to 1 000-ml mark. Transfer 100 ml of this solution to 1 000-ml volumetric flask and dilute again to 1 000-ml mark. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

A-8.3 Procedure

A-8.3.1 For Caustic Soda, Solid

Transfer by means of a pipette 25 ml of the sample solution (*see* A-2.1) for pure grade, or 10 ml of the diluted sample solution (*see* A-5.1.6) for technical grade, dilute to 50 ml and make acidic with concentrated hydrochloric acid. Add 30 mg of ammonium persulphate and boil to oxidize the iron. Cool and transfer to a Nessler cylinder, add 2 ml of potassium thiocyanate solution (or ammonium thiocyanate solution) and dilute to 100-ml mark with water. In another Nessler cylinder, take the same amount of concentrated hydrochloric acid, ammonium persulphate and potassium thiocyanate solution (ammonium thiocyanate solution), as used with the material and dilute to about 85 ml. From a burette add standard iron solution in small portions at a time so that after dilution to 100 ml, the colour obtained matches with that obtained with the material.

A-8.3.2 For Caustic Soda, Lye

Weigh accurately an amount of the material containing not more than 0.035 mg of iron (as Fe) and carry out the test as in A-8.3.1.

A-8.4 Calculation

A-8.4.1 For Caustic Soda, Solid (Technical)

$$\text{Iron (as Fe) , ppm} = \frac{2\ 500\ V}{M}$$

A-8.4.2 For Caustic Soda (pure)

$$\text{Iron (as Fe) . ppm} = \frac{200\ V}{M}$$

where

V = volume in ml of standard iron solution used in **A-8.3.1**, and

M = mass, in g of the material taken for the test (see **A-2.1** and **A-5.1.6**).

A-8.4.3 For Caustic Soda, Lye

$$\text{Iron (as Fe), ppm} = \frac{10 V}{M}$$

where

V = volume, in ml, of standard iron solution used in **A-8.3.2**, and

M = mass in g of the material taken for the test in **A-8.3.2**.

A.9 DETERMINATION OF COPPER

A-9.1 Apparatus

A-9.1.1 Nessler Cylinders — 100-ml capacity.

A-9.2 Reagents

A-9.2.1 Concentrated Sulphuric Acid

A-9.2.2 Litmus Paper

A-9.2.3 Ammonium Chloride

A-9.2.4 Concentrated Ammonium Hydroxide — sp gr 0.92.

A-9.2.5 Citric Acid Solution — Dissolve 100 g of citric acid (monohydrate) crystals in 100 ml of water.

A-9.2.6 Dilute Ammonium Hydroxide — approximately 3 N.

A-9.2.7 Gum Acacia Suspension — Dissolve 1.0 g of gum acacia in 100 ml of boiling water, filter and dilute to 200 ml. (This solution shall be prepared fresh.)

A-9.2.8 Tetrasodium Pyrophosphate Solution — Dissolve 4 g of tetrasodium pyrophosphate in 100 ml of water.

A-9.2.9 Sodium Diethyl Dithiocarbamate Solution — Dissolve 0.2 g sodium diethyl dithiocarbamate in water and make up the volume to 100 ml.

A-9.2.10 Standard Copper Solution — Dissolve 0.392 8 g of copper sulphate pentahydrate ($\text{Cu SO}_4 \cdot 5\text{H}_2\text{O}$) in slightly acidulated water and make up the volume to 1 000 ml in a volumetric flask. Pipette out exactly 10 ml of the solution in a 100-ml volumetric flask and make up the volume to 100-ml mark. One millilitre of this solution is equivalent to 0.01 mg of copper.

A-9.2.11 pH-Meter

A-9 3 Procedure

A-9.3.1 Weigh about 10 g of material, to the nearest 0.1 g, and transfer it into a 400-ml beaker. Dissolve it in about 20 ml of water and then neutralize with concentrated sulphuric acid using litmus paper. To the neutralized solution add ammonium chloride and concentrated ammonium hydroxide to precipitate out iron. Heat the solution to coagulate the precipitate. Filter the precipitate through a Gooch crucible with an asbestos mat and wash the residue twice with water. Collect the filtrate and the washing in a beaker. Cover the beaker with a watch glass and then evaporate to a volume of about 70 ml. Transfer the solution to 100-ml Nessler cylinder. To this add 2 ml citric acid solution and add dilute ammonium hydroxide (till pH 8.5). Mix the solution thoroughly and then add 10 ml of gum acacia suspension, 5 ml of tetrasodium pyrophosphate solution and 10 ml of the sodium diethyl dithiocarbamate solution and dilute to 100-ml mark with distilled water. To another Nessler cylinder add 2 ml of citric acid solution dilute ammonium hydroxide (till pH 8.5), 10 ml gum acacia suspension, 5 ml of tetrasodium pyrophosphate solution, 10 ml of sodium diethyl dithiocarbamate solution and then 2 ml of standard copper solution. Make up to 100-ml mark with distilled water and mix well. Compare the colour produced in the two cylinders after 10 minutes.

A-9.3.1.1 The limit prescribed shall be taken as not having exceeded if the intensity of colour produced in the test with the material is not greater than that in the control test.

A-10 DETERMINATION OF MANGANESE

A-10.1 Apparatus — Nessler cylinder, 100-ml capacity.

A-10.2 Reagents

A-10.2.1 Phosphoric Acid — 85 percent (manganese free).

A-10.2.2 Potassium Periodate

A-10.2.3 Standard Manganese Solution—Dissolve 0.307 7 g of manganese sulphate, monohydrate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$) in water, add 1 ml of concentrated sulphuric acid and make up the volume to 1 000-ml in a volumetric flask. Pipette out 10 ml of this solution in a 100-ml volumetric flask and dilute to the mark. One ml of this solution is equivalent to 0.01 mg of manganese.

A-10.3 Procedure

A-10.3.4 Weigh about 10 g of the material to the nearest 0.1 g and transfer it to a 400 ml beaker and dissolve in about 40 ml water. To this solution, add 10 to 15 ml of phosphoric acid and 0.6 to 0.8 g potassium periodate. Heat the solution to boiling. Boil for 20 minutes. Cool the solution to room temperature. Transfer the solution to a 100 ml Nessler cylinder. Make up to the mark with distilled water and mix well. Compare the colour with that in the other Nessler cylinder containing 1 ml of standard manganese solution in place of the test solution treated similarly under similar conditions with the same amounts of reagents for the same time as in the test solution, and make up to 100 ml mark.

A-10.3.2 The limit prescribed shall be taken as not having exceeded if the intensity of colour produced in the test with the material is not greater than that in the control test.

A-11 DETERMINATION OF CHLORATES AND PERCHLORATES (AS SODIUM CHLORATE)

A-11.0 Two methods, namely, Method A and Method B have been prescribed.

A-11.1 Method A**A-11.1.0 Outline of the Method**

A little excess of ferrous ammonium sulphate added to the slightly acidified solution of caustic soda is oxidized quantitatively by the chlorate present in caustic soda. The chlorate is estimated by back titrating the excess ferrous ammonium sulphate with potassium permanganate.

A-11.1.1 Reagents

A-11.1.1.1 Ferrous ammonium sulphate solution — 0.02 N Dissolve 3.92 g of ferrous ammonium sulphate crystals in 300 ml of water. Add 1 ml of concentrated sulphuric acid and make up to 500 ml.

A-11.1.1.2 Sulphuric acid — 1:1 (v/v).

A-11.1.1.3 Potassium permanganate solution — 0.1 N Weigh about 3.0–3.25 g of potassium permanganate into a beaker, add a little of water and boil it gently for 15 minutes. After cooling, filter the solution through funnel with a plug of glass wool and dilute to 1 000 ml. Collect the filtrate in a brown-coloured bottle and standardize with sodium oxalate solution.

A-11.1.1.4 Potassium permanganate solution — 0.02 N Dilute 50 ml of 0.1 N potassium permanganate solution (see A-11.1.1.3) to 250 ml in a volumetric flask.

A-11.1.2 Procedure

A-11.1.2.1 Weigh accurately about 50 g of caustic soda sample in a beaker and neutralize it with sulphuric acid carefully after placing the beaker in cold water. Add 5 ml of the acid in excess and dilute to 250 ml. Transfer the contents of the beaker to a 500 ml-conical flask add 25 ml of ferrous ammonium sulphate solution, close the flask with a bunsen valve and boil the contents gently for 15 minutes. Allow the flask to cool to room temperature. After cooling, titrate the contents of the flask with 0.02 N potassium permanganate, solution slowly with stirring till a pink colour persists. Note the volume V_2 .

A-11.1.2.2 Run a blank by titrating 25 ml of ferrous ammonium sulphate containing 5 ml of sulphuric acid and 200 ml of water against 0.02 N potassium permanganate, proceeding in the same way as that of test. Note the volume as V_1 .

A-11.1.3 Calculation

Chlorates (as NaClO_3 ,),

$$\text{ppm} = \frac{(V_1 - V_2) \times N \times 17.74 \times 10^3}{M}$$

where

V_1 = volume, in ml, of standard potassium permanganate solution used in the blank;

V_3 = volume, in ml, of standard potassium permanganate solution used in the test;

N = normality of standard potassium permanganate solution; and

M = mass, in g, of the material taken for the test.

A-11.2 Method B**A-11.2.0 Outline of the Method**

A little excess of ferrous sulphate added to a slightly acidified solution of caustic soda is oxidized quantitatively by the chlorate in caustic soda. The chlorate is estimated by back titrating the excess ferrous ammonium sulphate with standard potassium dichromate solution.

A-11.2.1 Reagents

A-11.2.1.1 Ferrous ammonium sulphate — 0.02 N Dissolve 3.92 g of ferrous ammonium sulphate crystals in 300 ml of water. Add 1 ml of concentrated sulphuric acid and dilute to 500 ml.

A-11.2.1.2 Dilute sulphuric acid — 1:1 (v/v).

A-11.2.1.3 Phosphoric acid — 1:1 (v/v).

A-11.2.1.4 Sodium diphenyl amine sulphonate solution — Dissolve 0.2 g of sodium diphenyl amine sulphonate in 100 ml of water.

A-11.2.1.5 Potassium dichromate solution — 0.02N. Powder finely about 2 g of potassium dichromate (AR grade) and dry in an air oven at 140-150 C for one hour and cool in a desiccator. Accurately weigh 0.98 g of the dried potassium dichromate and dissolve in 1 000 ml of water in a volumetric flask.

A-11.2.2 Procedure

A-11.2.2.1 Weigh accurately about 50 g of caustic soda in a 500 ml-beaker. Neutralize it with dilute sulphuric acid (1:1) carefully, keeping the beaker in cold water. Add an excess of 20 ml of dilute sulphuric acid. Transfer the contents of the beaker into a 500 ml conical flask. Pipette out 25 ml of ferrous ammonium sulphate solution into the flask. Close the flask with a Bunsen valve, boil the contents for 15 minutes and allow to cool to room temperature. After cooling, add 20 ml of phosphoric acid (1:1) and 0.5 ml sodium diphenyl amine sulphonate solution into the flask. Titrate the solution with potassium dichromate solution to a violet colour. Note the volume as V_2 .

A-11.2.2.2 Run a blank by titrating 25 ml of ferrous ammonium sulphate, 20 ml of dilute sulphuric acid, 20 ml of phosphoric acid and 0.5 ml of sodium diphenyl amine sulphonate against potassium dichromate solution by proceeding in the same way as in the test. Note the volume as V_1 .

A-11.2.3 Calculation

Chlorates (as NaClO_3),

$$\text{ppm} = \frac{(V_1 - V_2) \times N \times 17.74 \times 10^3}{M}$$

where

V_1 = volume, in ml, of standard potassium dichromate solution used in the blank;

V_2 = volume, in ml, of standard potassium dichromate solution used in the test;

N = normality of standard potassium dichromate solution; and

M = mass, in g, of the material taken for the test.

A-12 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-12.1 Reagent

A-12.1.1 Concentrated Hydrochloric Acid — See IS 265:1987.

A-12.2 Procedure

Weigh accurately about 50 g of the material and transfer it into a 600-ml beaker, add 300 ml of water and stir until dissolved. Add concentrated hydrochloric acid till the solution is just alkaline to phenolphthalein. Bring to boil and allow the solution to settle on the hot-plate for 15 minutes. Filter through a weighed Gooch crucible or tared sintered glass crucible (G No. 4) and wash with hot water to free it from alkali allowing water to drain completely after each washing. Dry it in an oven for one hour at 105 to 110°C. Cool it in a desiccator and weigh.

A-12.3 Calculation

Matter insoluble in water,

$$\text{percent by mass} = 100 \frac{M_1}{M}$$

where

M_1 = mass, in g, of the insoluble residue, and

M = mass, in g, of the material taken for the test.

A-13 CALCULATION OF RESULTS ON THE DRY BASIS

A-13.1 For the purpose of this standard add the percentage of sodium hydroxide, carbonates (as Na_2CO_3), chlorides (as NaCl), sulphates (as Na_2SO_4) and silicates (as SiO_2) and treat the total as the total dry solid content of the solution. On this basis calculate the content of the different components on the dry basis.

A-13.1.1 Example

A-13.1.1.1 If the total percentage of the determined components of a solution is 50.4 and the percentage of sodium hydroxide content is 50.2, the percentage of sodium hydroxide on dry basis will be

$$\frac{50.2 \times 100}{50.4} = 99.6$$

Again the above example, if the iron content is 10 ppm, the content of iron on dry basis will be

$$\frac{10 \times 100}{50.4} = 19.8 \text{ ppm.}$$

A-13.1.1.2 If the total percentage of the determined components of the solid is 99.5 and the percentage of sodium hydroxide content is 99.1, the percentage of sodium hydroxide on

dry basis will be

$$\frac{99.1 \times 100}{99.5} = 99.6$$

ANNEX B

(Clause 7.1)

SAMPLING OF CAUSTIC SODA, PURE AND TECHNICAL

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination, particularly from absorption of water and carbon dioxide.

B-1.2 To draw a representative sample, the contents of each container selected from sampling shall be mixed thoroughly by suitable means.

B-1.3 The sample shall be placed in clean, dry and air-tight alkali resistant glass containers.

B-1.4 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and the batch number.

R-2 SCALE OF SAMPUNG

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.

B-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the specified requirements. The number of container (*n*) to be selected at random from lots of different sizes (*N*) shall be in accordance with Table 2.

B-2.3 The container shall be drawn at random from the lot, and to ensure randomness, the following procedure may be adopted;

Arrange all the containers in the lot in a systematic manner, and starting from any

one, count them as 1, 2, up to *r*, where *r* is the integral part of *N/n*. Every *r*th container thus counted shall be included in the sample till the required number of containers specified in col 2 of Table 2 is taken out.

Table 2 Number of Containers to be Selected for Sampling
(Clause B-2.2)

Lot Size	Number of Containers to be Selected
<i>N</i>	<i>n</i>
(1)	(2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1 000	7

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Caustic Soda, Solid

B-3.1.1 Scrap off 50 mm of the material from the top centre and then take the sample. The quantity of the material so drawn shall be not less than 200 g and the mass of the total material taken out shall not exceed 1 kg. The number of portions to be collected and the quantity of material taken from each portion may be suitably reduced in case of big containers to keep the size of the material taken out at 1 kg. Mix rapidly the material so collected from any particular container on a clean dry surface after scrapping off the surface carbonate that may be formed during sampling.

B-3.1.2 From each of the portions representing the containers selected, take out 600 g and mix thoroughly the material collected. This shall

constitute the composite sample. Divide this composite sample into three parts, transfer each part to a sample container which shall then be sealed air-tight with well-fitting corks coated with paraffin wax.

B-3.1.3 The portion of the material left (after the quantity for the preparation of the composite sample has been taken out) in respect of each container shall also be divided into three parts. Each such part shall constitute an individual test sample and shall be transferred to a sample container which shall then be closed as in **B-3.1.2** and labeled giving full identification particulars. One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.2 Caustic Soda Lye

B-3.2J *From Tank Cars or Tank Wagons*

Draw samples from each tank car or tank wagon from different levels by means of a suitable sampling instrument. Mix the material so collected and transfer about 5 litres of it to a clean resistant glass bottle. Divide the material into three parts and transfer each part to a sample container which shall then be closed as in **B-3.1.2** and labelled giving full identification particulars. Each such bottle shall constitute an individual sample; one of these shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.2.2 *For Containers*

B-3.2.2.1 Draw representative portions from each container selected from sampling after thoroughly mixing the container; take out about 2 litres from each selected container.

B-3.2.2.2 From each of the portions representing the selected containers, take out about 1 200 ml and mix the material so obtained. This shall constitute the composite sample. Divide this composite sample into three parts, one for the purchaser, another for the supplier and the third for the referee. Transfer each part to a sample container which shall then be closed as in **B-3.1.2**.

B-3.2.3 The portion of the material (after the quantity required for the preparation of the

composite sample has been taken out) in respect of each container shall also be divided into three parts. Each such part shall constitute an individual sample and shall be transferred to sample container which shall then be closed as in **B-3.1.2** and labelled giving full identification particulars. One of these three sets shall be marked for the purchaser another for the supplier and the third for the referee.

B-3.3 Referee Sample

The referee sample (*see* **B-3.1.2**, **B-3.1.3**, **B-3.2.1**, **B-3.2.2.2** and **B-3.2.3**) shall bear the names of the purchaser and the supplier and shall be used in case of a dispute between the two. It shall be kept at a place agreed to between the purchaser and the supplier.

B.4 NUMBER OF TESTS

B.4.1 For Tank Car or Tank Wagons

Tests for all the characteristics prescribed in **4.3** shall be carried out on the individual samples.

B.4.2 For Containers

B-4.2.1 Tests for the determination of iron shall be conducted on each of the individual samples.

B-4.2.2 Tests for the remaining characteristics prescribed in **4.3** shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Tank Cars or Tank Wagons

The tank car or tank wagon shall be declared as conforming to the specification if the individual sample satisfies all the requirements prescribed.

B-5.2 For Containers

B-5.2.1 *For Iron*

The lot shall be considered conforming to the requirement for iron if each of the individual samples satisfies the tests in **A-8**.

B-5.2.2 *For Composite Sample*

The test results on the composite sample shall meet the specified requirements.

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