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भारतीय मानक मसौदा

डाईएसीटोन अल्कोहल — विशिष्टि

(IS 2252 *का पांचवां पुनरीक्षण*)

Draft *Indian Standard*

DIACETONE ALCOHOL — SPECIFICATION

(*Fifth Revision of* IS 2252)

(ICS 71.080.80)

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09	Last date for comment **4 July 2025**

FOREWORD

(Formal clauses to be added later)

Diacetone alcohol is a pale straw colour liquid with non-residual odour. It is soluble in water. It is used as a solvent for variety of products, such as cellulose acetate, nitrocellulose, vinyl chloride, acetate resins, epoxy resins and also used for surface coating. Besides, it is used as a dyestuff solvent in the textile printing industry.

This standard was first published in 1962 and subsequently revised in 1972, 1988, 2001 and 2018. In the first revision, the requirement for acidity and water content were modified to reflect the improved quality of material. In order to limit the other impurities, such as acetone a new requirement for flash point was also introduced. In the second revision, the requirement for colour, refractive index and flash point were modified.

In the third revision, new requirement of distillation range was included. The requirement for residue on evaporation was replaced by the requirement for non-volatile matter. The requirements for water content and colour were modified. The requirements of flash point and refractive index were deleted. In the fourth revision, the requirement for colour was modified and the references were updated.

In this revision, the changes are as follows:

a) description of the material has been modified;
b) purity of the material determined by gas chromatography has been included as a new requirement to be tested;

c) requirement limits of characteristics including moisture content, colour, acetic acid and distillation range have been modified; and
d) packing and marking clause has been modified.

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, methods of sampling and test for diacetone alcohol suitable for industrial use.

2 REFERENCES

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

IS No.	*Title*
IS 229 : 2021	Ethyl acetate – Specification (*fourth revision*)
IS 878 : 2008/ISO 4788 : 2005	Laboratory glassware – Graduated measuring cylinders (*second revision*)
IS 1070 : 2023	Reagent grade water – Specification (*fourth revision*)
IS 2362 : 1993	Determination of water by Karl Fischer method – Test method (*second revision*)
IS 5298 : 2013	Method for determination of distillation range and distillation yield (*second revision*)
IS 8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale (*second revision*)

3 REQUIREMENTS

3.1 Description

Diacetone alcohol shall be a clear colourless liquid, free from sediments and shall consist essentially of 4-hydroxy-4-methylpentane-2-one (This name is approved by the International Union of Pure and Applied Chemistry) [$CH_3C(O)CH_2C(OH)(CH_3)_2$].

3.2 Miscibility with Water

It shall be miscible with water in all proportion.

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

Table 1 Requirements for Diacetone Alcohol
(*Clause* 3.3)

Sl No.	**Characteristic**	**Requirements**	**Method of Test, Ref to**	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Colour, Pt-Co scale, *Max*	15	—	8768
ii)	Relative density, 27 °C/27 °C	0.931 to 0.937	A	—
iii)	Distillation range[1]:			
	a) Initial boiling point[2], 760 mm Hg, *Min*	150 °C	B	—
	b) Dry point, 760 mm Hg, *Max*	170 °C		
iv)	Water content, percent by mass, *Max*	0.1	C	—
v)	Acidity (as acetic acid), percent by mass, *Max*	0.01	D	—
vi)	Non-volatile matter, g/100 ml, *Max*	0.01	E	—
vii)	Purity (by GC), percent by area, *Min*	99.5	F	—

[1] Optional parameter to be tested.

[2] In order to avoid an erratic value for the initial boiling point the distillation flask should be clean and free from any residual carbon deposit. This suggestion is specifically applicable to diacetone alcohol. Particular care should be given to the heating rate so that the initial boiling point is obtained within 5 min to 10 min.

3.4 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.

4 PRECAUTIONS IN HANDLING

4.1 Diacetone alcohol is considered as practically non-toxic but splashes in the eye cause irritation and can result in injury. Eye shields should be worn during handling operations. Splashes in the eye should be removed immediately by washing with copious quantities of water.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in suitable mild steel drums or aluminum drums or ISO containers or stainless-steel tankers, well closed with preferably replaceable closure or as agreed between the purchaser and the supplier, subject to the provisions of law in force at the time.

> NOTE — The container shall withstand untoward corrosion and shall be free from impurities harmful to the end use of the material.

5.2 Marking

5.2.1 Each container shall be suitably marked with the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trademark, if any;
c) Net mass of the material in the container;
d) Batch number or lot number, in code or otherwise;
e) Month and year of manufacture; and
f) Any other statutory requirement.

5.2.2 For supplies of material in bulk, a test certificate certified by authorized person of the manufacturer’s organization containing the details mentioned at **5.2.1** shall be provided for each consignment.

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

6.1 The procedure for sampling of the material shall be as prescribed in Annex F of IS 229.

6.2 Criteria for Conformity

The lot shall be declared as conforming to the standard, if the test results of the composite test sample satisfy the requirements prescribed. Otherwise the lot shall be rejected.

ANNEX A

[*Table* 1, *S1 No.* (ii)]

DETERMINATION OF RELATIVE DENSITY

A-1 OUTLINE OF THE METHOD

In this test method, the standard mass of equal volumes of the material and water are compared at 27 ºC.

A-2 APPARATUS

A-2.1 Relative Density Bottle — 25 ml capacity.

A-2.2 Water Bath — maintained at (27.0 ± 0.2) ºC.

A-2.3 Thermometer — any convenient thermometer of a suitable range with 0.1 °C or 0.2 °C subdivisions.

A-2.3.1 The thermometer shall bear a certificate from any institution authorized to issue certificate traceable to international or national measurement standards.

A-3 PROCEDURE

Clean and dry the relative density bottle. Weigh and fill with recently boiled and cooled water at 27 °C. Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in the water-bath. Keep the entire bulb covered with water and hold at that temperature for 30 min. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely, dry and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-4 CALCULATION

$$\text{Relative density at 27 °C/27 ºC} = \frac{M_1 - M_2}{M_3 - M_2}$$

where

M_1 = mass, in g, of the relative density bottle with the material;
M_2 = mass, in g, of the relative density bottle; and
M_3 = mass, in g, of the relative density bottle with water.

ANNEX B

[*Table* 1, *S1 No*. (iii)]

DETERMINATION OF DISTILLATION RANGE

B-1 PROCEDURE

Determine the distillation range by the procedure as prescribed in IS 5298 applying following corrections.

B-1.1 Correction for Thermometer Reading

B-1.1.1 *Error of Scale*

B-1.2 Correction for Barometric Pressure

If the barometric pressure prevailing during the determination is 760 mm Hg, no correction need be applied to the specified temperature and the thermometer scale as corrected for error of scale may be used as such. If however the prevailing barometric pressure deviates from 760 mm Hg, the specified temperature shall also be corrected as follows:

a) For every mm above 760 mm Hg, subtract 0.05 °C from the specified temperature; and
b) For every mm below 760 mm Hg, add 0.05 °C to the specified temperature.

NOTE — These corrections are valid only for pressure above 700 mm Hg.

ANNEX C

[*Table* 1, *S1 No*. (iv)]

DETERMINATION OF WATER CONTENT

C-1 GENERAL

Water is determined by the Karl Fischer method.

C-2 PROCEDURE

Take about 20 g of the material, weigh accurately and determine the water content by the procedure given in IS 2362.

ANNEX D

[*Table* 1, *S1 No*. (v)]

DETERMINATION OF ACIDITY (*as* ACETIC ACID)

D-1 OUTLINE OF THE METHOD

The material is titrated with standard sodium hydroxide solution to the phenolphthalein end point and from the volume of standard sodium hydroxide solution, used acidity is calculated as acetic acid.

D-2 REAGENTS

D-2.1 Sodium Hydroxide Solution — approximately 0.01 N.

D-2.2 Hydrochloric Acid — approximately 0.01 N.

D-2.3 Phenolphthalein Indicator — 0.5 percent (*m/m*) in *iso*-propyl alcohol or in rectified spirit.

D-2.4 Neutralized Freshly Boiled and Cooled Distilled Water — by adding 0.01 N sodium hydroxide solution (NaOH) drop-wise to the phenolphthalein indicator end point and further neutralize excess of sodium hydroxide solution by adding drop-wise 0.01 N hydrochloric acid till colourless.

D-3 PROCEDURE

Weigh accurately about 100 g of the material to the nearest 0.l g in a 500 ml conical flask. Add 50 ml of neutralized distilled water and few drops of phenolphthalein indicator. Titrate with 0.01 N sodium hydroxide solution using a microburette, until the pink colour persists for 15 s.

D-4 CALCULATION

$$\text{Acidity (\textit{as} acetic acid), percent by mass} = \frac{6 \times V \times N}{M}$$

where

V = volume, in ml, of standard sodium hydroxide solution;
N = normality of standard sodium hydroxide solution; and
M = mass, in g, of the material taken for the test.

ANNEX E

[*Table* 1, *S1 No.* (vi)]

DETERMINATION OF NON-VOLATILE MATTER

E-1 APPARATUS

E-1.1 Evaporating Dish, made of platinum, silica or borosilicate glass

E-1.2 Desiccator

E-1.3 Oven, capable of maintaining temperature up to 110 ºC

E-2 PROCEDURE

With a graduated measuring cylinder (*see* IS 878) introduce 100 ml of the sample into a 125 ml weighed to the constant mass, platinum, silica or borosilicate glass evaporating dish, previously heated to constant mass at a temperature of 105 ºC to 110 ºC and cooled in a desiccator. Evaporate the sample to dryness on a hot water bath and place the dish in an oven at a temperature of (100 ± 2) °C. Cool the dish in a desiccator and weigh along with non-volatile matter to the nearest 0.1 mg. The difference in mass in g gives g/100 ml of the non-volatile matter.

E-3 CALCULATION

$$\text{Non-volatile matter, g/100 ml} = M_1 - M_2$$

where

M_1 = mass, in g, of the evaporating dish with non-volatile matter; and
M_2 = mass, in g, of the empty evaporating dish.

ANNEX F

[*Table* 1, *S1 No*. (vii)]

DETERMINATION OF DIACETONE ALCOHOL CONTENT (PURITY) BY GAS CHROMATOGRAPHIC METHOD

F-1 OUTLINE OF THE METHOD

F-1.1 A representative sample is introduced into a gas chromatograph equipped 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.

F-2 APPARATUS

F-2.1 Gas Chromatograph

F-2.1.1 Any gas chromatograph equipped with a flame ionization detector (FID), a split injector and a suitable electronic integrator/software, capable of operating at the conditions listed below, may be used:

Column	:	fused silica capillary column with polyethylene glycol with length 30 m; internal diameter 0.25 mm and film thickness 0.5 µm or equivalent

Carrier gas : Nitrogen/Helium
Column flow, ml/min : 1.1
Purge flow, ml/min : 3
Split ratio : 1 : 100 or suitable to allow adequate sensitivity
Detector type : Flame ionization
Injection volume, µl : 0.2

F-2.1.2 *Temperature Programme of Oven, Detector and Injector*

Injector Temperature, °C	*Detector Temperature, °C*		*Oven*	
		Temperature, °C	*Hold Time, min*	*Ramp Rate, °C /min*
200	250	70	1.5	8
		225	10	—

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 techniques (internal standard, external standard, area normalization) may be used provided standardization / calibrations are done after setting up chromatographic conditions for required resolution.

F-3 REAGENTS

F-3.1 Diacetone Alcohol, certified reference material

NOTE — In case certified reference material of regents as mentioned at **F-3.1** is unavailable, high purity chemical (known purity) may also be used as an alternative to certified reference material.

F-4 DATA ACQUISITION SYSTEM

Any suitable data integrator or PC based gas chromatograph software.

F-5 IDENTIFICATION

Determine the retention time of each component by injecting small amounts of highly pure material of diacetone alcohol (*see* **F-3.1**) individually. Typical data of retention time is given in Table 2.

Table 2 Typical Retention Time and Area Percentage of Peaks
(*Clause* F-5)

Peak (1)	**Retention time, min** (2)	**Area** (3)	**Area percent** (4)
	2.773	51 726	0.174 19
	5.887	5 160	0.017 38
	6.147	6 078	0.020 47

	6.376	9 786	0.032 96
	8.206	1 579	0.005 32
	8.666	9 765	0.032 88
Diacetone Alcohol	10.457	2 96 00 662	99.682 83
	10.767	506	0.001 70
	12.015	1 082	0.003 64
	12.596	4 098	0.013 80
	15.289	1 629	0.005 49
	18.681	2 774	0.009 34
Total		2 96 94 645	100.000 00

F-6 PROCEDURE

Inject 0.2 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.

F-7 CALCULATION

F-7.1 Calculate concentration of diacetone alcohol:

$$\text{Diacetone alcohol, percent by area} = \frac{Area\ of\ diacetone\ alcohol\ peak\ in\ sample}{Sum\ of\ area\ of\ all\ peaks\ in\ sample} \times 100$$

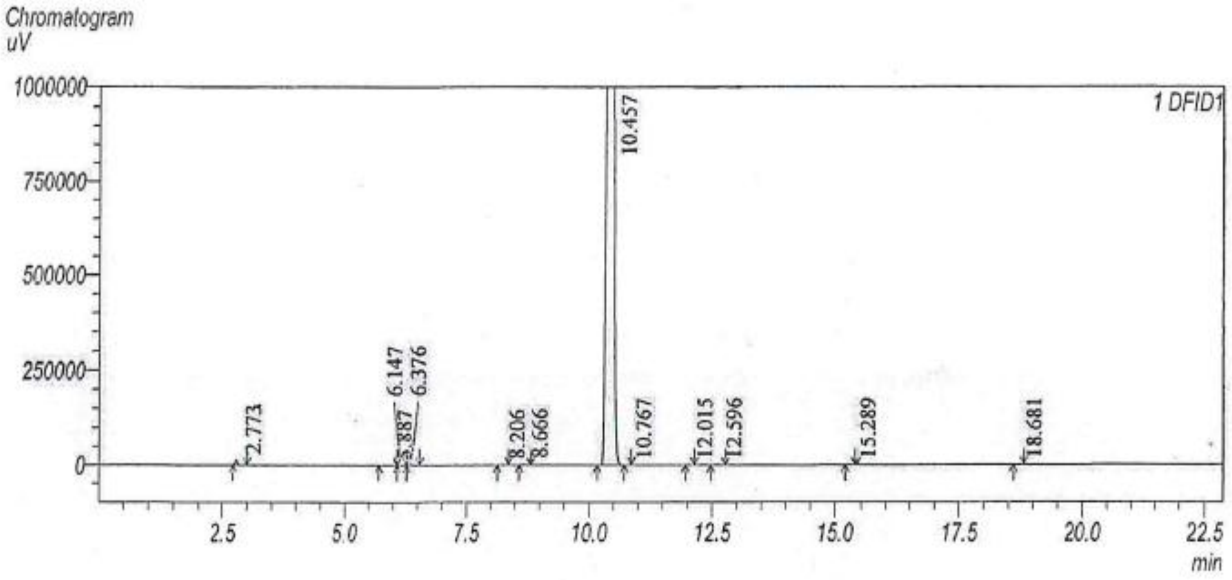

FIG. 1 TYPICAL CHROMATOGRAM