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BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

ACETALDEHYDE – SPECIFICATION

(*First Revision of* IS 15356)

(ICS 71.080.80)

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09	Last date for Comments **22 December 2023**

FOREWORD

(*Formal Clauses will be added later*)

Acetaldehyde is present in various plants, ripe fruits, vegetables. exhaust from the engine etc. Acetaldehyde is also an important intermediate in the production of acetic acid, acetic anhydride, ethyl acetate, peracetic acid, pentaerythritol, chloral, glycol, alkyl amines and pyridines.

Acetaldehyde has a general narcotic action, which prevents coughing. It causes irritation of the eye, mucus membranes and accelerates heart action. Acetaldehyde also appears to paralyze respiratory muscles when breathed in high concentration it causes headache and sore throat. Prolonged exposure causes a decrease of red and white blood cells and also sustained rise in blood pressure. The maximum allowable concentration of acetaldehyde in air is 200 ppm. However, in a normal industrial operation, there is no hazard in handling acetaldehyde provided normal precautions are taken.

In this (*first*) revision, gas chromatographic method for determination of assay has been incorporated as alternate method. Method for determination of acidity and paraldehyde have been modified. Also, the limit of paraldehyde has been modified to 0.5 from 0.7, percent by mass, *Max*.

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 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for acetaldehyde intended for industrial purposes.

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 this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water - Specification (*fourth revision*)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods (*first revision*)
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
IS 4905 : 2015 / ISO 24153: 2009	Random sampling and randomization procedures (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be clear liquid free from suspended matter and having a characteristic odour.

3.2 Colour

The material shall be colorless to light yellow.

3.3 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 and col 5 of Table 1.

Table 1 Requirements of Acetaldehyde
(*Clauses* 3.3 *and* D-5.1**)**

Sl No.	**Characteristics**	**Requirement**	**Method of test , Ref to**	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i.	Total aldehydes (*as* acetaldehyde), percent by mass, *Min*	98.5	A/B	—
ii.	Acidity (*as* acetic acid), percent by mass, *Max*	0.1	C	—
iii.	Water content, percent by mass, *Max*	0.5	—	IS 2362
iv.	Paraldehyde content, percent by mass, *Max*	0.5	B	—

3.3.1 *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 result of analysis.

4 PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in suitable containers as agreed to between the purchaser and the supplier and subject to the provisions of **Red Tariff No. for Rules and Rates for Conveyance by Rail of Explosives and Other Dangerous Goods**, issued by the Indian Railways Conference Association with any alternations or additions made thereafter.

4.1.2 All containers in which the material is packed shall be dry and clean so that no impurities harmful to the end use of the material are introduced.

4.2 Marking

4.2.1 Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his trade-mark, if any;
c) Net mass of the material;
d) Month and year of manufacture;
e) Lot or batch number; and
f) Any other statutory requirement.

4.2.2 The material shall also be marked in accordance with the marking and delivery instructions given by the purchaser.

4.2.3 Each container shall also be marked with the caution label 'FLAMMABLE' together with the corresponding symbol for labelling [*see* IS 1260 (Part l)].

4.2.4 *BIS Certification Marking*

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

5 SAMPLING

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

ANNEX A
[*Table* 1, *Sl No.* (i)**]**
DETERMINATION OF TOTAL ALDEHYDES (*as* ACETALDEHYDE)

A-1 OUTLINE OF THE METHOD

A known amount of sample is treated with hydroxylamine hydrochloride solution and the hydrochloric acid liberated in the oxime reaction is titrated with standard sodium hydroxide solution. From the titre values, the acetaldehyde content is calculated.

A-2 APPARATUS

A-2.1 Conical Flask, 250 ml capacity fitted with a B24 stopper.

A-3 REAGENTS

A-3.1 Standard Sodium Hydroxide Solution, 1 N.

A-3.2 Hydroxylamine Hydrochloride Solution, 1 N**,** 7 percent neutral to bromophenol blue.

A-3.3 Bromophenol Blue Aqueous Solution, 0.02 percent.

A-4 PROCEDURE

To 50 ml of hydroxylamine hydrochloride solution in a stoppered conical flask, add 4 to 5 drops of bromophenol blue solution (weigh to the nearest 0.002 g). Transfer about 1 g sample in the flask and mix thoroughly. Open the flask to let air in, stopper it again and weigh. Titrate the liberated acid with standard sodium hydroxide solution until blue green colour appears. Allow the flask with the contents to stand for 15 min and if the colour changes, titrate again with sodium hydroxide. Note the volume of sodium hydroxide solution used (*V*, in ml). Carry out a blank titration using all reagents except the sample. Note the volume of sodium hydroxide solution used (*v*, in ml).

A-5 CALCULATION

Calculate the total percentage of acetaldehyde by the following formula**:**

$$\text{Total Aldehyde (}as\text{ acetaldehyde), percent by mass} = \frac{(V-v)\times 4.4\times N}{M}$$

where

V = volume of 1 N standard sodium hydroxide solution used, in ml

v = volume of 1 N standard sodium hydroxide solution used for blank titration, in ml

N = normality of standard sodium hydroxide solution; and

M = mass of the sample taken, in g.

ANNEX B

[*Table* 1, *Sl No.* (i)**]**

DETERMINATION OF TOTAL ALDEHYDES (*as* ACETALDEHYDE) AND ITS IMPURITIES BY GAS CHROMATOGRAPHIC METHOD

B-1 GENERAL

This test method covers the determination of the acetaldehyde content and impurity such as paraldehyde by gas chromatography. Assay as acetaldehyde is derived by subtracting sum of the various impurities like acidity and moisture.

B-2 SUMMARY OF TEST METHOD

A representative sample is introduced into a gas chromatograph equipped with 6 percent cyanopropylphenyl, 94 percent dimethylpoly-siloxane bonded phase fused silica capillary column. Suitable carrier gas transports the vaporized sample through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The product and other components are identified by comparing their retention times to the ones identified by analysing standards under identical conditions. The concentration of all components are determined in mass percent area by area normalization of the peak areas.

B-3 APPARATUS

B-3.1 Gas Chromatograph

Any Gas chromatograph equipped with a flame ionization detector (FID), a split injector (for example, split ratio – 2 : 1) and a suitable electronic integrator/software, capable of operating at the conditions listed below, may be used:

Column	:	Fused silica capillary column coated with 6 percent cyanopropylphenyl, 94 percent dimethylpoly-siloxane with length 30 m; internal diameter 0.53 mm or 0.32 mm or 0.25 mm and film thickness 1.8 or 3.0 µm or equivalent
Injector		
Temperature	:	240 °C
Carrier gas	:	Nitrogen/Helium (3 ml/min or suitable as per column internal diameter)
Split ratio	:	1.25 or suitable
Detector		
Type	:	Flame ionization

Temperature	:	240 °C
Sample size	:	1 µl
Oven Program		
Initial temperature	:	60°C
Initial hold time	:	0 min
Programme rate	:	12 °C/min
Final temperature	:	240°C
Final hold time	:	0 min
Total run time	:	15 min

NOTE — The above Gas chromatographic conditions are suggestive. However any GC having different columns (packed / Capillary having different length / diameter / film thickness) and different carrier gas (He, H_2 or N_2), with different calibration technique (Internal standard, External standard, Area normalization) may be used provided standardization / calibrations are done after setting up chromatographic conditions for required resolution.

B-4 REAGENTS

B-4.1 Acetaldehyde, 99 percent pure

B-4.2 Paraldehyde, pure

B-5 DATA ACQUISITION SYSTEM

Any suitable data integrator or PC based gas chromatograph software, which can handle features like external / internal standard calculations, etc.

B-6 IDENTIFICATION AND CALIBRATION

B-6.1 Identification

Determine the retention time of each component by injecting small amount of highly pure material either individually or synthetic blend mixture**.**

B-6.2 Calibration

B-6.2.1 Accurately prepare calibration standard mixture of known concentration for each component of acetaldehyde and paraldehyde.

B-6.2.2 Inject with the help of a clean and dry glass micro syringe, 1 µl of standard in the column taking care that no air bubble is trapped in the syringe. Inject each standard at least twice or till the repeatable results are obtained. Carry out the calibration by external standard method. Area of each component is to be measured with suitable data acquisition system.

The calibration factor is calculated by the following formula:

$$\text{Response factor of paraldehyde} = \frac{Concentration\ of\ standard\ paraldehyde\ obtained}{Area\ percent\ of\ standard\ paraldehyde}$$

NOTE — Suitable software for auto calibration with respect to mass of calibration standard may also be used.

B-7 PROCEDURE

Inject 1 µl of sample by using manual or automatic liquid syringe, without any air bubble trapped in the syringe. Determine the mass concentration of all components by area normalization method.

B-8 CALCULATION

B-8.1 Calculate concentrations of impurities by correcting with respective response:

Concentration of paraldehyde, percent by mass = Area of paraldehyde x Response Factor

B-8.2 Calculate the assay of Acetaldehyde on dry basis as below:

$$\text{Assay of Acetaldehyde} = \text{Acetaldehyde by GC} - \left[\frac{\text{Water x Acetaldehyde .by GC}}{100} + \frac{\text{Acidity x Acetaldehyde by GC}}{100} \right]$$

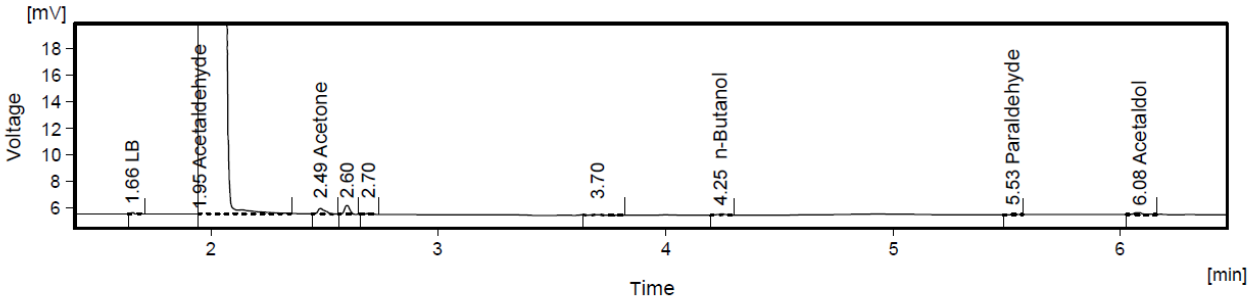


FIG. 1 TYPICAL CHROMATOGRAPH

ANNEX C
[*Table* 1, *Sl No.* (iii)**]**
DETERMINATION OF ACIDITY (*as* ACETIC ACID)

C-1 REAGENTS

C-1.1 Rectified Spirit, 95 percent (*see* IS 323) or methanol, pure

C-1.2 Standard Sodium Hydroxide Solution, 0.02 N or different normality with appropriate sample weight.

C-1.3 Phenolphthalein Indicator

Dissolve 0.5 g of the phenolphthalein in 100 ml of 95 percent rectified spirit. Make the solution faintly pink by adding dilute sodium hydroxide solution.

C-2 PROCEDURE

C-2.1 Take 50 ml of rectified spirit or pure methanol. Add 0.5 ml of phenolphthalein indicator and neutralize with sodium hydroxide solution. Add 50 ml of the sample and titrate with the standard sodium hydroxide solution until the first pink colour persists for at least 10 s.

C-3 CALCULATION

$$\text{Acidity (}as\text{ acetic acid), percent by mass} = \frac{6.005 \times V \times N}{50 \times d}$$

where

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

N = normality of standard sodium hydroxide solution, and

d = relative density of acetaldehyde at the test temperature.

ANNEX D
(*Clause* 5**)**
SAMPLING OF ACETALDEHYDE

D-1 GENERAL REQUIREMENTS OF SAMPLING

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

D-1.1 Samples shall be taken in a protected area with good ventilation.

D-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking, stirring, rolling or by any other suitable means.

D-1.4 The samples shall be placed in suitable, clean, dry and cooled air-tight, amber-coloured glass or metal containers on which the material has no action.

D-1.5 The sample containers shall be of such a size that they are almost completely tilled by the sample.

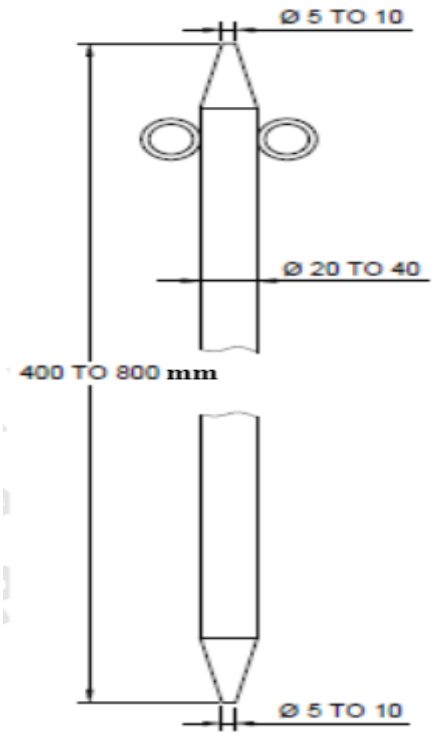
D-1.6 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the manufacturer’s name or trade-mark, the month and year of manufacture of the material, the batch number and other details of sampling, such as the date of sampling, sampler’s name, etc.

D-1.7 The sample of acetaldehyde should be handled only in a fume hood which is free from open flames, electric heaters and other sources of ignitions. All samples shall be cooled in ice baths before the containers are opened. Acetaldehyde is weighed in sealed glass ampoule. The actual procedure for filling and sealing the ampoule varies. One convenient method is to pack commercial available ampoules in powdered solid carbon dioxide, introduce the specimen by means of chilled hypodermic syringe and seal the ampoule with a glass torch.

D-2 SAMPLING INSTRUMENT

D-2.1 It is made of thick glass or metal on which the material has no action and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2).

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 end. For drawing sample, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short period to admit the material at the desired depth and finally closed and withdrawn.



All dimensions in milliliter

FIG. 2 SAMPLING TUBE

D-2.1.1 For small containers, the size of the sampling tube may be altered suitably.

D-2.1.2 In case of sampling from bulk storage tank (through circulation pump sampling valve), suitable precooled glass bottle as sampling container may be used.

D-2.1.3 In case of filled tanker (sampling through bottom valve), suitable precooled glass bottle as sampling container may be used.

D-3 SCALE OF SAMPLING

D-3.1 Lot

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

D-3.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. For this purpose the number of containers to be selected from a lot shall depend on the size of the lot and shall be in accordance with the col 2 and 3 of Table 2.

D-3.3 These containers shall be selected at random from the lot. For this purpose, reference may be made to IS 4905. However, if this standard is not available, the following procedure may be followed:

Table 2 Number of Containers to be selected from Lots of Different Sizes

(*Clause* C-3.2)

Sl No.	Lot Size	No. of containers to be selected
	N	*n*
(1)	(2)	(3)
i.	Up to 2	All
ii.	3 to 15	2
iii.	16 to 25	3
iv.	26 to 100	5
v.	101 to 150	8
vi.	151 to 500	13
vii.	501 and above	20

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2.....up to r, where r is the integral part of the *N/n* (*N* being the lot size and *n* the number of containers to be selected). Every r^{th} container thus counted shall be withdrawn till the requisite number of containers is obtained.

D-4 PREPARATION OF TEST SAMPLES

D-4.1 From each of the containers selected according to **D-3.3**, a small representative portion of the material sufficient for carrying out tests as indicated in **3** shall be drawn with the help of the sampling tube (*see* **D-2.1**). These shall constitute the individual samples.

D-4.2 Out of these portions, equal quantity of material shall be taken and mixed thoroughly to form a composite sample, not less than 1 500 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third to be used as a referee sample.

D-4.3 The composite samples shall be transferred to containers of 600 ml capacity and shall be sealed and marked with full identification particulars given in **D-1.6**.

D-4.4 The referee test samples shall also bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two.

D-5 NUMBER OF TESTS

D-5.1 Test for 'total aldehydes (as acetaldehyde)' (as per Table 1) shall be performed on the individual sample.

D-5.2 Tests for the determination of all the remaining characteristics, specified in **3**, shall be performed on the composite sample.

D-6 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to the requirements of the specification, if the test results for each of the characteristic satisfy the relevant requirement as given in **3**. Otherwise, the lot shall be rejected.