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(Reaffirmed 2005)

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Indian Standard
**SPECIFICATION FOR
ACETIC ANHYDRIDE**
(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5

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Indian Standard

SPECIFICATION FOR ACETIC ANHYDRIDE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 22 April 1988, after the draft finalized by the Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1958 and it was subsequently amended in 1979. This first revision has been prepared as a result of review of the standard by the Committee in the light of present day requirements of the product. In this revision, the requirements for chlorides, sulphates and reducing substance have been modified, additional requirement for colour, insoluble matter, non-volatile residue and iron content have been introduced to meet the requirements of various consumers.

0.3 Acetic anhydride is largely used in the manufacture of various grades of cellulose acetate. It is also used in the manufacture of various dye-stuffs, perfumery and pharmaceutical products.

0.4 Taking into consideration the views of the producers, consumers and technologists, the Sectional Committee responsible for the prepara-

tion of this standard felt that it should be related to the existing trade and manufacturing practices followed in the country in this field. Furthermore, due weightage had to be given to the need for international co-ordination between standards prevailing in different countries of the world. This consideration led the committee to draw freely upon BS 2068 : 1970* and consulted other overseas standards on the subject.

0.5 This standard also requires reference to Red Tariff No. 17 of 1954 (with any additions or alterations made thereafter) issued by the Indian Railway Conference Association, New Delhi.

0.6 This standard is intended chiefly to cover the technical provisions relating to the supply of the material, and it does not include all the necessary provisions of a contract.

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

*Specification for acetic anhydride.

†Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and methods of test for acetic anhydride.

2. SAMPLING

2.1 Representative samples of the material shall be drawn as prescribed in Appendix A.

3. REQUIREMENTS

3.1 Description — The material shall be a clear liquid, free from matter in suspension and having a pungent odour. It shall consist essentially of acetic anhydride.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix B. References to the relevant clauses of Appendix B are given in col 4 of Table 1.

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in suitable containers as agreed between the purchaser and the supplier, and subject to the provisions of Red Tariff No. 17 of 1954 issued by the Indian Railway Conference Association with any alterations or additions made thereafter.

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TABLE 1 REQUIREMENTS FOR ACETIC ANHYDRIDE

(Clause 3.2)

Sl No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN APPENDIX B)
(1)	(2)	(3)	(4)
i)	Distillation range	The difference between initial boiling point (IBP) and dry point (DP) shall not exceed 4.0°C including 139.5°C (temperature being corrected for a pressure of 760 mm Hg)	B-2
ii)	Ash, percent by mass, <i>Max</i>	0.02	B-3
iii)	Colour (Hazen), <i>Max</i>	20	B-4 or IS : 8768-1978*
iv)	Insoluble matter	To pass the test	B-5
v)	Non-volatile residue, percent by mass, <i>Max</i>	0.01	B-6
vi)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.001	B-7
vii)	Sulphates (as SO ₄), percent by mass, <i>Max</i>	0.003	B-8
viii)	Iron, percent by mass, <i>Max</i>	0.000 5	B-9
ix)	Reducing substances, percent by mass, <i>Max</i>	0.015	B-10
x)	Acetic anhydride content, percent by mass, <i>Min</i>	98	B-11

*Method of measurement of colour in liquid chemical products in Hazen units.

4.2 Marking

4.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

4.2.2 Each container shall be marked with the following:

- a) Name of the material;
- b) Manufacturer's name, initials or trade-mark, if any;
- c) Mass, net or gross; and
- d) Year of manufacture.

4.2.3 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard 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 BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

A P P E N D I X A

(Clause 2.1)

SAMPLING OF ACETIC ANHYDRIDE

A-1. GENERAL REQUIREMENTS

A-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

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

A-1.2 The sampling instrument shall be clean and dry, when used.

A-1.3 To draw a representative sample, the contents of each container selected for sampling

shall be mixed as thoroughly as possible by suitable means.

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

A-1.5 The samples shall be placed in suitable clean, dry and air-tight glass containers.

A-1.6 The sample containers shall be of such a size that an ullage of at least 10 percent is left after pouring in the sample.

A-1.7 Each sample container shall be sealed air-tight with a stopper-after filling, and marked with full details of sampling and the particulars given under 4.2.

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

A-2. SCALE OF SAMPLING

A-2.1 Lot — All 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.

A-2.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, samples shall be tested from each lot separately. The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
(1)	(2)
Up to 15	3
16 to 50	5
51 to 100	7
101 and above	10

A-2.2.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed.

A-3. TEST SAMPLES AND REFEREE SAMPLES

A-3.1 Preparation of Composite Sample — To prepare composite samples for distillation range, ash, chlorides, sulphates and reducing substances, draw with an appropriate sampling instrument, small portions of the material from different parts of each container in the test sample selected for sampling (see A-2.2) and freshly opened. Keep the samples from different containers separate. Mix equal portions from different containers selected for sampling and obtain a composite sample of not more than 1 litre. Divide this composite sample into three test samples. Transfer them immediately to thoroughly dried bottles and seal the bottles air-tight with glass stoppers. Label as 'Sample for general tests' and give all the particulars of sampling specified under A-1.7. Send one test sample to the purchaser and one to the supplier. Reserve the third test sample, bearing the seals of the purchaser and the supplier, as referee

*Methods for random sampling.

sample to be kept at a place agreed between the purchaser and the supplier, and used in case of dispute between the purchaser and the supplier.

A-3.2 Preparation of Sets of Samples for Acetic Anhydride Content — For the determination of acetic anhydride content, a set of samples representing each of the containers in the test sample shall constitute the test sample to represent the lot. Draw the material in three portions, each about 50 ml from each of the separate sample containers collected as prescribed under A-3.1, into thoroughly dried glass bottles, thus obtaining three sets of test samples. Seal the bottles air-tight. Send one set of test samples to the purchaser and one to the supplier. Reserve the third set of test samples, bearing the seals of the purchaser and the supplier, as referee set to be kept at a place agreed between the purchaser and the supplier, and used in case of dispute between the purchaser and the supplier.

A-4. NUMBER OF TESTS

A-4.1 General Tests — Tests for distillation range, ash, chlorides, sulphates and reducing substances shall be carried on the composite sample to represent the lot (see A-3.1).

A-4.2 Test for Acetic Anhydride Content — The determination of acetic anhydride content shall be carried out individually on the test samples representing the lot (see A-3.2).

A-5. CRITERION FOR ACCEPTANCE

A-5.1 The material in any lot shall be accepted as conforming to the specification, if the results of testing the corresponding test samples satisfy the requirements of A-5.2 and A-5.3. Otherwise, the lot shall be rejected.

A-5.2 When tested for requirements other than acetic anhydride content, the test results shall satisfy the requirements given in Table 1 [items (i) to (ix)].

A-5.3 From the results of testing acetic anhydride content (see A-4.2), the mean and range shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{sum of the test results}}{\text{number of test results so added, and}}$$

$$\text{Range } (R) = \text{difference between the maximum and minimum values of the test results.}$$

The range (R) so obtained shall be multiplied by 0.6 and the expression ($\bar{X} - 0.6 R$) shall be calculated. The value of this expression shall be not less than 98 percent by mass. The following table is illustrative:

Test Result	Mean	Range	Criterion for Acceptance
1, 2, ... n	\bar{X}	R	$\bar{X} - 0.6 R$ shall be not less than 98

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APPENDIX B

(Clause 3.2)

ANALYSIS OF ACETIC ANHYDRIDE

B-1. QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals shall be employed in tests, and distilled water shall be used where the use of water as a reagent is intended.

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

B-2. DETERMINATION OF DISTILLATION RANGE

B-2.1 Apparatus

B-2.1.1 Distillation Flask — of the shape and dimension given in Fig. 1. Fix the flask in the vertical position by means of a clamp at the extreme upper end of the neck.

B-2.1.2 Thermometer — so fitted in the flask that the bottom of the capillary is in level with the

lower edge of the side-tube joint and the immersion mark is in level with the top of the cork.

The recommended dimension, tolerances and graduations of the thermometer are as follows:

Range	: 98 to 152°C.
Graduation at each	: 0.2°C
Longer lines at each	: 1°C
Fractional figuring at each	: 2 and 10°C
Fully figured at each	: 100°C
Maximum error	: 0.4°C
Maximum error in an interval	: 0.4/10°C

The thermometer shall bear a certificate of the National Physical Laboratory of India or of any other institution authorized by the Government of India to issue such a certificate.

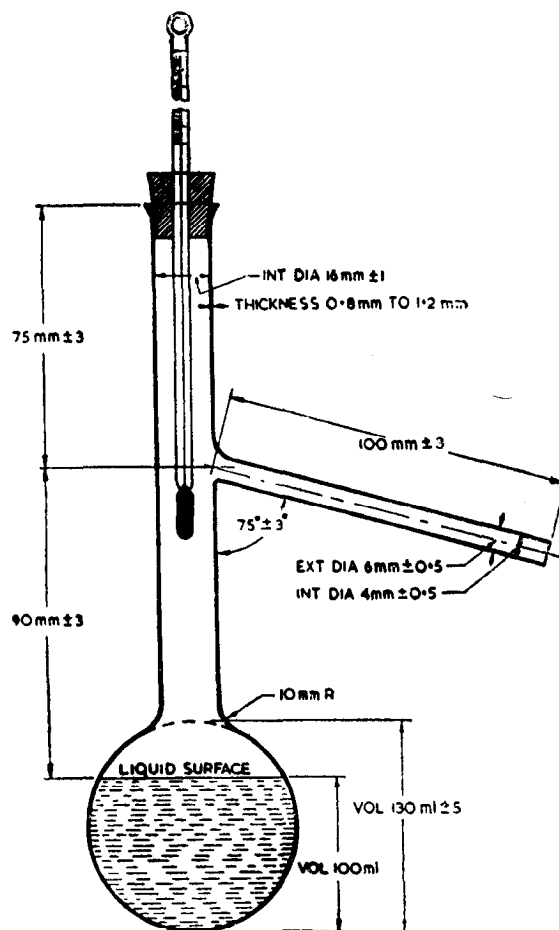


FIG. 1 DISTILLATION FLASK

B-2.1.3 Rectangular Draught Screen — made of 22 gauge sheet metal, with the dimensions shown in Fig. 2 open at the top and bottom, and fitted with hard asbestos board with a central hole of 100 mm diameter. It shall comply with the following requirements.

B-2.1.3.1 In each of the two narrow sides of the draught screen, there shall be two circular holes, each 25 mm in diameter, and in each of the 4 sides of the draught screen, there shall be 3 holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the position as shown in Fig. 2. The diameter of each of the holes, centrally situated in the longer side, shall be 25 mm and of the remaining ten holes 12.5 mm. At the middle of each of the wider sides, a vertical slot with the dimensions shown in Fig. 2 shall be cut downwards from the top of the screen.

B-2.1.3.2 A sheet of hard asbestos (6 mm in thickness) and having a central circular hole 50 mm in diameter shall be supported horizontally in the screen, and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

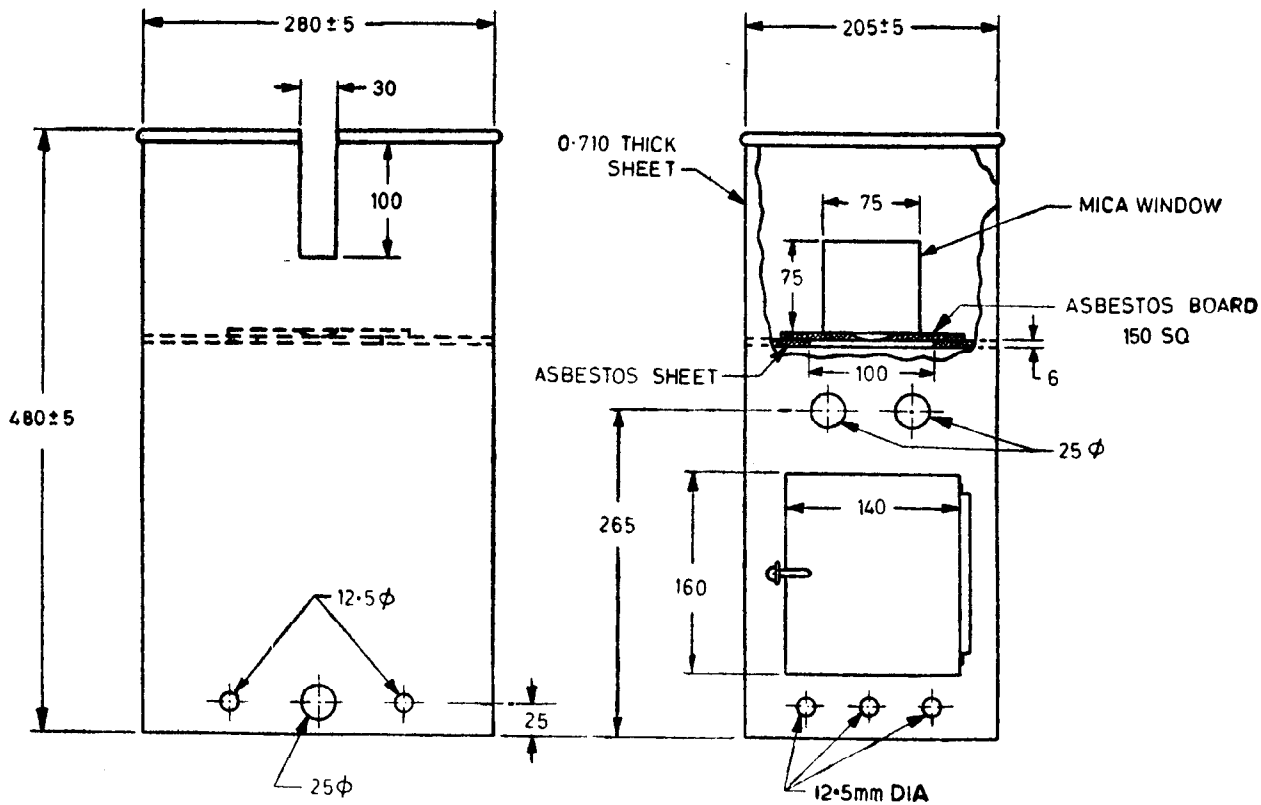
B-2.1.3.3 In one of the narrow sides of the screen, a door shall be provided having the position and dimensions shown in Fig. 2. In each of the narrow sides of the screen, a mica window shall be placed centrally with window on a level with the top of the asbestos shelf. The dimensions and positions of windows are shown in Fig. 2.

B-2.1.4 Liebig Condenser — with bent end, made of good quality resistance glass with a wall thickness of 1 to 1.5 mm and conforming to the shape and dimensions given in Fig. 3.

B-2.1.4.1 Alternatively — The bent portion may be substituted by an adaptor fitted externally such that the distillate does not come in contact with the cork.

B-2.1.5 Crow Receiver or Graduated Cylinder — 100 ml, with one millilitre marks running half-way round the circumference, five millilitre marks running three quarters-way round, and ten millilitre marks running all round the circumference and numbered (see Fig. 4).

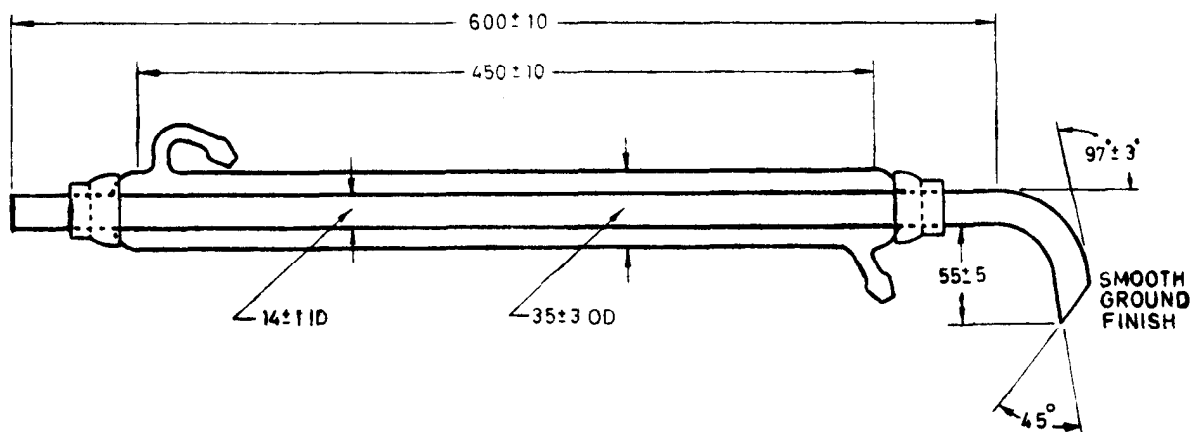
B-2.1.6 Bunsen Burner — giving an entirely non-emoky flame and provided with an adjustable screw clip with the help of which the flame can be lowered or raised according to requirements.



All dimensions in millimetres.

FIG. 2 RECTANGULAR DRAUGHT SCREEN

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All dimensions in millimetres.
FIG. 3 LIEBIG CONDENSER

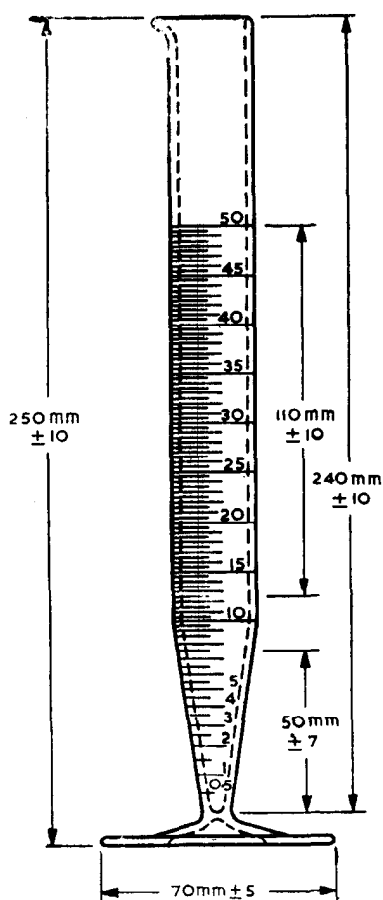


FIG. 4 100-ml CROW RECEIVER

B-2.2 Procedure

B-2.2.1 Assemble the apparatus as shown in Fig. 5. Measure 100 ml of the material at laboratory temperature into the graduated cylinder and transfer it to the distillation flask, the contents of the graduated cylinder being allowed to drain for 15 seconds into the flask. Add a fragment of a porous material (about 2 mm cube)

or some other suitable inert material to avoid bumping, connect the flask to the condenser and insert the thermometer. Pass an adequate supply of cold water through the condenser. To receive the distillate, use the graduated cylinder in which the sample was measured, without rinsing or drying. Heat the flask slowly especially after the ebullition has begun in order that the mercury column of the thermometer may become

fully expanded before the first drop of distillate drops into the receiver, care being taken that the total period of this preliminary heating is not less than 12 nor longer than 17 minutes. Place the receiver so that the condensate flows down its inside walls, continue the distillation at the rate of 4 to 5 ml per minute by suitable regulation of flame and take the readings of volume at the specified temperature.

B-3. DETERMINATION OF ASH

B-3.1 Procedure — Evaporate about 10 g of the material, accurately weighed, to dryness in a weighed platinum or silica basin, and gently ignite the residue until all carbonaceous matter has disappeared. Cool in a desiccator and weigh.

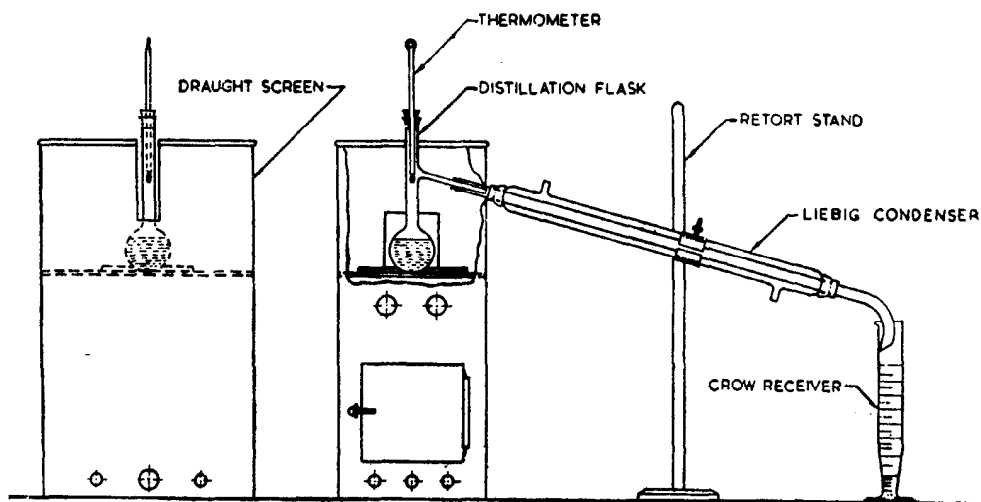


FIG. 5 ASSEMBLY OF APPARATUS

B-2.2.2 Read the volume of distillate in the receiver when the thermometer indicates each of the corrected specified distillation temperatures. The temperatures on the thermometer scale shall be corrected as specified under **B-2.3** before commencing the distillation. The difference between the columns so recorded is the percentage by volume distilling between the specified temperatures at 760 mm pressure.

B-2.3 Correction of Thermometer Reading

B-2.3.1 Error of Scale — In all thermometer readings, make the corrections as indicated on the certificate of the instrument.

B-2.3.2 Correction for Barometric Pressure — If the barometric pressure prevailing during the determination is normal, namely, 760 mm of mercury, no correction need be applied to the specified temperature and the thermometer scale as corrected under **B-2.3.1** shall be used as such. If the prevailing barometric pressure deviates from 760 mm, the specified temperature shall be corrected with the correction factor given below.

B-2.3.2.1 For every 10 mm above 760 mm, subtract 0.5°C to the specified temperatures and for every 10 mm below 760 mm add 0.5°C to the specified temperatures. These corrections shall be applied for any prevailing barometric pressure in the above mentioned proportion and are valid down to a pressure of 700 mm of mercury.

B-3.2 Calculation

$$\text{Ash, percent by mass} = \frac{100 m}{M}$$

where

m = mass in g of the ignited residue, and
 M = mass in g of the material taken for the test.

B-4. TEST FOR THE DETERMINATION OF COLOUR

B-4.0 Outline of the Method — The colour of the material is compared with that of the colour standard and expressed in terms of Hazen colour units. The Hazen colour is defined as the colour of an aqueous solution containing 1 ppm of platinum in the form of chloroplatinic acid and 2 ppm of cobaltous chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$).

B-4.1 Apparatus

B-4.1.1 Nessler Cylinders — two, 100 ml capacity (see IS : 4161-1967*).

B-4.1.2 One-Mark Graduated Flasks — 250 and 500 ml capacities (see IS : 915-1975†).

*Specification for Nessler cylinders.

†Specification for one-mark volumetric flasks (first revision).

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B-4.2 Reagents

B-4.2.1 Cobaltous Chloride Hexahydrate

B-4.2.2 Hydrochloric Acid — relative density 1.18 (see IS : 265-1976*).

B-4.2.3 Chloroplatinic Acid — Dissolve 250 mg of platinum in a small quantity of *aqua regia* contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of the hydrochloric acid and again evaporate to dryness. Repeat this operation twice more.

B-4.3 Preparation of Colour Standards —

Dissolve 0.50 g of the cobaltous chloride hexahydrate and entire quantity of the chloroplatinic acid (see B-4.2.3) in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500-ml graduated flask. Dilute with water to the mark.

B-4.3.1 Pipette 10 ml of this solution (see B-4.3) into a 250-ml graduated flask. Dilute with water up to the graduation mark. This diluted solution is equivalent to 20 Hazen units and should always be freshly prepared.

B-4.4 Procedure

B-4.4.1 Fill one of the Nessler cylinders to the mark with material to be tested, and the other with the colour standard using a white background. Compare the colours.

B-5. TEST FOR INSOLUBLE MATTER

B-5.1 Procedure

B-5.1.1 Add 50 g of the sample in small portions to 150 ml of hot distilled water, stirring to dissolve each portion separately, and finally dilute to 250 ml.

B-5.1.2 The material shall be taken to have passed the test if the resultant solution is clear and colourless.

B-6. DETERMINATION OF NON-VOLATILE RESIDUE

B-6.1 Procedure

B-6.1.1 From a graduated measuring cylinder introduce 28 ml of the sample (M_1 g) into a clean 125 ml platinum, silica or borosilicate glass evaporating dish, which has previously been heated to constant mass at a temperature of 105 to 110°C and cooled in a desiccator. Evaporate on a steam bath to dryness and place the dish in an oven at $100 \pm 2^\circ\text{C}$. Cool in a desiccator and weigh the residue to the nearest 0.1 mg (M_2 g).

*Specification for hydrochloric acid (second revision).

B-6.2 Calculation

Non-volatile residue,

$$\text{percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_1 = mass in g of the sample, and

M_2 = mass in g of the residue.

B-7. DETERMINATION OF CHLORIDES (AS Cl)

B-7.1 Procedure

B-7.1.1 Take 5 ml of the solution obtained in B-5.1, and add 45 ml of distilled water, 1 ml of 5 N nitric acid and 1 ml of silver nitrate solution (42 g/litre) and mix.

B-7.1.2 The prescribed limit shall be taken as not having been exceeded if the solution shows no opalescence.

B-8. DETERMINATION OF SULPHATES (AS SO₄)

B-8.1 Apparatus

B-8.1.1 Nessler Tubes

B-8.2 Reagents

B-8.2.1 Dilute Hydrochloric Acid — approximately 5 N.

B-8.2.2 Barium Chloride Solution — Dissolve 10 g of barium chloride crystals in water and make up to 100 ml.

B-8.2.3 Standard Sulphuric Acid — 0.01 N.

B-8.3 Procedure

B-8.3.1 Add 4 g of the material to 40 ml of water. When dissolved, add 1 ml of dilute hydrochloric acid and transfer to a Nessler tube. Dilute to 50 ml with distilled water, add 1 ml of barium chloride solution, mix thoroughly and set aside for five minutes. Carry out a control test in the other Nessler tube using 0.25 ml of standard sulphuric acid in place of the material. Compare the turbidities produced in the two tubes after five minutes.

B-8.3.2 The prescribed limit shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

B-9. DETERMINATION OF IRON

B-9.1 Procedure

B-9.1.1 To the residue in B-6.1, add 3 ml of 5 N hydrochloric acid and 10 ml water. Dissolve the residue by heating to boiling. Cool and make up to 30 ml with distilled water. In a

stoppered cylinder, take 10 ml of this solution, add 1 drop of 0.1 N potassium permanganate solution and mix. Add 5 ml ammonium thiocyanate solution (570 g/litre) and 10 ml of a mixture of equal volumes of amyl alcohol and amyl acetate. Shake vigorously and allow to stand.

B-9.1.2 Take 1 ml of standard iron solution (1 ml = 0.05 mg Fe) (0.4325 g ammoniac ferric sulphate A.R. + 50 ml concentrated nitric acid diluted to 1 litre with distilled water). Add 1 ml of 5 N hydrochloric acid and dilute to 10 ml with distilled water in a stoppered cylinder. Add 1 drop of 0.1 N potassium permanganate solution and follow the same procedure as for the sample.

B-9.1.3 The prescribed limit shall be taken as not having been exceeded if the red colour of the upper layer of the sample is not darker than that of the standard.

B-10. DETERMINATION OF REDUCING SUBSTANCES

B-10.1 Procedure

B-10.1.1 Dissolve 2 g sample in 10 ml of distilled water, add 0.4 ml of 0.1 N potassium permanganate and mix well.

B-10.1.2 The material shall be taken to have passed the limit if the pink shade of the solution persists at least for 5 minutes.

B-11. DETERMINATION OF ACETIC ANHYDRIDE CONTENT

B-11.0 Outline of the Method — The acetic anhydride content in the sample is determined by subtracting the acidity determined with aniline from the total acidity determined by hydrolysing the sample with caustic soda solution and back titrating with strong acid.

B-11.1 Apparatus

B-11.1.1 Weighing Pipette — capacity about 5 ml.

B-11.1.2 Flasks — stoppered, two of capacity 500 ml and one of capacity 100 ml.

B-11.2 Reagents

B-11.2.1 Standard Sodium Hydroxide Solution — approximately 1 N.

B-11.2.2 Standard Hydrochloric Acid — approximately 1 N.

B-11.2.3 Rectified Spirit — 95 percent by volume (see IS : 323-1959*). Neutralize to phenolphthalein indicator.

B-11.2.4 Phenolphthalein Indicator — Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit (see IS : 323-1959*) and add the standard sodium hydroxide solution (see B-11.2.1) until the colour turns faint pink.

B-11.2.5 Benzene — dry, freshly distilled.

B-11.2.6 Aniline — dry, freshly distilled.

B-11.3 Procedure

B-11.3.1 Dissolve about 2 g of the material accurately weighed by means of a weighing pipette in 50.0 ml of the standard sodium hydroxide solution contained in a stoppered flask, and allow to stand for one hour. Titrate the excess of alkali with the standard hydrochloric acid using 0.5 ml of phenolphthalein indicator. Calculate the number of millilitres of normal sodium hydroxide solution required (A) for 1 g of the acetic anhydride.

B-11.3.2 Dissolve about 2 g of the acetic anhydride, accurately weighed, by means of a weighing pipette in 20 ml of benzene in a stoppered flask, cool in ice and add a cold solution of 10 ml of aniline in 20 ml of benzene. Allow the mixture to stand for one hour in ice. Add 200 ml of rectified spirit and 50.0 ml of standard sodium hydroxide solution. Titrate the excess of alkali with standard hydrochloric acid using 0.5 ml of phenolphthalein indicator. From the number of millilitres of normal sodium hydroxide solution consumed, subtract a blank determined on the reagents, using the method described above, but without adding the sample. Calculate the number of millilitres of normal sodium hydroxide required (B) for 1 g of the acetic anhydride.

B-11.4 Calculation

Acetic anhydride,

$$\text{percent by mass} = 10.21 (A - B).$$

*Specification for rectified spirit (revised).

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 3 31 01 31 3 31 13 75

Telegrams : Manaksanstha
(Common to all Offices)

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