भारतीय मानक
Indian Standard

IS 695 : 2020

एसिटिक एसिड — विशिष्टि

(चौथा पुनरीक्षण)

Acetic Acid — Specification

(Fourth Revision)

ICS 71.080.40

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

May 2020 **Price Group 7**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Acetic acid is used in the manufacture of cellulose acetate, vinyl acetate and in textile industry for dyeing purposes. It is also used in pharmacy and in the manufacture of rubber balloons, sanitary goads and surgical gloves, from rubber latex. Various chemical compounds like acetates of lead, sodium and calcium are produced from acetic acid. Some of the popular thinners, such as butyl and ethyl acetates used in the paint industry, are also prepared from acetic acid.

This standard was first issued in 1955, covering the requirements of three grades of acetic acid, namely pure, pharmaceutical and technical. With increase in production and on the basis of experience gained in use of the earlier version of this standard, it was considered imperative to upgrade the minimum quality requirements stipulated for different grades of the material and accordingly the standard was revised in 1967. The revised standard covered three grades of acetic acid, namely, technical, pure and analytical reagent. The requirements of the pure grade covered those for pharmaceutical grade acetic acid given in the Indian Pharmacopoeia, second edition.

A new sampling scheme and criteria for conformity was also included. This standard was again revised in 1975 in which limits for formic acid and acetaldehyde for AR grade were introduced. Requirement for colour was also modified. The third revision was undertaken in which requirements for water for AR grade and relative density were included.

The requirements for acetaldehyde for AR grade were modified.

The Indian standards listed in Annex C which are referred in this standard are informative in nature. All standards are subject to revision, and parties to agreement based on standard are encouraged to apply the most recent editions of the standards.

The Committee, responsible for revision of the standard, has observed that, now a days more sophisticated instruments having improved minimum detection limits are available for testing iron and purity. Considering this, the committee decided to revise the standard. In this revision alternate test methods for iron and purity have been added and Amendment no. 1 and 2 have also been incorporated.

The composition of the Committee responsible for the formulation of this standard is given in 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 of 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

ACETIC ACID — SPECIFICATION

(Fourth Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for acetic acid.

2 REFERENCES

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

IS No.	*Title*
82 : 1973	Methods of sampling and test for thinners and solvents for paints (*first revision*)
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)
915 : 2012	Laboratory glassware — One — mark volumetric flasks (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1745 : 2018	Petroleum hydrocarbon solvents — Specification (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)
1446 : 2002	Classification of dangerous goods (*second revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
2362 : 1993	Determination of water by Karl fischer method — Test method (*second revision*)
2618 : 2016	Laboratory glassware — Test tubes (*third revision*)
4905 : 2015	Random sampling and randomization procedures (*first revision*)
4161 : 1967	Specification for Nessler cylinders
8768 : 2000	Method for measurement of colour in liquid chemical products platinum — Cobalt scale

3 GRADES

The material shall be of three grades, namely:

a) Technical
b) Pure, and
c) Analytical Reagent (AR).

4 REQUIREMENTS

4.1 Description

At all temperatures above the crystallizing point, the material shall be a clear liquid, free from suspended matter and having a characteristic pungent odour.

4.2 Colour

4.2.1 The colour of pure and analytical reagent grades of the material shall not be more than 20 Hazen units when tested as per IS 8768.

4.2.2 The technical grade material shall be colourless or may have apale yellow / straw colour.

NOTE — The material which has been stored in aluminium or wax lined containers may exhibit slight opalescence or may develop opalescence on dilution with water. Material showing such slight opalescence but otherwise complying with the requirements given in Table 1 shall be deemed to conform to this standard.

4.3 The material shall also comply with the requirements given in Table 1.

4.4 The material, when required for pharmaceutical purposes, shall contain not more than 2 parts per million of arsenic (as As) which is equivalent to 2.664 parts (as As_2O3) when tested as prescribed in IS 2088.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier, and

shall be subject to the provisions of law in force in the country for the time being.

NOTE — Acetic acid expands on freezing and due precautions shall be taken in its packing and storing.

5.2 Marking

5.2.1 Each container shall be securely closed and marked with the following:

a) Manufacturer's name;

b) Grade of the material;

c) Tare, gross and net mass;

d) Year of manufacture; and

e) Recognized trade – mark, if any

5.2.2 Each container shall bear the caution label worded as under

CORROSIVE! VAPOUR AND LIQUID DANGEROUS TO EYES!

NOTES:

1 Necessary safeguards against the risk arising from storage and handling of large volumes of corrosive liquids(*see* IS1446) shall be provided and all due precautions shall be taken [*see* also IS 1260 (Part1)].

2 Except when they are opened for the purpose of cleaning and rendering them free from acetic acid vapour, all empty tanks or other containers shall be kept securely closed unless they have been cleaned and freed from acetic acid vapour.

5.2.3 *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.'

6 SAMPLING

The method of drawing representative samples of the material shall be as prescribed in Annex B.

7 TEST METHODS

7.1 Tests shall be conducted according to the methods prescribed in column 6 of Table 1.

7.2 Quality of Reagents

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

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

Table 1 Requirements for Acetic Acid

[*Clauses* 4.2.2 (*Note*) 4.3 *and* 7.1]

Sl No.	Characteristic	Requirements for Grade			Method of Test, Ref to
		Technical	Pure	Analytical Reagent	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Solubility in water	-	Shall be completely miscible	No turbidity within one hour	A – 1
ii)	Relative density at 27/27 °C *Max*	1.0528	1.0496	1.048 3	6 of IS 82
iii)	Colour Hazen units *Max*	-	20	20	IS8768
iv)	Acetic acid content (CH_3COOH), percent by mass, *Min*	98.0	99.5	99.7	A - 2
v)	Crystallizing point, °C, *Min*	-	15.6	16	A - 3
vi)	Residue on evaporation, percent by mass, *Max*	0.02	0.01	0.001	A - 4
vii)	Chloride (as Cl), ppm, *Max*	35	15	1	A - 5
viii)	Iron (as Fe), ppm, *Max*	-	2	1	A - 6
ix)	Sulphate (as SO_4), ppm, *Max*	35	15	1	A - 7
x)	Heavy metals [(including iron) calculated as Pb], ppm, *Max*	20	5	2	A - 8
xi)	Formic acid (HCOOH), percent by mass, *Max*	0.35	0.15	0.025	A - 9
xii)	Acetaldehyde (CH_3CHO), percent by mass, Max	0.15	0.05	0.003	A - 10
xiii)	Oxidizable impurities	-	To pass the test	To pass the test	A - 11
xiv)	Water content, percent by mass, *Max*	-	-	0.2	IS 2362

ANNEXA

(*Table* 1 and *Clause* 7.1)

METHODS OF TEST FOR ACETICACID

A-1 DETERMINATION OF SOLUBILTY IN WATER

A-1.0 Outline of the Method

A known volume of acetic acid is mixed with a known volume of water and observed for turbidity.

A-1.1 Procedure

Dilute 1 volume of acid with 3 volumes of water in a clean test tube. Shake well and allow to stand for 1 h. Compare with an equal volume of water in a test tube.

A-1.2 The test solution shall be as clear as in the comparator tube.

A-2 DETERMINATION OF ACETIC ACID CONTENT

A-2.1 Using Phenolphthalein Indicator

A-2.1.0 *Outline of the Method*

A known amount of the material is titrated against standard sodium hydroxide solution using phenolphthalein as indicator. From the titer values, the percent acetic acid equivalent in the sample is calculated. This is then corrected for the formic acid content to get the percentage of acetic acid in the sample.

A-2.1.1 *Apparatus*

A-2.1.1.1 Lunge-rey pipette (Fig. 1) or flat-bottom weighing bottle of suitable size with lid or any other suitable device for weighing of sample like syringe/ micro pipette (Glass/HEPE/Plastic) for accurate weighment of sample.

A-2.1.1.2 *Conical flask* of 250 ml capacity.

A-**2.1.1.3** *Burette of 25 ml or 50 ml capacity*

A-2.1.2 *Reagents*

A-2.1.2.1 *Phenolphthalein indicator solution* — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent ethyl alcohol and add solution of sodium hydroxide **(A-2.2.2**) until the colour turns faint pink.

A-2.1.2.2 *Standard sodium hydroxide solution* — 1 N or 0.5 N.

A-2.1.3 *Procedure*

Transfer about 1 to 2.5 g of the material, accurately weighed by any means mentioned at **A-2.1.1.1** into a conical flask, add about 50 ml of freshly boiled and cooled water and 0.5 ml of phenolphthalein indicator solution and titrate with sodium hydroxide solution.

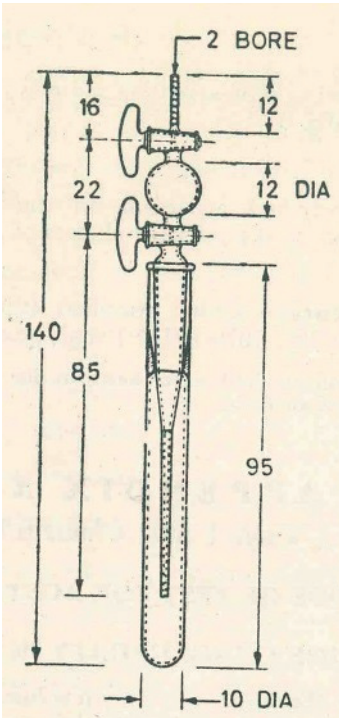

FIG. 1 LUNGE-REY PIPETTE

A-2.1.4 Calculation

Find the mass of acetic acid on the basis that 1 ml of normal sodium hydroxide solution is equivalent to 0.060 05 g of acetic acid (CH_3COOH), and deduct the acetic acid equivalent of any formic acid present, as determined under **A-9**.

Acetic acid (CH_3COOH), percent =

$$\frac{6.005 \times V \times N}{M} - 1.299 \times S$$

Where,

V = volume in ml, of the standard sodium hydroxide solution used;

N = normality of standard sodium hydroxide solution;

S = percentage of formic acid as determined in **A-9**; and

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

A-2.2 Alternate Test Method : Determination of Acetic Acid Purity by Crystallising Point

A-2.2.1 *Outline of the Method*

The method is suitable for determining the purity of relatively pure acetic acid from crystallizing point.

A-2.2.2 *Procedure*

Determine the crystalizing point of the sample using apparatus and following procedure described at (**A-3**) and obtained the percentage acetic acid from the graph illustrated in Fig. 2.

A-2.2.3 *Relationship Between Crystallizing Point and Acetic Acid Content*

A-2.2.3.1 A graph of the relationship between crystallizing point and acetic acid content is shown in Fig. 2. This graph is based on the assumption that water is the only impurity present.

A-2.2.3.2 Relation between crystallization point and Acetic acid purity is correlated from graph (Fig. 2) is placed in Table 2.

Table 2 Relation Between Crystallization Point and Acetic Acid Purity

Crystallizing Point ^{O}C	Acetic Acid by Percent Mass
14.1	98.55
14.2	98.61
14.3	98.66
14.4	98.74
14.5	98.81
14.6	98.88
14.7	98.94
14.8	98.98
14.9	99.05
15.0	99.12
15.1	99.16
15.2	99.24
15.3	99.30
15.4	99.35
15.5	99.41
15.6	99.46
15.7	99.54
15.8	99.58
15.9	99.64
16.0	99.68
16.1	99.74
16.2	99.78
16.3	99.84
16.4	99.88
16.5	99.92
16.6	99.96

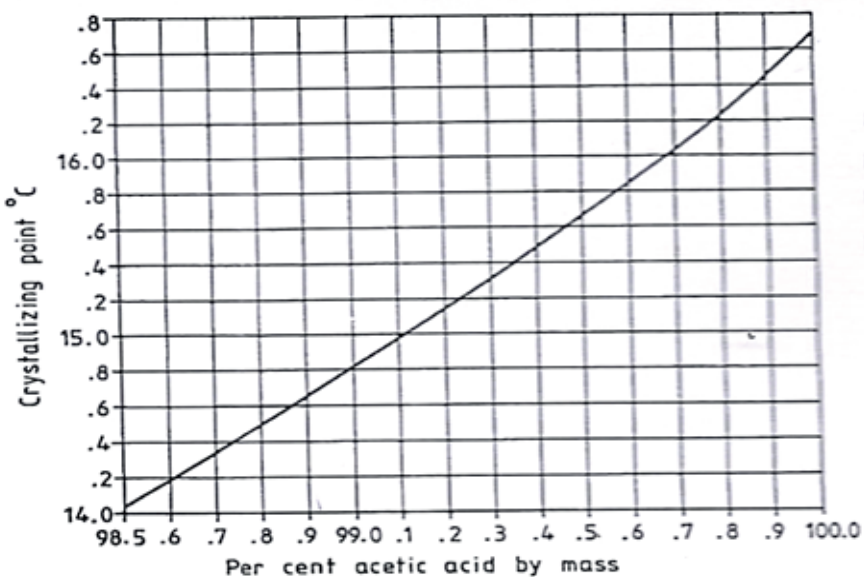

FIG. 2 RELATION BETWEEN CRYSTALIZING POINT AND ACETIC ACID CONTENT

A-3 DETERMINATION OF CRYSTALLIZING POINT

A-3.1 Apparatus

A-3.1.1 *Test-Tube* — About 25 mm in diameter and 150 mm in length (*see* IS 2618).

A-3.1.2 *Thermometer* — Graduated for partial immersion to a depth of 100 mm and possessing the following characteristics:

a)	Range	-0.5 to + 40.5°C
b)	Graduations	0.1°C
c)	Overall length	About 400 mm
d)	Length of main scale, Min	280 mm
e)	Bulb length	10 to 15 mm
f)	Stem diameter	5.5 to 7.0 mm
g)	Distance from bottom of bulb to main scale	Not less than 30 mm
h)	Maximum error	0.4°C
j)	Maximum error in an interval	0.4/0.5°C

A-3.1.2.1 Any other thermometer of different range but similar graduation and accuracy may also be used.

A-3.1.2.2 The thermometer shall bear certificate from the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

A-3.2 Procedure

Fill the dried test-tube with the material to a depth of about 100 mm and insert the thermometer. Place the test-tube in water at 10 to 11°C so that the portion occupied by the material is completely immersed. Allow the material to cool, without stirring, until the thermometer indicates about 2°C below the expected crystallizing point. Lift the tube out of water and Stir rapidly with the thermometer to induce the formation of minute crystals. At the moment the crystallization begins, the temperature will rise rapidly and then remain constant for a few moments. As soon as the steady temperature is approached, stop stirring and suspend the thermometer so that its bulb is centrally disposed in the crystallizing mass. Record the temperature at which the thermometer remains constant as the crystalizing point.

A-3.2.1 If after cooling and stirring as described under **A-3.2**, the temperature rise exceeds 3°C, the observed crystallizing point is liable to be below the true figure. In such cases, repeat the operation with less super-cooling.

A-3.2.2 If crystallization does not begin after removal of the test-tube from the cold water and vigorous stirring, withdraw the thermometer and touch against some solid acetic acid previously prepared and then quickly re-insert it in the material under test and resume stirring.

A-3.2.3 Precautions shall be taken to prevent contamination of the sample with moisture during the test.

A-4 DETERMINATION OF RESIDUE ON EVAPORATION

A-4.0 Outline of Method

A known amount of the material is evaporated to dryness, the residue cooled and weighed.

A-4.1 Procedure

Accurately weigh about 100 g of the material in a tarred silica basin. Gently evaporate it to dryness on a water bath. Dry the residue for one hour in an oven at 120 ± 2°C and cool in a desiccator and weigh again. Retain the residue for further tests.

A-4.2 Calculation

Difference in mass of the silica basin gives the mass of the residue on evaporation. Express the mass of the residue as percentage of the mass of the material taken for the test.

A-5 TEST FOR CHLORIDES

A-5.0 Outline of the Method

A solution of the material of known concentration is treated with silver nitrate solution in presence of dilute nitric acid. The opalescence produced is compared with the opalescence in a standard solution treated similarly with equal amounts of the reagents.

A-5.1 Apparatus

A-5.1.1 *Volumetric Flask* — 250 ml capacity.

A-5.1.2 *Nessler Cylinders*— 100 ml capacity (*see* IS 4161).

A-5.2 Reagents

A-5.2.1 *Dilute Nitric Acid* — Approximately 5 N.

A-5.2.2 *Silver Nitrate Solution* — 5 percent *(m/v)*.

A-5.2.3 *Standard Chloride Solution* — Dissolve 0.1649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out 10 ml of this solution and dilute again with water to 100 ml. One milliliter of the final solution contains 0.01 mg of chloride (as Cl).

A-5.3 Procedure for Pure and Technical Grades

A-5.3.1 Dilute 25.0 g of the material up to the mark with water in a 250 ml volumetric flask. Filter the solution if it is opalescent. If opalescence persists, it may be

removed by shaking with petroleum hydrocarbon solvent (*see* IS 1745). Transfer 50 ml of this solution in case of pure grade and 20 ml in the case of technical grade of the material to a Nessler cylinder, add 2 ml of nitric acid and make the volume to the mark with water. Add 1 ml of silver nitrate solution and mix well. Carry out a control test with 7.5 ml of standard chloride solution in case of pure grade and 7 ml in the case of technical grade in place of the material. Allow the cylinders to stand in dark for 5 min and then compare the opalescence of the solutions.

A-5.3.2 The limits prescribed in Table 1 shall be taken as not having been exceeded the opalescence produced in the test with the material is not greater man that produced in the control test.

A-5.4 Procedure for Analytical Reagent Grade

A-5.4.1 Dilute 25 g of the material with 50 ml of water in a Nessler cylinder, add 10 ml of dilute nitric acid and make up to 100 ml with water. Add 1 ml of silver nitrate solution, stir immediately with a glass rod, set aside for 5 min and compare the opalescence produced with that produced in another Nessler cylinder using 2.5 ml of standard chloride solution instead of the material.

A-5.4.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced in the test with the material is not greater than that produced in the control test.

A-6 TEST FOR IRON

A-6.1 Using Thioglycolic Acid

A-6.1.0 *Outline of the Method*

An extract of residue on evaporation of the material in hydrochloric acid is treated with thioglycollic acid and ammonium hydroxide. The colour developed is then compared with that of a standard solution of iron treated similarly.

A-6.1.1 *Apparatus*

A-6.1.1.1 *Nessler cylinders* — 50 ml capacity.

A-6.1.2 *Reagents*

A-6.1.2.1 *Concentrated hydrochloric acid* — Conforming to IS 265

A-6.1.2.2 *Thioglycollic acid* — 50 percent (*m/ v*).

A-6.1.2.3 *Ammonium hydroxide* — Relative density 0.92 at 25/25°C

A-6.1.2.4 *Standard iron solution* — Dissolve 0·702g of ferrous ammonium sulphate [$FeSO_4.(NH4)_2SO_4.6H_2O$] in water containing 10 ml of concentrated sulphuric acid and dilute with water to 1 000 ml. Pipette 10 ml of this solution and dilute to 100 ml. One milliliter of the solution contains 0 01 mg of iron (as Fe).

A-6.1.3 *Procedure*

Heat the residue obtained under **A-4.1** to a uniform dull red heat and continue heating until fumes are no longer evolved. Allow the basin to cool. Add 5 ml of concentrated hydrochloric acid followed by 10 ml of water.Warm to dissolve, cool and transfer the solution to a 100-ml volumetric flask, filtering, if necessary, to obtain a clear solution. Dilute to the mark with water and mix well.

A-6.1.3.1 Transfer 25 ml of the solution to a Nessler cylinder, add 2 drops (approximately 0.1 ml) of thioglycollic acid and then ammonium hydroxide drop by drop until a pink colour just persists after stirring. Add a further 0·5 ml of ammonium hydroxide, dilute to 50 ml with water and mix well. Carry out a control test using 5 ml of standard iron solution in case of pure grade and 2.5 ml in the case of analytical reagent grade and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour in the two tubes.

A-6.1.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not darker than that produced in control test.

A-6.2 Alternative Method using UV-visible Spectrophotometer

A 6.2.0 *Outline of the Method*

Iron(Fe^{+2}) reacts with 1, l0 phenanthrolinein to form an orange-red complex [$(C_{12}, H8N2)_3Fe]^{2+}$. The colour intensity is independent of the acidity in the *p*H range 2-9, and is stable for long periods. The complex which follows Beers's Law closely and its absorbance is very stable for long period.

Ferric iron reduced with hydroquinone and *p*H is adjusted with an acetate buffer. The solution, slightly acid with hydrochloric acid, is treated with 1, 10-phenanthroline, and buffered with sodium citrate at a *p*H of 4. The iron - phenanthroline complex is measured at 510 nm against reagent blank.

A-6.2.1 *Apparatus*

A-6.2.1.1 *One-mark volumetric flasks* — 1 000 ml capacity (*see* IS 915).

A-6.2.1.2 *One-mark volumetric flasks* — 500 ml capacity (*see* IS 915).

A-6.2.1.3 *One-mark volumetric flasks* — 50 ml capacity (*see* IS 915).

A-6.2.1.4 *UV-Vis spectrophotometer*

A–6.2.1.5 *Glass cell of 50 mm path length* — 2 Nos.

A–6.2.1.6 *Standard micro burette 10 ml capacity*.

A-6.2.2 *Reagents*

A-6.2.2.1 *Hydrochloric acid* — Relative density 1.16 (*see* IS 265) 1 : 1 HCl : Dilute hydrochloric acid with equal volume of DM water

A-6.2.2.2 1 Percent hydroquinone solution in DM water. Kept in brown bottle and discard when turbid.

A-6.2.2.3 25 Percent of sodium citrate solution in DM water

A-6.2.2.4 0.10 Percent of 1, 10 phenanthrolinein DM water

A-6.2.2.5 *Standard iron solution* — Dissolve 3.511 gm of ammonium ferrous sulphate ($FeSO_4$ $(NH_4)2SO_4$, $6H_2O$) in 500 ml DM water containing 10 ml con H_2SO_4.

1 ml = 1000 micro gram iron -----> (A).

Take 2 ml of solution (A) with micro burette in 1 000 ml volumetric flask. Make up with DM water. It will give 1 ml = 2 micro gram -------> (B)

A-6.2.3 *Procedure*

To the basin containing the residue from the determination of residue on evaporation (*see* **A-4.1**), add 5 ml of concentrated hydrochloric acid. Heat the basin on a boiling water-bath, agitating with a glass rod until all the residue has dissolved. Allow to cool, transfer to the 50 ml volumetric flask, (if high iron content is expected, then make up the volume to 50 ml and take suitable aliquot into another 50 ml volumetric flask). Add 1 ml 1:1 HCl, add 1.0 ml of 1 percent Hydroquinone solution, stir well and wait for 5 min. Add 5 ml 0.1 percent 1, 10 phenanthroline and 5 ml 25 percent sodium citrate solution. Stir well and make up the volume up to the mark with DM water. Measure the absorbance or direct concentration after 30 min using 50 mm cell at 510 nm wavelength.

Calculation:

$$\text{Iron as Fe, ppm} = \frac{\text{Micro gm of Fe} \times \text{dilution factor if applicable}}{\text{M} \times \text{aliquot of sample stock}}$$

Where, M = mass in g, of sample taken for the determination of non-volatile matter (**A-4.1**).

A-6.2.4 *Calibration of Spectrophotometer for Iron : Calibration Range: 0 to 10 Micro gram.*

A-6.2.4.1 *Procedure*

Take 1, 2, 3, 4, and 5 ml of std. (B) (*see* **6.2.2.5**) in 50 ml volumetric flask keeping sixth as blank. Add 1 ml 1:1 HCl, add 1.0 ml of 1 percent hydroquinone solution, stir well and wait for 5 min. Add 5 ml 0.1 percent 1, 10 phenanthroline and 5 ml 25 percent sodium citrate solution. Stir well and make up the volume up to the mark with DM water. Measure the absorbance or direct concentration after 30 min using 50 mm cell at 510 nm wavelength. Measured absorbance data and slope value are mentioned.

NOTES

1 Other type of spectrophotometric method may be used, provided slope determination for the specified range is established and accuracy level as specified in this standard is achievable.

2 For analysis of iron instrumental method of analysis like AAS, ICP may be used, provided calibration of instrument is done in accordance with detection levels as necessary with requirements of this standard.

A-7 TEST FOR SULPHATES

A-7.0 Outline of the Method

A known quantity of the material is evaporated to dryness with sodium carbonate solution, the residue is extracted with hydrochloric acid and then treated with barium chloride solution. The turbidity, if any, is compared with the turbidity produced in a standard sulphate solution treated with hydrochloric acid and barium chloride solution.

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50 ml capacity (*see* IS 4161).

A-7.2 Reagents

A-7.2.1 *Sodium Carbonate Solution* — 1N approximately.

A-7.2.2 *Dilute Hydrochloric Acid* — 1N approximately.

A-7.2.3 *Barium Chloride Solution* — Dissolve 10 g of barium chloride crystals in water and make up to 100 ml.

A-7.2.4 *Standard Sulphate Solution* — Dissolve 0.1814 g of potassium sulphate in water and makeup the volume to 1 000 ml. Pipette 10 ml of this solution and dilute to 100 ml. One milliliter of this solution is equivalent to 0.01 mg of sulphate (as SO_4).

A-7.2.5 *Standard Sulphuric* — 0.01N.

A-7.3 Procedure for Pure and Technical Grades

Transfer 50 ml in case of pure grade and 20 ml in the case of technical grade of solution prepared under **A-5.3.1** into a 250 ml beaker, and add 0.2 ml of

sodium carbonate solution. Evaporate it to dryness on a water bath. Dissolve the residue in 15 ml of water, add 1 ml of dilute hydrochloric acid, filter and collect the filtrate in one of the Nessler cylinders. Add 2 ml of barium chloride solution, dilute to 50 ml, mix well, set aside for 5 min and mix again. Carry out a control test in the other Nessler cylinder using 7.5 ml of standard sulphate solution in case of pure grade and 7 ml in the case of technical grade in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Stir the solution immediately with glass rod and set aside for 5 min. Compare the turbidities produced in the two cylinders.

A-7.4 Procedure for Analytical Reagent Grade

A-7.4.1 Transfer 50 g of the material into a 250 ml beaker and add 0.2 ml of sodium carbonate solution. Evaporate it to dryness on a water bath. Dissolve the residue in 15 ml of water and add 1 ml of dilute hydrochloric acid, filter and collect the filtrate in one of the Nessler cylinders. Add 2 ml of barium chloride solution, dilute to 50 ml, mix well, set aside for 5 min and mix again. Carry out a control test in the other Nessler cylinder using 5.0 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Stir the solution immediately with glass rod and set aside for 5 min. Compare the turbidities produced in the two cylinders.

A-7.4.2 The limits prescribed in Table1 shall be regarded 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.

A-8 TEST FOR HEAVY METALS

A-8.0 Outline of the Method

A known quantity of the material is treated with ammonium hydroxide and then with sodium sulphide. A standard solution of lead nitrate is also similarly treated with equal amounts of the reagents and the colours are compared.

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50 ml capacity (*see* IS 4161).

A-8.2 Reagents

A-8.2.1 *Ammonium Hydroxide* — Relative density 0.92 at 25/25°C.

A-8.2.2 *Litmus Paper*

A-8.2.3 *Sodium Sulphide Solution* — Solution of sodium sulphide crystals in water, approximate 10 percent (*m/v*).

A-8.2.4 *Standard Lead Solution* — Dissolve 1.60 g of lead nitrate [Pb $(NO_3)_2$] in water to produce 1 000 ml of solution. Pipette 10 ml of this solution and dilute again to 1 000 ml with water. One milliliter of the final solution is equivalent to 0.01 mg of lead (Pb).

A-8.3 Procedure

Transfer 10 g of the material in case of analytical reagent grade, 4 g in case of pure grade and 1 g in the case of technical grade in one of the Nessler cylinders. Make it just alkaline to litmus paper with ammonium hydroxide. Add sufficient water to make up the volume to 50 ml. Carry out a control test in the other Nessler cylinder using 2 ml of standard lead solution and the same quantities of other reagents in the same total volume of the reaction mixture. Add 2 drops of sodium sulphide solution to each cylinder and shake well. Compare the colour produced in the two tubes.

A-8.3.1 The limits prescribed in Table 1 shall be regarded as not having been exceeded if the colour produced in the test with the material is not darker than that produced in the control test.

A-9 DETERMINATION OF FORMIC ACID

A-9.0 Outline of the Method

A known amount of the material is treated with sodium hypobromite, potassium iodide and dilute hydrochloric acid. The iodine liberated is titrated with standard sodium thiosulphate solution. From the difference in titre values, the formic acid content is calculated.

A-9.1 Reagents

A-9.1.1 *Sodium Hypobromite Solution* — Approximately 0.1 N, prepared by dissolving, with thorough mixing, 2.8 ml of bromine and 100 ml of 2 N sodium hydroxide solution in 500 ml of water and diluting further with sufficient water to make up the volume to one litre.

A-9.1.2 *Potassium Iodide Solution* — Approximately 25 percent (m/v).

A-9.1.3 *Dilute Hydrochloric Acid* — Approximately 5 N.

A-9.1.4 *Standard Sodium Thiosulphate Solution* — 0.1 N.

A-9.2 Procedure

In each of two iodine flasks, place 100 ml of water and 25 ml of sodium hypobromite solution. To one flask, add about 10 g of the material, accurately weighed, and allow to stand for 5 min, taking precautions against loss of bromine vapour. To both flasks, add 5 ml of potassium iodide solution and then 20 ml of dilute

hydrochloric acid, and titrate the liberated iodine with standard sodium thiosulphate solution.

A-9.3 Calculation

From the difference in volumes of standard sodium thiosulphate solution required for titration with the material and for the blank, fined the mass of formic acid on the basis that 1 ml of normal sodium thiosulphate solution is equivalent to 0.023 g of formic acid (HCOOH), and calculate using the following formula

Formic acid (HCOOH), percent by mass =

$$\frac{23\ (V_1 - V_2)\ N}{M}$$

Where,

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

V_2 = volume in ml, of standard thiosulphate solution used in the test with the material;

N = normality of standard thiosulphate solution; and

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

A-10 DETERMINATION OF ACETALDEHYDE

A-l0.0 Outline of the Method

A known excess of sodium hydrogen sulphite is added to a known amount of the material. The excess of sodium hydrogen sulphite is back titrated with standard iodine solution. A blank determination, is also carried out.

A-10.1 Reagents

A-10.1.1 *Sodium Hydrogen Sulphite Solution* — Approximately 1.25 percent (m/v).

A-10.1.2 *Standard Iodine Solution* — 0.1 N.

A-10.2 Procedure

To 50 ml of water in a 250 ml glass stoppered flask, add about 10 g of the material, accurately weighed, and 10 ml of sodium hydrogen sulphite solution. Shake well and let stand for 30 min. Titrate the excess of sodium hydrogen sulphite with standard iodine solution. Carry out a blank determination with the same amount of sodium hydrogen sulphite solution but without the material.

A-10.3 Calculation

From the difference in volumes of standard iodine solution required in the titration with the material and in the blank determination, find the mass of acetaldehyde on the basis that 1 ml of normal iodine solution is equivalent to 0.022 g of acetaldehyde (CH_3CHO) and calculate using the following formula:

$$\text{Acetaldehyde, percent by mass} = \frac{2.2\ (V_1 - V_2)\ N}{\text{M}}$$

Where,

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

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

N = normality of standard iodine solution; and

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

A-11 TEST FOR OXIDIZABLE IMPURITIES

A-11.1 Reagents

A-11.1.1 *Potassium Permanganate Solution* — 0.1 N.

A-11.2 Procedure

Dilute 25 g of acetic acid under test, up to the mark in a 250 ml volumetric flask. Transfer 5 ml of this diluted acetic acid in a 100 ml test-tube and add 0.5 ml of 0.1 N potassium permanganate solution and shake.

A-11.2.1 The material shall be taken to have passed the test if the pink colour does not entirely disappear within half a minute.

ANNEX B

(*Clause* 6.1)

SAMPLING OF ACETIC ACID

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.1 Samples shall be taken in a protected area with good ventilation. Keep the samples away from flame.

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

B-1.3 The samples, material being sampled, sampling instrument and the containers for samples shall be protected from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

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

B-1.6 The sample containers shall be of such a size that they are almost but not completely filled by the sample.

B-1.7 Each sample Container shall be sealed air-tight with a suitable stopper after filling and marked with the manufacturer's name or trade-mark, the month and year of manufacture of the material, the batch number (if available), and other details of sampling, such as the date of sampling and sampler's name.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2 SAMPLING INSTRUMENTS

B-2.1 The following forms of sampling instruments may be used:

a) Weighted sampling can for taking samples from various depths in large tanks,

b) Sampling tube, and

c) Sample point from pump discharge material after thorough re-circulation of the material in tank (wherever possible).

B-2.2 Weighted Sampling Can

Of suitable capacity, 500 to 1 000 ml, and of such a mass as to sink readily in the material to be sampled. It has a long chain or cord attached to permit filling at any desired level (*see* Fig. 3). The metal used to weigh the apparatus shall be fitted externally as irregularities in the metal are likely to contaminate the sample if the mass is fitted internally.

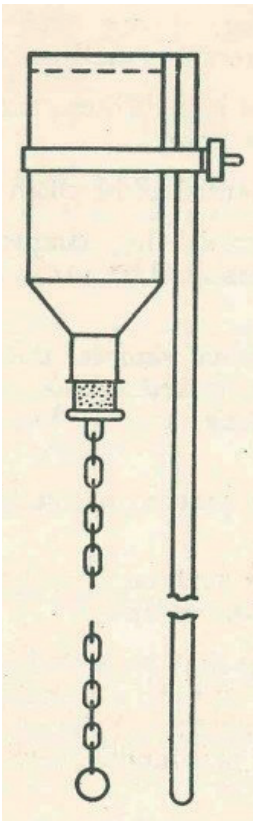

FIG. 3 WEIGHTED SAMPLING CAN

B-2.3 Sampling Tube

It is made of metal or thick glass and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 4). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper ends. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

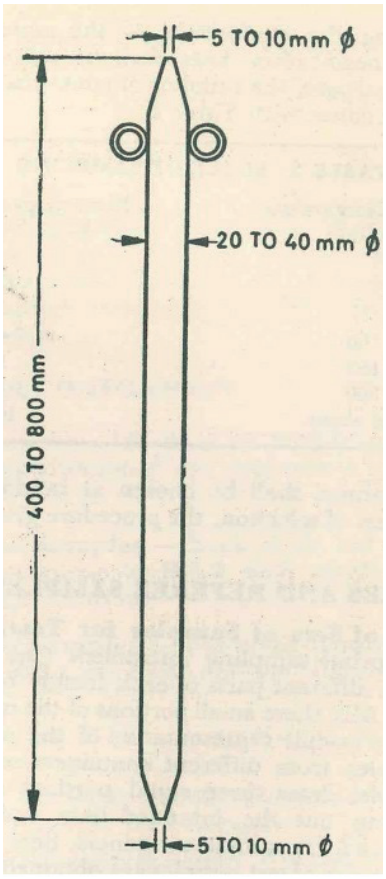

FIG. 4 SAMPLING TUBE

B-2.3.1 For small containers, the size of the sampling tube may be altered suitably.

B-3 SCALE OF SAMPLING

B-3.1 Lot

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

B-3.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot separately. For this purpose, the number of containers to be selected from a lot shall be in accordance with Table 3.

B-3.2.1 The containers shall be chosen at random from the lot. To ensure the randomness of selection, the procedure given in IS 4905 may be followed.

Table 3 Scale of Sampling

(*Clause* B-3.2)

Number of Containers in the Lot	Number of Containers to be Selected
(N)	(n)
(1)	(2)
Up to 25	3
26 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

B-4 TEST SAMPLES AND REFEREE SAMPLES

B-4.1 Preparation of Sets of Samples for Testing Individually

Draw with an appropriate sampling instrument (*see* **B-2**) equal portions of the material from different parts of each freshly opened container, and selected as in **B-3.2**. Mix these sample portions of the material from the same, container to obtain a sample representative of the container. Keep these representative sample from different container separately. From each representative sample, draw three equal portions of the material, each sufficient for carrying out the intended tests and transfer them into thoroughly cleaned and dry sample containers. Seal the sample containers air-tight. Thus, three sets of test samples are obtained such that each set has a test sample from each selected container. Send one each of these sets to the purchaser and the supplier. Reserve the third set as referee sample bearing the seals of the purchaser and the supplier. Keep the referee sample at a place agreed to between the purchaser and the supplier.

B-4.2 Preparation of Composite Samples

Draw with an appropriate sampling instrument (*see* **B-2**) equal portions of material from different parts of each freshly opened container selected as in **B-3.2** and mix them thoroughly together to constitute a single composite sample. Divide this composite sample into three parts, each sufficient for carrying out the intended tests, and transfer them to thoroughly cleaned and dry sample containers. Send one each of these to the purchaser and the supplier. Reserve the third composite sample as referee sample bearing the seals of the purchaser and the supplier. Keep the referee sample at a place agreed to between the purchaser and the supplier.

B-5 NUMBER OF TESTS

B-5.1 Individual Samples

Tests for acetic acid content shall be carried out individually on each of the test samples (*see* **B-4.1)**.

B-5.2 Composite Samples

Tests for determination of the remaining characteristics of this specification shall be carried out on the composite sample prepared in **B-4.2**.

B-6 CRITERIA FOR CONFORMITY

B-6.1 The material in any lot shall be accepted as conforming to the requirements of this specification if the test results satisfy the conditions of **B-6.2** and **B-6.3**.

B-6.2 For Composite Samples

Each of the test-results made on the composite sample according to **B-5.2** shall satisfy the corresponding requirements of this specification.

B-6.3 For Individual Samples

For acetic acid content of individual samples according to **B-5.1**, the mean and the range of test results shall be calculated as follows:

Mean $\bar{X}$ = sum of test results divided by the number of test results, and

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

The expression ($\bar{X} - 0.6R$) and ($\bar{X} + 0.6R$) shall then be calculated. The lot shall be considered to conform to the requirement of this specification if ($\bar{X} - 0.6R$) is greater than the minimum specified value and ($\bar{X} + 0\ .6R$) is less than the maximum specified value. When the sample size is ten, the test results shall be grouped into two, each constituting five test results taking them consecutively in the same order as obtained. For each group, the range shall be calculated and the mean range R shall be calculated by dividing the sum of the ranges by two. In the expression ($\bar{X} + 0.6$R), R shall be replaced by $\bar{R}$.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Organic, Chemicals Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
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Review of Indian Standards

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Regional Offices: *Telephones*

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 — 2323 7617, 2323 3841

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