भारतीय मानक
Indian Standard

IS 646 : 2020

तरल क्लोरीन, तकनीकी — विशिष्टि

(तीसरा पुनरीक्षण)

Liquid Chlorine, Technical — Specification

(Third Revision)

ICS 71.100.80

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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June 2020 **Price Group 6**

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

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

This standard was originally published in 1956 and was subsequently revised in 1970 and 1986 to incorporate an improved sampling procedure and gravimetric method for determination of moisture and an alternate routine method for the determination of chlorine content using Orsat apparatus. Now, this standard is again being revised to incorporate requirements and methods of test for mercury, arsenic and lead. Also, Amendments No. 1 and 2 have been incorporated in this revision.

Although the product is packed and sold as a liquid, it is usually used as a gas obtained by evaporating the liquid from the cylinder. It is used mainly in paper, pulp and textile bleaching, water sterilization, and manufacture of chemicals. Chlorine is a powerful irritant to skin, mucous membrane and respiratory system. Because of the hazardous nature of liquid chlorine, intending users are strongly advised to take guidance from IS 4263.

In India, chlorine is deemed to be an explosive, when contained in any metal container, in a compressed or liquefied state, within the meaning of the *Indian Explosives Act,* 1984. The filling, possession, transport and importation is governed by the Gas Cylinder Rules, 2016.

This standard was formulated by CHD 01 technical committee. The list of experts who had made significant contribution to the formulation of this standard is given at Annex C.

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

LIQUID CHLORINE, TECHNICAL — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for liquid chlorine, technical, used in the bleaching of pulp and textiles, water sterilization, and manufacture of chemicals.

2 REFERENCES

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

IS No.	*Title*
5 : 2007	Colours for ready mixed paints and enamels (*sixth revision*)
299 : 2012	Alumino-ferric — Specification (*fifth revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
4263 : 1967	Code of safety for chlorine
11124 : 1984	Methods for determination of arsenic by atomic absorption spectrophotometry — Test method
12074 : 1987	Methods for determination of lead by atomic absorption spectrophotometry — Test method

3 REQUIREMENTS

3.1 Description

The material shall be clear yellow liquid and when evaporated over a clean white tile should leave no residue (solid or liquid).

3.2 Composition

The material when tested according to the methods given in Annex A, shall comply with the requirement given in Table 1.

Table 1 Requirement for Liquid Chlorine

(*Clause* 3.2)

Sl No.	**Characteristic**	**Requirement**	**Method of Test Ref to Clause No.**
(1)	(2)	(3)	(4)
i)	Chlorine in vapourized liquid, percent by volume, *Min.*	99.8	A-2
ii)	Moisture, parts per million (ppm)	150	B
iii)	Mercury (as Hg), parts per million (ppm)	20	A-3
iv)	Arsenic (as As), parts per million (ppm), *Max*	200	A-4
v)	Lead (as Pb), parts per million (ppm), *Max*	200	A-5

4 PACKING AND MARKING

4.1 Packing

Chlorine shall be supplied in liquefied condition in suitable cylinders or other containers whose capacity shall be subject to agreement between the purchaser and the supplier.

4.2 The cylinders shall comply with the requirements for cylinders for liquid gases given in the *Gas Cylinder Rules*, 2016, of the Government of India, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India, or any other duly constituted authority. Other containers shall also conform to the requirements set out by the above mentioned authority and be approved by such authority.

4.3 The packing, marking and labelling of cylinders shall be in accordance with the requirements of cylinders for liquid gases given in the *Gas Cylinder Rules*, 2016, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India, or any other duly constituted authority. Other containers shall also be marked and labelled in accordance with the instructions issued from time to time by the above mentioned authority.

4.4 Chlorine cylinders shall be painted with a coat of golden yellow IS Colour No. 356 (*see* IS 5) paint.

4.5 Marking

4.5.1 Each container/cylinder shall be marked with the following:

a) Name of the material, that is, 'liquid chlorine'.

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

c) Mass, in kg;

d) Date of filling/packaging of container/cylinder; and

e) Lot number or batch number or otherwise.

NOTE — The marking should be large enough to convey immediate understanding and to allow the use of readily legible characters.

4.5.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 Samples shall be drawn from cylinders or containers or from the filling rack during the time the cylinders are in the process of filling. Samples shall be collected following the procedure given in **5.1.1** to **5.1.4** and in **A-2.1** and **A-2.2**. It is preferable to take the sample over a period of about 25 to 30 min at a purge rate of 50 bubbles/min. It is recommended that the purged gas may be absorbed in milk of lime or caustic soda solution as a safety precaution.

5.1.1 *Sampling from Containers* — Place the container on its side with the valve in the vertical line to permit withdrawal of liquid chlorine through the lower valve.

5.1.2 *Sampling from Cylinders* — Support the cylinder at an angle of 45° to ensure that no air is sucked in along with chlorine with the valve end down as shown in Fig. 1, to permit withdrawal of liquid chlorine.

5.1.3 In each of the cases mentioned under **5.1.1** and **5.1.2**, it is advisable to employ the filtering tube connected to the supply valve and needle valve by couplings. From the needle valve, a 3 mm copper tubing is joined with close fitting rubber tubing for taking the sample in the collecting bomb (*see* Fig. 1) as described under **A-2.1**.

5.1.4 The samples of chlorine drawn from cylinders and containers shall be allowed to come to thermal equilibrium and the pressure inside the collecting bomb *D* brought to room conditions before analysis is attempted.

NOTE — The analysis of liquid chlorine is a difficult and potentially dangerous operation and should be attempted only by those persons who are thoroughly familiar with the handling of this substance. Sampling should never be attempted within a building and the outdoor location selected must be such that the escapipg gas or liquid will not endanger the operator or others. Use of a suitable gas mask is recommended.

ANNEX A

(*Clause* 3.2)

METHODS OF TEST FOR CHLORINE GAS

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) 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 CHLORINE GAS

Two methods are prescribed for the determination of chlorine, namely, mercurimetric and Orsat method. The Orsat method shall be regarded as the routine method.

A-2.1 Mercurimetric Method

A-2.1.1 *Apparatus*

A-2.1.1.1 The apparatus shall consist of the following parts assembled as shown in Fig. 1.

A-2.1.1.2 *Sample cylinder A*

A-2.1.1.3 *Filtering tube B* — Consisting of a 150 × 6 mm iron nipple.

A-2.1.1.4 *Needle valve C*

A-2.1.1.5 *Gas collecting bomb* D — Of 250- capacity and provided with a calibrated neck, made of 5 mm bore thick-walled tubing. The capacity of the calibrated neck shall be 2.5 ml, subdivided to 0.02 ml with every 0.1 ml numbered. Graduation shall extend at least half way around and every fifth mark shall be completed around the circumference. The glass burette shall be calibrated with water at room temperature.

A-2.1.1.6 *Levelling bottle E*

A-2.1.1.7 *Two-way stop cock F, G and L* — Of 2 mm bore.

A-2.1.1.8 *Bubbler H* — Containing concentrated sulphuric acid.

A-2.1.1.9 *Empty catch bottle J*

A-2.1.1.10 *Pail K* — Containing milk of lime or caustic soda solution.

NOTE — The apparatus and mercury used shall be as dry as possible.

A-2.1.2 *Procedure*

A-2.1.2.1 Connect the dry levelling bottle *E* to one of the connections of stopcock *F* by means of a piece of heavy rubber tubing, 60 cm long. Connect the rubber tubing to both the levelling bottle *E* and gas collecting bomb *D.* Fill the levelling bottle with clean, dry mercury. Adjust stopcock *F* to allow the levelling bottle *E* to communicate with the gas collecting bomb through the connecting tubing and let a little mercury into the gas collecting bomb, then close the stopcock *F.* Now raise and lower the levelling bottle *E* several times, collapsing the tubing toward the levelling bottle *E* to completely exclude air bubbles. Turn stopcock *F* to allow all the mercury in the gas collecting bomb *D* to run into a beaker.

A-2.1.2.2 The apparatus shall be connected as shown in Fig. 1. The needle valve *C* shall be closed and stopcocks on the gas collected bomb *D* shall be opened to allow the gas to flow through the bomb to the bubbler *H.* The needle valve *C* shall then be opened slowly to allow the gas sample to pass through the apparatus at a rate of about 50 bubbles per minute as seen in *H.* Take this sample preferably for a period of 25 to 30 min. Care shall be taken to avoid liquid chlorine remaining in the filling bottle *B.* When the gas collecting bomb *D* is completely purged and filled with the sample, close the needle valve *C* and scopcocks *F, G* and *L* in order so that a slight positive pressure is left in the bomb *D.*

During sampling, the chlorine gas emerging from *H* should be passed through an empty catch bottle and finally absorbed in caustic soda solution or milk of lime to avoid fouling of the atmosphere.

A-2.1.3 Simply turning of stopcock *G* quickly two or three times will be adequate to bring down the pressure inside the bulb *D* to normal.

A-2.1.4 Open the stopcock *F* to connect *D* to *E* and allow the mercury to flow into the bomb *D* shaking *D* constantly during this operation.

A-2.1.4.1 Under no condition allow the mercury to flow into the bomb *D* without constantly shaking *D,* as an explosion is apt to occur if this precaution is not followed. Shake the apparatus gently but constantly, so that a fresh surface of mercury is exposed to the chlorine. Avoid shaking so vigorously that the mercury splashed into the graduated portion of the burette until the bulb is completely filled.

A-2.1.4.2 When the bulb is completely filled with mercury, attach the apparatus again securely to the ring stand. Support the levelling bottle on the same ring stand to which the bomb is attached, and adjust the level of the mercury in the bottle to about the same height as in the graduated portion of the bomb. Keep the surface of the mercury in the bomb moving slightly by pressing the connecting rubber tubing between the thumb and fingers. If no further change in height of the

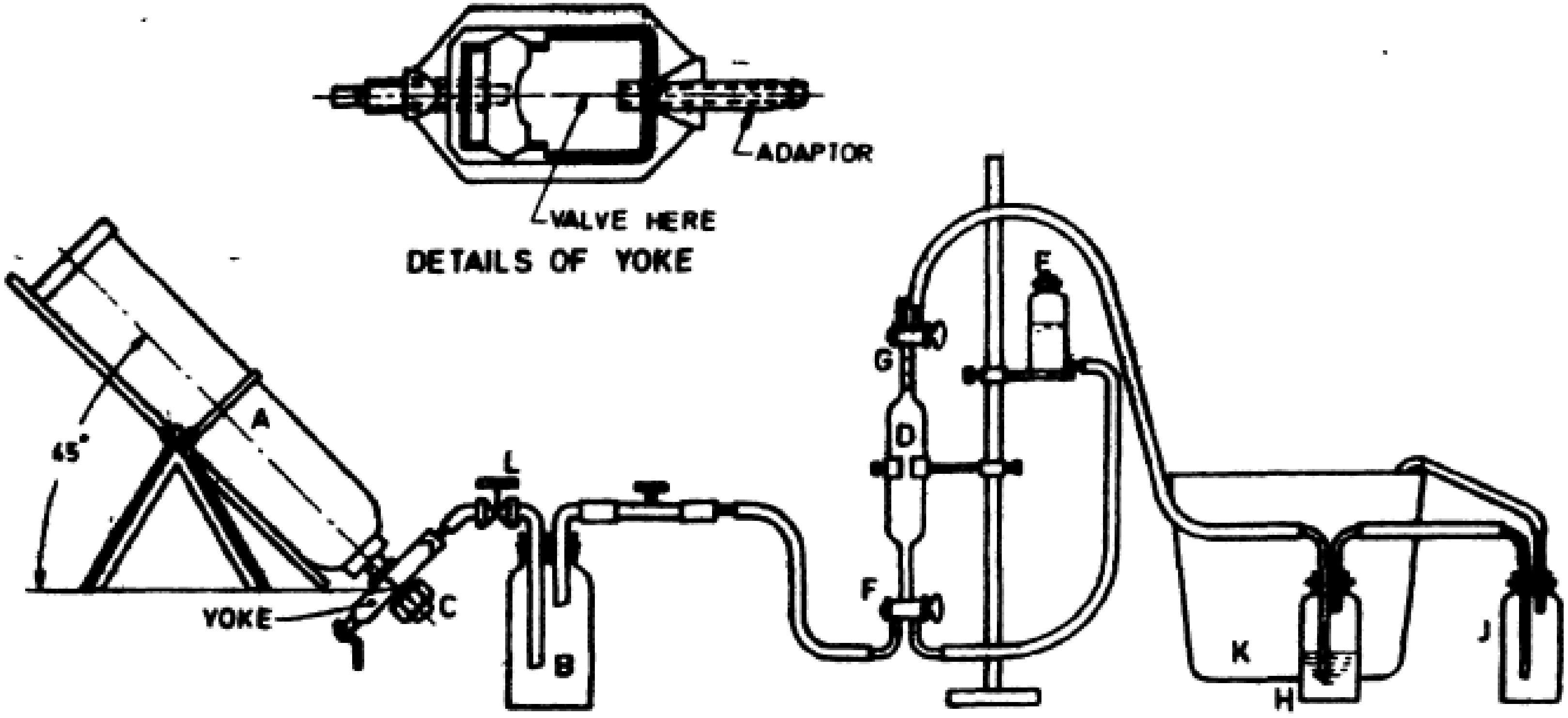


FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF CHLORINE

mercury is noted after 5 min, bring the liquid surfaces in the levelling bottle and bomb to the same level and read off accurately the volume of residual gas (V_1 ml). *This volume V_1 ml represents the total non-absorbed gas.*

NOTE — The time required to absorb the chlorine gas in the bulb is 30 to 40 min, and in the graduated portion about 25 min.

A-2.1.5 *Calculation*

$$\text{Chlorine gas, percent by volume} = \frac{100\,(V_2 - V_1)}{V_2}$$

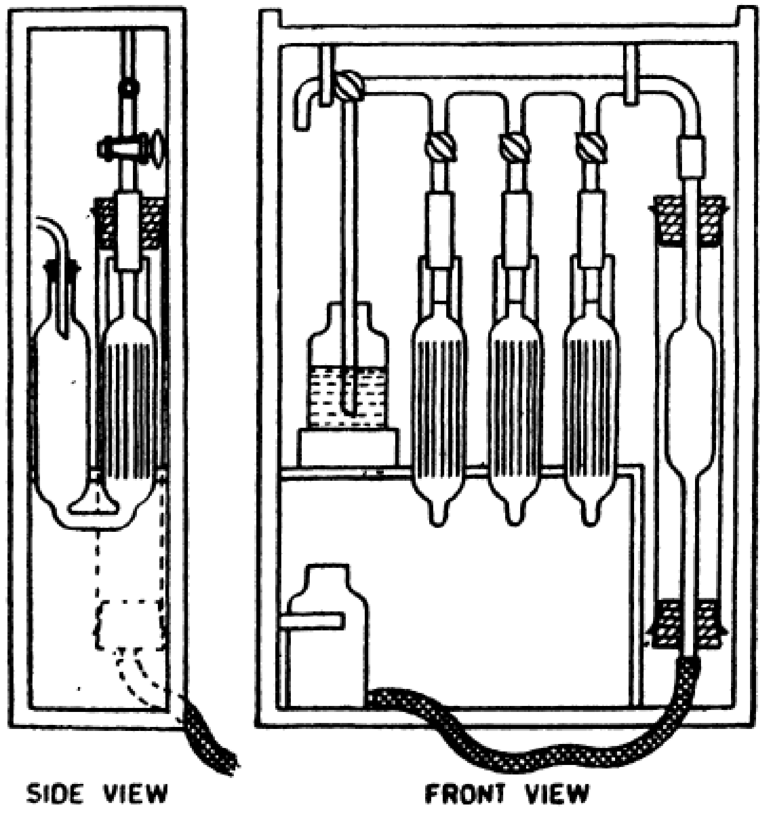
Where,

V_2 = Capacity in ml, of the bomb, and

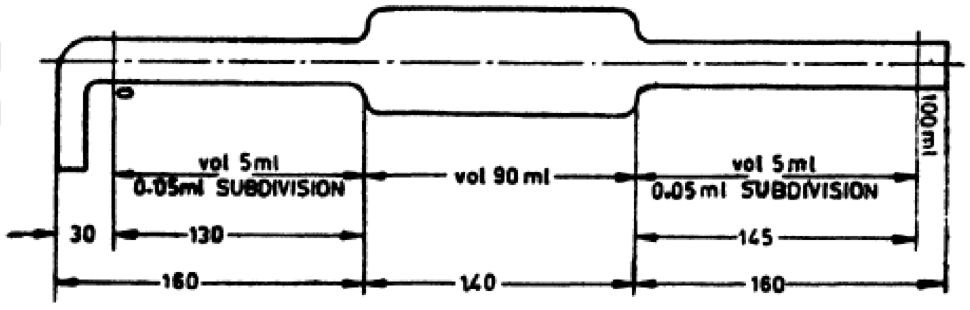
V_1 = Volume in ml, of the residual gas.

A-2.2 Orsat Method

A-2.2.1 *Apparatus* — Orsat apparatus (*see* Fig. 2) with a special burette as in Fig. 2B. The top portion of the burette is drawn into a capillary and calibrated from 95 to 100 ml in subdivision of 0.05 ml.



2A Orsat Apparatus



All dimensions in millimetres.

2B Special Burette

FIG. 2 ORSAT APPARATUS FOR GAS ANALYSIS

A-2.2.2 *Reagents*

A-2.2.2.1 *Potassium iodide solution* — 10 percent (*m/v*).

A-2.2.2.2 *Acidic brine* — Saturated sodium chloride brine made acidic with hydrochloric acid.

A-2.2.2.3 *Caustic soda solution* — 30 percent (*m/v*).

A-2.2.3 *Procedure*

A-2.2.3.1 Only the absorption bulb *A* is used for this analysis. This is filled up to the mark with 10 percent potassium iodide solution.

A-2.2.3.2 Connect the Orsat apparatus to the sampling point and make sure that there is no air leakage. Open the 3-way stopcock so as to connect the gas inlet tube to the Orsat burette. By lowering the levelling bottle containing the acidic brine, the chlorine gas is sucked into the burette. When the burette is full of gas, turn the 3-way stopcock so as to connect the burette to the purge line. By raising the levelling bottle, purge out the gas in the burette. The purged gas is absorbed in the caustic soda solution contained in the bottle into which the purge line is dipped. Repeat this operation 4-5 times so as to thoroughly purge the burette and the connecting tubing. Collect 100 ml chlorine gas in the burette and adjust the level of brine in the burette to the zero mark by means of the levelling bottle. Close the 3-way stopcock. Take care that the level of the brine in the burette and levelling bottle is the same while adjusting zero.

A-2.2.3.3 Open the two-way stopcock at the top of the absorption bulb containing potassium iodide so that the bulb is now connected to the burette. Open the 3-way stopcock. By raising and lowering the levelling bottle, pass the gas in the burette into the absorption bulb. The chlorine in the gas is absorbed by the potassium iodide and iodine is liberated in the bulb. There will be a reduction in volume of the gas as more and more chlorine gas is absorbed.

A-2.2.4 *Calculation*

When all the chlorine is absorbed, equalize the level of brine in the burette and levelling bottle and read off the volume of gas absorbed. This gives the percentage of chlorine in the gas.

A-3 DETERMINATION OF MERCURY

A-3.1 Procedure

During the test for determination of moisture content as per Annex B, chlorine under test is absorbed in sodium hydroxide solution, which is finally weighed as per method described in **B-1.4.2**; label it as "solution - X". Weigh accurately 10 g of above solution X in a 100 ml beaker, neutralize it with dilute hydrochloric acid (1 : 1) to *p*H 6-8 and then transfer it quantitatively to a 100 ml volumetric flask with distilled water. Make up to mark with distilled water and label it as "sample solution - S.1".

Weigh accurately 10 g of sodium hydroxide solution used for absorption of chlorine, neutralize it with dilute hydrochloric acid (1 : 1) to *p*H 6-8 and then transfer it quantitatively to a 100 ml volumetric flask with distilled water. Make it up to mark with distilled water and label it as "blank solution - B.1".

Follow the method for determination of mercury as described in **A-10** of IS 299 till **A-10.5** and calculations to be followed as mentioned below.

Transfer 5 ml of sample solution - S.1 by pipette into a 250 ml volumetric flask, add about 50 ml distilled water. Add drop wise four percent(w/v) potassium permanganate till purple colour remains, shake well and add 2-3 drops of concentrated nitric acid, mix thoroughly and keep it for 5-10 min. Decolourize the solution by adding drop wise hydroxylamine hydrochloride solution 10 percent (w/v), shake well and make up to the mark with distilled water, mark it as "sample solution S.2". Prepare a fresh blank (*see* **A-10.5.1** of IS 299), stir it for 5 min, stop the stirring, take reading for the mercury content, if any in the blank. Transfer 5 ml of sample solution S.2 to the reaction vessel containing blank and determine the concentration of the mercury with the help of the standard graph (*see* **A-10.4.3** of IS 299). Prepare a blank solution using all reagents in same quantities except sample solution S.1 in a 250 ml volumetric flask while using blank solution B.1 in place of sample solution S.1. Make up to the mark with distilled water, shake well and mark it as "blank solution B.2". Transfer 5 ml of this blank solution B.2 to reaction vessel containing blank in the same way as sample, due to presence of any mercury content in the reagents which shall be used in analysis. Calculate mercury content from standard graph and subtract mercury content of this blank solution from sample result, and take for calculation of mercury content in sample.

A-3.2 Calculation

Mercury (as Hg) in liquid chlorine, ppm =

$$\frac{50\,000 \times M \times Y}{X}$$

Where,

M = total mass of sodium hydroxide solution after absorption of chlorine in the test for determination of moisture, in grams;

X = total weight of chlorine absorbed in the test for determination of moisture, in grams; and

Y = mg of mercury as Hg, in 5 ml of sample solution S.2.

A-4 DETERMINATION OF ARSENIC

A-4.1 Procedure

From total mass (M g) of sodium hydroxide solution after absorption of chlorine (X g) in the test for determination of moisture, weigh accurately M/X grams of this solution to nearest 10 mg; neutralize it with 1:1 sulphuric acid and then make it to 50.0 g by careful addition of distilled water. Mark it as Solution –L.

Take 1.0 g of this solution-L , and carry out the test for Arsenic as prescribed in IS 2088, using for comparison of a stain obtained with 0.005 mg of arsenic trioxide.

A-4.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length and intensity of the stain is not greater than that produced in the control sample.

A-4.3 Referee Method

Follow the method by Atomic Absorption Spectrophotometer (AAS) as prescribed in IS 11124. Alternately, it can be also analyzed by ICP-OES.

A-5 DETERMINATION OF LEAD

Follow the method as prescribed in **A-8** of IS 299 but weighing 5.0 g of "Solution-L" prepared in above test for arsenic (*see* **A-4.1**) and in **A-8.3** of IS 299 instead of 1.0 g and comparing with control test using 2 ml of standard lead solution. Alternatively, it can also be analyzed by ICP-OES.

A-5.1 Referee Method

Follow the method by Atomic Absorption Spectrophotometer (AAS) as prescribed in IS 12074.

ANNEX B

(*Clause* 3.2)

B-1 DETERMINATION OF MOISTURE CONTENT

B-1.1 General

This moisture (volatile water) volatilizes at the same time as the chlorine during the gasification of liquid chlorine for industrial use. The water is absorbed by passing the gasified sample into tared absorber containing phosphorus pentoxide or magnesium perchlorate. The chlorine leaving the absorber is passed through a tared bottle containing sodium hydroxide solution. The absorbers and the bottle containing sodium hydroxide solution are reweighed and the mass of water and that of the chlorine sample are obtained by difference.

B-1.2 Reagents

B-1.2.1 *Phosphorus Pentoxide Powder*

B-1.2.2 *Magnesium Perchlorate* — Granules in the size range 3 to 5 mm (to avoid loss of material during the absorption of water). Magnesium perchlorate should not be used if the presence of organic matter is suspected.

B-1.2.3 *Sodium Hydroxide* — Approximately 5 N.

B-1.2.4 *Iodized Starch Indicator* — 0.2 percent starch solution containing 40 g of potassium iodide and 4 g of sodium hydrogen carbonate per litre.

B-1.2.5 *Acetone*

B-1.2.6 *Chlorine-Resistant Grease* — Greases based on fluorinated or chlorofluorinated products are suitable.

B-1.3 Apparatus

The apparatus is shown in Fig. 3.

B-1.4 Procedure

When the absorber tubes are freshly charged, condition them by passing gaseous chlorine at a rate of 25 litres per hour for 2 h in order to allow the chlorine to react with certain impurities in the absorbent. Unless this is done, the first result will be in excess of the correct value. Close the chlorine feed. Disconnect the absorber tubes and weigh them separately, to the nearest 0.1 mg.

B-1.4.1 Turn on the chlorine supply so as to allow the filtered and subsequently vapourized chlorine flow towards absorber. Allow about 100 litres of gas passed into the absorption. Weigh the bottle of sodium hydroxide after the determination and calculate the mass of chlorine absorbed.

B-1.4.2 During the determination, which lasts about 4 h, it is essential that all chlorine gas passed through the tubes be totally absorbed in caustic soda. Such is the case if the iodized starch indicator does not develop an intense blue colour. When sufficient chlorine has been passed, turn off the chlorine supply and purge for 10 min at the rate of 25 litres per hour with dry air or nitrogen heated to about 80°C. Disconnect the absorber tubes and reweigh them separately to the nearest 0.1 mg.

B-1.5 Calculation

$$\text{Moisture, parts per million} = \frac{M_1 \times 10^6}{M + M_1}$$

Where,

M_1 = mass in g, of the weighed water in the absorber tubes; and

M = mass in g, of chlorine passed through the tubes.

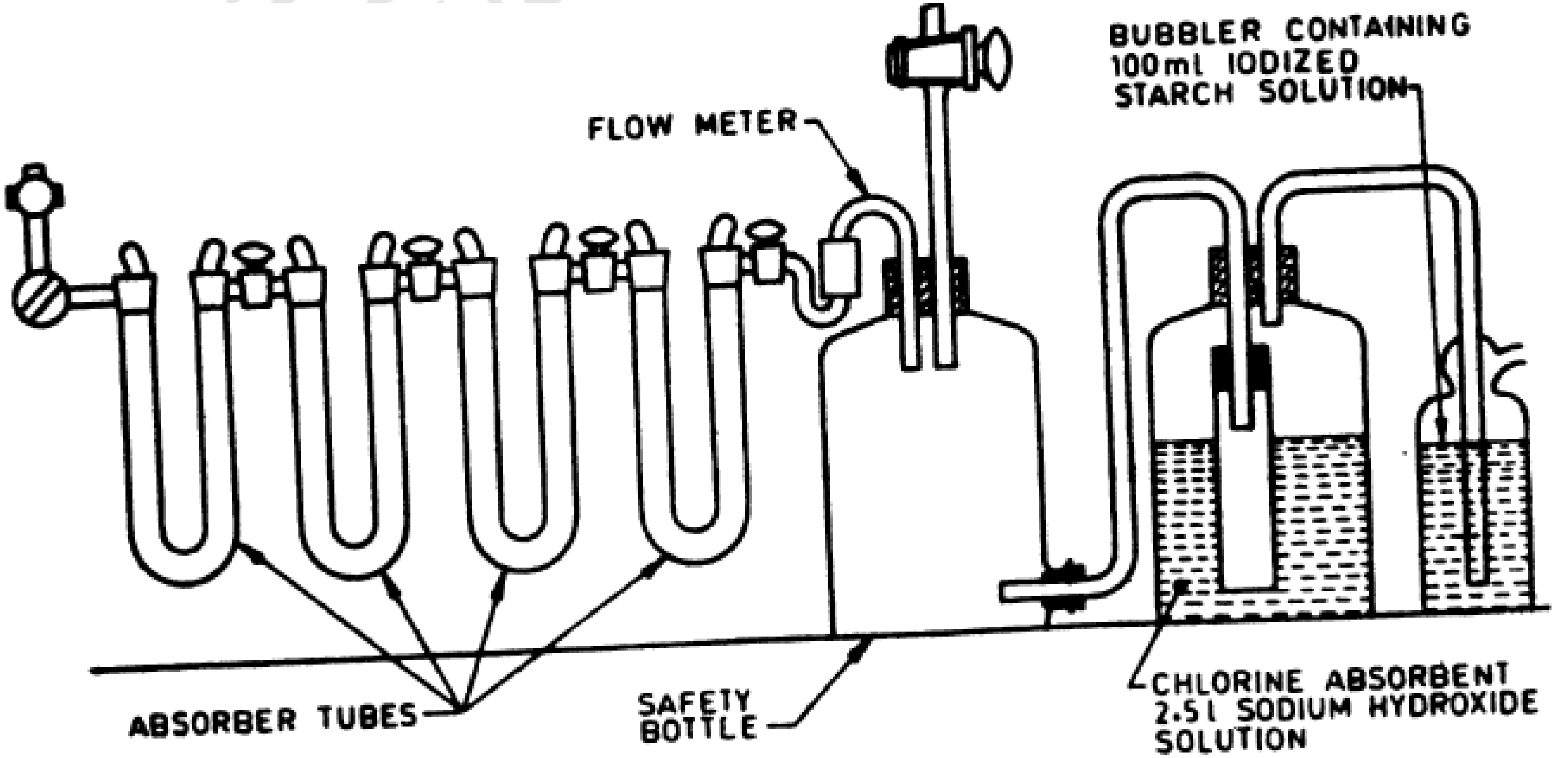


FIG. 3 APPARATUS FOR DETERMINATION OF MOISTURE

ANNEX C

(*Foreword*)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

Organization	*Representative(s)*
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VOICE, Delhi	Shri H. Wadhwa Shri K. C. Chaudhary (*Alternate*)
In Personal Capacity	Shri D. K. Jain
Directorate General, BIS	Shri U. K. Das, Scientist 'E' and Head (CHD) [Representing Director General (*Ex-officio*)]

Member Secretary

Sagar Singh
Scientist 'C' (CHD), BIS

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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