

X

इंटरनेट

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार" Mazdoor Kisan Shakti Sangathan "The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ" Jawaharlal Nehru "Step Out From the Old to the New"

मानक

IS 11305 (1985): Thionyl Chloride [CHD 1: Inorganic Chemicals]





51111111

Made Available By Public, Resource, Org



"ज्ञान से एक नये भारत का निर्माण″ Satyanarayan Gangaram Pitroda "Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता Bhartrhari-Nītiśatakam "Knowledge is such a treasure which cannot be stolen"





BLANK PAGE



PROTECTED BY COPYRIGHT

Indian Standard SPECIFICATION FOR THIONYL CHLORIDE

UDC 661-231



C Copyright 1986

INDIAN STANDARDS INSTITUTION MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 116002

May 1986

Indian Standard

SPECIFICATION FOR THIONYL CHLORIDE

Acids, Alkalis and Halides Sectional Committee, CDC 56

Chairman	Representing		
PROF M. M. TAQUI KHAN	Central Salt and Marine Chemicals Research Institute (CSIR), Bhavnagar		
Members			
DR G. D. BHAT (Alternate to Prof M. M. Taqui Khan) Shri B. K. Anand	Punjab National Fertilizers and Chemicals Ltd, Chandigarh		
SHRI R. C. JAIN (Alternate) SHRI M. L. BHAMBANI SHRI T. K. DUTTA (Alternate)	National Test House, Calcutta		
SHRI R. C. BHATTACHARYA	Directorate General of Technical Development, New Delhi		
Shri R. N. Das	Bengal Chemicals and Pharmaceuticals Ltd,		
SHRI P. N. HALDAR (Alternate)		
SHRI M. V. DESAI	Atul Products Ltd, Atul		
DR J. M. TUREL (Alternate)	,		
SHRI B. L. GUPTA	Kothari Industrial Corporation Ltd, Madras		
DR H. V. R. IENGAR	E. I. DParry (India) Ltd, Madras		
DR V. C. NAIR (Alternate)			
SHRI N. V. KAMBLE	Saurashtra Chemicals, Porbandar		
SHRI L. K. MALAVIYA (Alteri	nate)		
Shri V. B. Khanna	Directorate General of Supplies and Disposals, New Delhi		
SHRI N. K. KANSAL (Alternate	?)		
Shri Manmohan Singh	Indian Paper Mills Association, Calcutta		
SHRI N. K. GUPTA (Alternate)			
DR B. S. NAGAR	Indian Dyestuff Industries Ltd, Bombay		
SHRI N. R. FADNIS (Alternate)			
DR V. N. NIGAM	Ministry of Defence (DGI)		
SHRI K. B. DUTTA (Alternate)			
DR ANIL PANDIT	Deepak Nitrite Ltd, Vadodara		
SHRI J. T. VORA (Alternate)	O to the The State Of Tell Velladare		
SHRIM. D. PATEL	Gujarat State Fertilizers Co Ltd, Vadodara		
SHRI K, N. CHOKSHI (Alternat	e)		

(Continued on page 2)

© Copyright 1986

INDIAN STANDARDS INSTITUTION

This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

(Continued from page 1)

Members	Representing			
SHRIG. C. DUTTA ROY	Shriram Foods & Fertiliser Industries, New Delhi			
SHRI S. K. NANDA (Alternate) SHRI N. K. SACHAR	Gujarat Alkalis & Chemicals Ltd, Vadodara			
SHRIJ. B. SHARMA (Automate) SHRIV. S. SAXENA	Ballarpur Industries Ltd, New Delhi			
SHRI J. S. TALWAR (Alternate) DR N. SEN) Indian Soap & Toiletries Makers' Association, Bombay			
DR N. N. SHARMA SHRI K. H. PAREKH (Alternate	Tata Chemicals Ltd, Bombay			
SHRI S. K. SUBBAROYAN DR A. N. BHAT (Alternate)	Hindustan Lever Ltd, Bombay			
SHRI P. SUBRAMANIAN SHRI B. REVANNA (Alternate)	Salt Commissioner, Jaipur			
DR M. S. VAIDYA DR A. S. VAIDYANATHAN	The Dharamsi Morarji Chemical Co Ltd, Bombay Standard Alkali (Chemicals Division), Bombay			
SHRI T. P. VENKATRAMAN (A	lternate)			
DR K. M. VERMA SHRI SATISH CHANDER,	Projects and Development India Ltd, Sindri Director General, ISI (Ex-officio Member)			
Director (Chem)	Secretary			
	2007 00 million			

SHRI M. BAKSHI GUPTA Deputy Director (Chem), ISI

Chlorine and its Inorganic Products Subcommittee, CDC 56:3

Convener

SHRI V. S. SAXENA

Ballarpur Industries Ltd, New Delhi

Members

SHRI J. S. TALWAR (Alternate	0
Shri V. S. Saxena)	United Pheenhorus Put I to Bombay
SHRIP MITRA (Alternate)	United Phospholds I vt Ltd, Dombay
SHRI R. C. BHATTACHARYA	Directorate General of Technical Development, New Delhi
SHRI R. C. SHARMA (Alternate)
Shri S. N. Bhattacharya	Tata Chemicals Ltd, Bombay
SHRI D. N. V. RAO (Alternate)	
SHRI L. N. CHOWDHARY	Indian Paper Mills Association, Calcutta
SHRI M. V. DESAI	Atul Products Ltd, Atul
DR J. M. TUREL (Alternate)	
SHRI S. C. GUDIMANI	Gudimani Enterprises (India), New Delhi
Shri O. P. Jejani	Kahoria Chemicals and Industries Ltd, Calcutta
SHRI P. K. BANERJEE (Alierna	te)

(Continued on page 17)

Indian Standard

SPECIFICATION FOR THIONYL CHLORIDE

$\mathbf{0}.\quad \mathbf{FOREWORD}$

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 August 1985, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Thionyl chloride finds wide use in dyestuff industries, pesticides, and as surface active agent. Use of the chemical in vulcanizing rubber, in wood hardening and in the extraction of gold from its ores is gradually increasing.

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

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for thionyl chloride.

2. GRADES

2.1 There shall be two grades of the material, namely:

- a) Technical, and
- b) Pure.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of pale-yellow liquid.

*Rules for rounding off numerical values (revised).

3.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given col 5 of the table.

SL	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST,
INO.		Technical	Pure	APPENDIX A
(1)	(2)	(3)	(4)	(5)
i)	Boiling range (95 percent distillation by volume) (5 to 95 ml)	75 to 78°C	75 to 77°C	A-2
ii)	Sulphur chloride (as S ₂ Cl ₂ and SCl ₂) percent by mass, Max	0.1	0.20	A-3
iii)	Iron (as Fe), ppm, Max	20	10	A-4
iv)	Sulphuryl chloride (SO ₂ Cl ₂), percent by mass, Max	2.0	1.0	A-5
v)	Assay (as SOCl ₂) percent by mass, <i>Min</i>	98.0	98.5	A-6
vi)	Relative density at 30°C	1.62	1.62	A-7
vii)	Refractive index (n ²⁰ D)	West Per	1.517	A-8
viii)	Sulphur dioxide (as SO ₂), percent by mass, Max	0.20	0•20	A- 9

TABLE 1 REQUIREMENTS FOR THIONYL CHLORIDE

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in clean galvanized iron containers.

4.2 Marking — Each container shall bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer and/or his recognized trade-mark, if any;
- c) Net mass of the contents;
- d) Date of manufacture;
- e) Batch number; and
- f) Warning Corrosive or vesicant to skin, avoid contact with eyes, skin and clothes.

4.2.1 The containers may also be marked with the ISI Certification Mark.

Norz — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.2)

METHODS OF TEST FOR THIONYL CHLORIDE

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. BOILING RANGE

A-2.1 Apparatus

A-2.1.1 Round Bottom Flask - 200 ml.

A-2.1.2 Thermometer — 0 to 110°C.

A-2.1.3 Leibig Condenser, Band with Long Stem

A-2.1.4 Measuring Cylinder — 100-ml.

A-2.1.5 Glass Beads

*Specification for water for-general laboratory use (second revision).

A-2.2 Procedure — Take 100 ml sample in a 200-ml round bottomed flask. Heat it to distil, then note down the temperature of very first drop that is starting temperature. Then take a reading after 5 ml and another after 95 ml keeping a rate of 120 to 125 drops/min.

A-2.3 Result

Boiling range = $X^{\circ}C/5$ ml to $\Upsilon^{\circ}C/95$ ml

where

X =temperature at 5 ml, and

 Υ = temperature at 95 ml.

A-3. SULPHUR CHLORIDE

A-3.0 Outline of the Method — It is necessary to remove both iron and chloride before analysis. This is achieved by carrying out the preliminary distillation of thionyl chloride after mixing it with tetrachloroethane.

A-3.1 Reagents

A-3.1.1 Potassium Iodide Solution — 10 percent m/v.

A-3.1.2 Standard Bromate Bromide Solution - 0.1 N.

A-3.1.3 Standard Sodium Thiosulphate Solution - 0.1 N.

A-3.1.4 Concentrated Hydrochloric Acid - See IS : 265-1976*.

A-3.1.5 Tetrachloroethane — Boiling point 140°C.

A-3.1.6 Sulphur Powder

A-3.1.7 Iodine Solution - 0.1 N.

A-3.1.8 Methyl Orange Indicator Solution — 0.5 percent m/v.

A-3.1.9 Starch Indicator — 1 percent (m/v).

A-3.2 Procedure

A-3.2.1 Preliminary Distillation with Titrachloroethane — The material 80 ml is diluted to 100 ml with tetrachloroethane (b. p. 147°C) and distilled at a pressure of 120 mm mercury in a ground glass joined apparatus. To prevent loss of material during distillation the air leak is kept as small as possible and an efficient dry cold ether reflux condenser is attached to the receiver, the vacuum being applied at the top of this condenser. Distillation is continued just to dryness (preserve the residue for iron estimation as in A-4.3), and the condenser is drained for 10 minutes. The distillate is diluted to 100 ml. Exactly

^{*}Specification for hydrochloric acid (second revision).

⁶

50 ml of this solution is removed and reserved for determination (A-3.2.3) below. The remaining 50 ml is again diluted to 100 ml with tetrachloroethane.

A-3.2.2 Hydrolysis and Titration with Bromate-Bromide Solution — Exactly 2 ml of the above solution (constantly from a 2 ml pipette) is hydrolysed with water (50 ml) at 0°C by shaking vigorously for 2 minutes. The hydrolisate is warmed to 50°C and a vigorous stream of carbon dioxide or nitrogen passed through for 10 minutes. The solution cooled to room temperature and iodine solution (about 01 N) added dropwise to the first yellow colour, subsequent slow fading being ignored. The iodine oxidises any sulphur dioxide not removed by gassing. Concentrated hydrochloric acid (30 ml) and methyl orange solution (2 drops) are added and the solution is titrated with 0°1 N bromate-bromide solution (10 ml) is immediately added and the solution diluted to 200 ml. The liberated iodine is back titrated with 0°1 N thiosulphate, solution (t_2 ml) volume of 0°1 N bromate-bromide used, $T_1 = (t_1 - t_2)$ ml.

A-3.2.3 The Sulphur Reflux — The second portion of the distillate (diluted) from **A-3.2.1**, 50 ml is very gently refluxed with 2 g sulphur powder (usually about 30 minutes). The solution is cooled and diluted to exactly 100 ml with tetrachloroethane. Exactly 2 ml of the diluted solution is hydrolysed and examined as in **A-3.2.2** above. Titrations t_3 ml of 0.1 N bromate-bromide and t_4 ml of 0.1 N thiosulphate volume of 0.1 N bromate-bromide used, $T_2 = (t_3 - t_4)$ ml.

A-3.3 Calculation

Sulphur chloride (as
$$S_2Cl_2$$
),
pecrent by mass = 3.21 $\frac{(7T_1 - 4T_2)}{M}$

and

Sulphur chloride (as SCl₂),
percent by mass =
$$\frac{17 \cdot 2 (T_2 - T_1)}{M}$$

where

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

A-4. DETERMINATION OF IRON

A-4.0 Outline of the Method — Thioglycolic acid gives a red purple colour with iron solution in alkaline medium.

A-4.1 Apparatus

A-4.1.1 Nessler Cylinder --- 50-ml capacity (see IS: 4161-1967*).

*Specification for Nessler cylinders.

A-4.2 Reagents

A-4.2.1 Thioglycolic Acid

A-4.2.2 Citric Acid Solution — 20 percent (m/v).

A-4.2.3 Dilute Ammonia Hydroxide Solution - 10 percent.

A-4.2.4 Standard Iron Solution — Dissolve 0.702 g of ferrous ammonium sulphate hexahydrate in 10 ml of dilute sulphuric acid (10 percent v/v). Make the volume up to 1 000 ml. Take 10 ml of this solution and dilute to 100 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-4.3 Procedure — Add approximately 50 ml water and 10 ml hydrochloric acid in bottom residue of the vacuum distillation flask from determination of sulphur chloride (**A-3.2.1**). Warm it, cool and make 500 ml. Take two 50-ml Nessler cylinders. In both the Nessler cylinders, take 2 ml citric acid, 5 to 6 drops thioglycolic acid, and 10 ml of 10 percent ammonium hydroxide solution. In one Nessler cylinder add 10 ml sample solution, red-purple coloured produced in alkaline medium. Bring same colour intensity in Nessler cylinder by standard solution of iron by drop-wise addition. Note the volume of standard solution consumed.

A-4.4 Calculation

Iron (as Fe), ppm =
$$\frac{38.58 \times R}{n}$$

where

R = volume in ml of standard iron solution consumed, and

v = volume in ml taken for the test.

A-4.5 Alternatively, the iron content can also be estimated by measuring the transmittancy of the coloured complex at 530 nm with the help of a spectrophotometer.

A-5. DETERMINATION OF SULPHURYL CHLORIDE

A-5.0 Outline of the Method — On hydrolysis sulphuryl chloride gives hydrochloric acid and sulphuric acid. Sulphate is estimated gravimetrically as barium sulphate.

A-5.1 Reagents

A-5.1.1 Concentrated Hydrochloric Acid - See IS : 265-1976*.

*Specification for hydrochloric acid (second revision).

A-5.1.2 Barrium Chloride — 10 percent (m/v).

A-5.2 Procedure — Weigh accurately about 15 g of sample in stoppered weighing bottle. Transfer it into 500-ml beaker containing ice-cold water under fuming hood. After reaction is over, add 20 ml of hydrochloric acid. Boil for five minutes, cool to room temperature and filter. Add 50 ml of 10 percent barium chloride solution. Digest on water bath for half an hour.

Filter through Whatman No. 42 filter paper. Wash the precipitate with hot water till free from chloride. Incinerate the filter paper on a weighed silica crucible at 800°C. Cool and weigh as barium sulphate.

A-5.3 Calculation

Sulphuryl chloride (as SO_2Cl_2), $=\frac{57.84 M_2}{M_1}$

where

 M_2 = mass in g of barium sulphate, and M_1 = mass in g of sample taken for the test.

A-6. ASSAY

A-6.0 Outline of the Method — Thionyl chloride is hydrolised by aqueous sodium hydroxide to give sodium sulphite. This is determined by iodimetry.

A-6.1 Reagents

A-6.1.1 Sodium Hydroxide — 10 percent (m/v).

A-6.1.2 Standard Sodium Thiosulphate - 0.1 N.

A-6.1.3 Standard Iodine Solution - 0.1 N.

A-6.1.4 Starch Solution — 1 percent (m/v).

A-6.1.5 Dilute Sulphuric Acid

A-6.2 Procedure — Weigh accurately about 0.8 to 1.0 g of sample in weighing bottle (2-ml capacity). Drop into stoppered glass flask containing cold (below 5°C) 50 ml sodium hydroxide. Keep it for 10 minutes shake well. Again keep it for 10 to 15 minutes to come to room temprature. Dilute it to 250 ml in volumetric flask. Pipette out 50 ml solution into a 500 ml glass stoppered flask containing 50 ml iodine and 20 ml dilute sulphuric acid. Shake well and titrate it against 01 N sodium thiosulphate solution using starch as indicator. Carry out the blank.

A-6.3 Calculation

Thionyl chloride (as SOCl₂), percent by mass = $\frac{(B-A) \times N \times 59.5}{M}$

where

B = volume required to titrate the blank,

A = volume required to titrate the sample,

 $\mathcal{N} =$ normality of sodium thiosulphate, and

M = mass of sample taken for test.

A-7. DETERMINATION OF RELATIVE DENSITY

A-7.1 Apparatus

A-7.1.1 Hydrometer

A-7.1.2 Thermometer

A-7.2 Procedure — Pour the material to be tested into the clean hydrometer jar the diameter of which shall be at least 2.5 cm greater than the diameter of the hydrometer used. Remove all air bubbles, that might have formed in the liquid. Keep the jar in a vertical position and in a water bath maintained at 30° C. When the sample in the jar attains the temperature of the bath, that is 30° C, lower the hydrometer gently into the material. When it has settled, depress it to about two scale divisions into the liquid. Keep the unimmersed portion of the stem dry, as any unnecessary liquid on the stem will change the effective weight of the instrument and affect the reading obtained. Allow the hydrometer to become stationery. Remove all air bubbles that might have formed during lowering of the hydrometer. Read the point on the hydrometer scale, to which the sample rises, with the eye placed at the principal surface of the material. This reading gives the relative density of the material under test.

A-8. REFRACTIVE INDEX

A-8.0 Outline of the Method — The principal of the instrument is the observation of the critical angle for total reflection between glass of high refractive index and substance to be examined.

A-8.1 Apparatus

A-8.1.1 Abbe Refractometer

A-8.2 Procedure — To determine the refractive index of a liquid at 20°C, circulate water at 20°C from a thermostat jacket through two prisms until the temperature on the thermometer has remained steady

for at least 10 minutes. Separate the prism jacket by opening the clamp and move the index arm if necessary until the face of the prism is horizontal. Wipe the water with a paper tissue. Place a drop or two of the liquid on the ground glass prism face and then clamp it to the upper polished prism. Focus the cross-wire of the telescope by rotating the eveniece and then adjust the mirror so as to give good illumination from suitably placed frosted electric lamp. By means of the rack and pinion controlling the arm at the side of apparatus, turn the prism box until the field of view becomes partly light and partly dark. When white light is used, the edge of light band will show a coloured fringe. By means of milled screw at the base of telescope, rotate the dispersion compensator until the coloured fringe disappears and the light band is bounded by a sharp edge coincides with the intersection of the crosswires in the telescope and read off directly refractive index for the D-Sodium line on the divided arc by means of the magnifying lens. Immediately the determination has been completed, wipe off the inorganic liquid with a paper tissue and clean the prism surface with a tissue soaked in acetone.

A-9. DETERMINATION OF SULPHUR DIOXIDE

A-9.0 Outline of the Method — In the hot state, the thionyl chloride is blasted with nitrogen. Sulphur dioxide, a little quantity of thionyl chloride and eventually the sulphur chloride present are expelled and they enter a gas washing bottle.

A-9.1 Reagents

A-9.1.1 Nitrogen Gas - Pure.

A-9.1.2 Standard Sodium Hydroxide Solution - 1 N.

A-9.1.3 Methyl Orange Indicator

A-9.1.4 Dilute Nitric Acid - 20 percent (v/v).

A-9.1.5 Dilute Hydrogen Peroxide — 3 percent (v/v).

A-9.1.6 Dilute Sodium Bicarbonate Solution - 10 percent (v/v).

A-9.1.7 Potassium Chromate Indicator

A-9.1.8 Standard Silver Nitrate Solution -- 0.1 N.

A-9.1.9 Standard Iodine Solution — 0.1 N.

A-9.2 Apparatus

A-9.2.1 Three-Necked Flask — As shown in Fig. 1.

A-9.2.2 Glass Wash Bottles - 250-ml.



FIG. 1 APPARATUS FOR THE DETERMINATION OF SULPHUR DIOXIDE

A-9.3 Produre — About 50 ml of the sample is placed in three-necked flask with a gas inlet pipe. The nitrogen gas blasted out passes through the intensive cooler, functioning as a reflux cooler, into two serially fitted glass bottles for gas washing; each containing 100 ml of 1 N sodium hydroxide solution (soda lye). These bottles are cooled with water. The flask containing thionyl chloride is heated till obtaining weak reflux. The sulphur dioxide is then blasted out with a weak stream of nitrogen from the round flask into the receiver (absorption bulb).

A-9.3.1 The blasting continues for about 30 minutes, as a result the absorption solution in the first wash bottle has to neutralize directly. In order to check an over acidification few drops of methyl orange can be added into second wash bottle. Subsequently, both the bottles transferred and washed in a 500-ml measuring flask, and dilute up to the mark with water.

Additional sulphur dioxide due to decomposition of thionyl chloride must be calculated with separate hydrochloric acid estimation. The total value of sulphur dioxide is then corrected with the share from the thionyl chloride.

A-9.4 Calculation

A-9.4.1 Hydrochloric Acid Estimation — The solution (50 ml) to be analysed is measured out from the measuring flask with a burette into a conical flask and the sulphur dioxide is oxidised with about 10 ml of 3 percent hydrogen peroxide and then make slightly acidic with dilute nitric acid.

After the solution is neutralized with sodium bicarbonate, 12 drops of potassium chromate indicator are added and the solution is titrated against 0.1 N silver nitrate solution. The indicator is added before the titration.

In case of consumption of 'a' ml of 0'l N silver nitrate solution, till a sudden change takes place the following relation is true:

'a' ml of 0.1 N silver nitrate \times 0.036 47 = (g) hydrochloric acid. or, (g) hydrochloric acid \times 0.877 5 = (g) sulphur dioxide from thionyl chloride = A (say).

A-9.4.2 Sulphur Dioxide Estimation — In a conical flask, about 100 ml of water is poured and to it, 20 ml of 0.1 N iodine solution is added from the burette and the solution is acidified with about 5 ml of sulphuric acid diluted in a ratio of 1:3.

The sample solution to be analyzed is filled in a burette and the pre-treated iodine solution is titrated till its decolouration. 20 ml of 0.1 N iodine solution corresponds to 64 mg of sulphur dioxide with a consumption of 'a' ml of the sample solution.

 $\frac{500 \times 0.0064}{a} = (g) \text{ sulphur dioxide total}$ (in 500 ml solution) = B (say)

Sulphur dioxide (as SO₂), percent by mass $= \frac{(A - B) \times 100}{M}$

where

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

APPENDIX B

(Clause 5.1)

SAMPLING OF THIONYL CHLORIDE

B-1. SCALE OF SAMPLING

B-1.1 Lot — All the containers in a single consignment of the material of the same grade and drawn from a single batch of manufacture shall constitute a lot. If the consignment is found to consist of different batches of packing, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-1.2 For ascertaining the conformity of the material in the lot to the requirements of the specification, samples shall be tested separately from each lot. The number of containers to be selected at random from the lot shall depend upon the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF	CONTAINERS TO BE SELECTED
No. of Containers in a Lot	SAMPLE SIZE
(\mathcal{N})	(n)
(1)	(2)
Up to 50	4
51 " 100	5
101 '' _300	7
301 "1 000	10
1 001 and above	15

B-1.2.1 In order to ensure randomness of selection, random number tables shall be used. For guidance and use of the random number tables reference may be made to IS : 4905-1968*. In case these tables are not available, the following procedure is recommended for use:

Starting from any one container, count them as $1, 2, \ldots, r$, where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

*Methods for random sampling.

B-2. PREPARATION OF TEST SAMPLES

B-2.1 From each of the containers selected according to **B-1.2.1**, a representative portion of the material, sufficient to make triplicate determination for all the requirements given in **3**, shall be drawn.

B-2.2 Out of these portions a small but approximately equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient to carry out triplicate determination for all the characteristics tested on the composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

B-2.3 The remaining portions of the material from each container shall be divided into three equal parts each forming an individual sample, one set of individual samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-2.4 All the individual and composite samples shall be transferred to separate containers. These containers shall be sealed air-tight and labelled with full indentification particulars.

B-2.5 The referee sample consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

B-3. NUMBER OF TESTS

B-3.1 Tests for the determination of sulphuryl chloride and assay (Table 1) of the material shall be conducted on each of the individual test samples and the test results shall be recorded separately for different test samples.

B-3.2 Tests for the determination of all other characteristics given in Table 1 shall be carried out on the composite test sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 From the results recorded according to **B-3.1**, the mean (\vec{X}) and range (R) of test results for any characteristic shall be computed as follows:

Mean (\bar{X}) = $\frac{\text{Sum of test results 1, 2, ..., n}}{\text{Number of test results, n}}$

Range (R) = difference between the maximum and minimum of the test results.

B-4.2 In the case of samples of size 10 or more, every 5 test results shall be grouped together and the range of each group shall be determined. Average range (R) shall then be computed from the group ranges as given below:

Average range (R) = $\frac{\text{Sum of group ranges}}{\text{Number of groups}}$

B-4.3 The lot shall be declared as conforming to the requirements of the specification for different characteristics if the corresponding criteria for conformity given below are complied:

Characteristics	Criteria for Conformity
a) Suphuryl chloride	$(\bar{X} + 0.6R^*)$ shall be less than or equal to the relevant limit prescribed in Table 1.
b) Assay	($\overline{X} - 0.6R^*$) shall be greater than or equal to the relevant limit prescribed in Table 1.
c) Other characteristics	Tests results on the composite test sample shall meet the corresponding requirement given in Table 1.

^{*}When the sample size is 10 or more, an average range (\overline{R}) (see B-4.2) shall be used in place of range (R).

-

(Continued from page 2)

Members	Representing
SHRI M. P. MISTRY	Excel Industries Ltd, Bombay
SHRID. B. MEHTA (Alternate)	
SHRI B. M. NAIK	Indian Dyestuff Industries Ltd, Bombay
SHRI D. P. PATEL (Alternate)	
SHRI K. M. SHAH	The Millowners' Association, Bombay
Shri V. D. Sharma	Shriram Foods & Fertiliser Industries, New Delhi
SHRI P. K. BANERJEE (Alterna	nte)
Shri M. R. Somayaji	The Mettur Chemical & Industrial Corporation Ltd,
	Mettur Dam
SHRI D. SESHASAYEE (Alternat	te)
SHRI S. P. SRIVASTAVA	Gujarat Alkalis & Chemicals Ltd, Vadodara
SHRI N. K. SACHAR (Alternate	

DR A. S. VAIDYANATHAN Standard Alkali, Chemicals Division, Bombay SHRI T. P. VENKATRAMAN (Alternate)

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	Symbol	
Length	metre	m	
Mass	kilogram	kg	
Time	second	3	
Electric current	ampere	Α	
Thermodynamic temperature	kelvin	К	
Luminous intensity	candela	cd	
Amount of substance	mole	mol	
Supplementary Units			
QUANTITY	Unit	SYMBOL	÷
Plane angle	radian	rad	
Solid angle	steradian	sr	
Derived Units			
QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1 N = 1 kg.m/s^2$
Energy	joule	J	$J = 1 N_{\bullet}m$
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V}_{\bullet} s$
Flux density	tesla	т	$1 T = 1 Wb/m^2$
Frequency	hertz	H_{Z}	$1 \text{ Hz} = 1 \text{ c/s} (\text{s}^{-1})$
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	v	1 V = 1 W/A
Pressure, stress	pascal	Pa	$1 Pa = 1 N/m^3$

AMENDMENT NO. 1 MARCH 1995 TO IS 11305 : 1985 SPECIFICATION FOR THIONYL CHLORIDE

[*Page* 4, *Table* 1, *Sl No.* (ii), *col* 3] — Substitute '0.5' for '0.1'. [*Page* 4, *Table* 1, *Sl No.* (ii), *col* 4] — Substitute '0.1' for '0.50'.

(CHD 002)

Reprography Unit, BIS, New Delhi, India