

IS 170 : 2004
(Reaffirmed 2015)

भारतीय मानक
ऐसीटोन — विशिष्टि
(चौथा पुनरीक्षण)

Indian Standard
ACETONE — SPECIFICATION
(*Fourth Revision*)

ICS 71.080.80

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

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Price Group 4

Alcohols and Allied Products Sectional Committee, PCD 10

FOREWORD

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

This standard was first issued in 1950 and subsequently revised in 1966, 1976 and 1986. At the time of first revision, a comparative study of the standard with the draft ISO Recommendation No. 656 'Sampling and methods of test for acetone' was undertaken. A revised scheme of sampling and criteria for conformity, a new procedure for determination of alcoholic impurities, refinement in the test for alkalinity and additional requirement for water content were included in the revised standard. As a result of experience gained through the use of first, second and third revisions and in order to meet the present day requirements of acetone, the fourth revision was undertaken. In this version requirements of purity and permanganate time have been incorporated using GC method for verification of purity. Requirement of residue on evaporation has been modified.

Acetone is an important commercial solvent and raw material with a wide usage in the chemical, explosives and lacquer industry. It is commonly used as a solvent for cellulose acetate; nitrocellulose; celluloid; cellulose ether; chlorinated rubber; various resins, fats and oils; and as an absorbent for acetylene. It is being increasingly used in the synthesis of a number of chemicals such as diacetone alcohol, methyl isobutyl ketone, phorone, isophorone, methyl methacrylate and certain resins, pharmaceuticals and perfumes.

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 off 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

ACETONE — SPECIFICATION

(Fourth Revision)

1 SCOPE

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

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute the 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 editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
82 : 1992	Method of sampling and test for thinners and solvent for paints (<i>first revision</i>)
250 : 1964	Potassium bichromate, technical and analytical reagent (<i>revised</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods (<i>first revision</i>)
1448 [P : 18] : 1991	Methods of test for petroleum and its products: [P : 18] Distillation (<i>second revision</i>)
2263 : 1979	Methods of preparation of indicator solutions (<i>first revision</i>)
2362 : 1993	Determination of water by the Karl Fischer method (<i>second revision</i>)
4825 : 1982	Specification for liquid-in-glass solid-stem reference thermometers (<i>first revision</i>)
4905 : 1968	Methods for random sampling
7445 : 1974	Code of safety for acetone
8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale (<i>second revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be clear and free from any suspended matter. It shall consist essentially of 2-propanone or dimethylketone (CH_3COCH_3).

3.2 Miscibility with Water

The material shall show no turbidity on mixing with distilled water in any proportion at $27 \pm 2^\circ\text{C}$.

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

4 PACKING AND MARKING

4.1 Packing

The material shall be supplied in sound and clean containers according to the marking and delivery instructions given by the purchaser.

4.2 Marking

Each container shall be marked legibly and indelibly with the following information:

- a) Name of the material;
- b) Manufacturer's name;
- c) Net, gross and tare mass;
- d) Recognized trade-mark, if any; and
- e) Date of packing.

4.2.1 Each container shall also be marked with the minimum cautionary notice worded as 'FLAMMABLE'.

4.2.1.1 It shall also bear the corresponding symbol for labelling of dangerous goods [see Fig. 5 of IS 1260 (Part 1)].

NOTES

1 Necessary safeguards against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion (see IS 7445).

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

4.2.2 BIS Certification Marking

The containers may also be marked with the BIS Certification Mark.

4.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to

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Table 1 Requirements for Acetone
(Clause 3.3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Purity, percent by mass, <i>Min</i>	99	A	—
ii)	Colour, Pt-Co scale, <i>Max</i>	10	—	8768
iii)	Relative density, at 27/27°C	0.784 to 0.786	—	6 of IS 82
iv)	Distillation range	The difference between initial boiling point (IBP) and dry point (DP) shall not exceed 1°C including 56.1°C (temperature being corrected ¹⁾ for a pressure of 760 mm Hg)	—	Method B in [P : 18] of IS 1448 with the thermometer conforming to the requirements given in Annex B of this standard
v)	Water content, percent by mass, <i>Max</i>	0.4	—	IS 2362
vi)	Residue on evaporation, mg/100 ml, <i>Max</i>	5	—	8 of IS 82
vii)	Acidity (as CH ₃ COOH), g/100 ml, <i>Max</i>	0.002	C	—
viii)	Alkalinity	To pass the test	D	—
ix)	Permanganate test, minutes, <i>Min</i>	30	E	—
x)	Alcoholic impurities	To pass the test	F	—

¹⁾In case the barometer pressure, *p*, deviates from 760 mm Hg, use the correction 0.037 (*p* - 760)°C for distillation temperature.

manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of drawing representative samples shall be as prescribed in Annex G.

5.2 Number of Tests

5.2.1 Individual Samples

Tests for distillation range and any other characteristic, if so agreed to between the parties, shall be carried out individually on each of the test samples in a set as prepared in G-4.1.

5.2.2 Composite Samples

Tests for determination of remaining characteristics of this standard shall be carried out on the composite sample as prepared in G-4.2.

5.3 Criteria of Conformity

5.3.1 For Composite Samples

The lot shall be declared as conforming to the requirements of this standard, if each of the test results on the composite sample according to 5.2.2 satisfies the corresponding requirements of this standard.

5.3.2 For each of the characteristics tested on the individual samples according to 5.2.1, the mean and the range of test results shall be calculated as below:

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

Range, R = difference between maximum and minimum values of test results.

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

6 TEST METHODS

6.1 Tests shall be conducted as prescribed in col 4 and 5 of Table 1.

6.2 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.

ANNEX A

[Table 1, Sl No. (i)]

METHOD FOR DETERMINATION OF PURITY

A-1 GENERAL

This test method is related to determination of the purity of acetone by gas chromatography and in addition, provides a means for identification of certain known impurities.

A-2 APPARATUS

A-2.1 Gas Chromatograph

Any gas liquid chromatograph equipped with TCD detector can be used with following accessories and operating conditions:

Column	: Chromosorb 101
Mesh size	: 80/100
Column length	: 3 m
I.D.	: 3 mm
Column tubing material	: Stainless steel (tubing must be non-reactive with substrate, sample and carrier gas)
Carrier gas	: Hydrogen (Purity — 99.99 mole percent, <i>Min</i>)
Syringe	: 10 microlitre
Sample size	: 0.2 microlitre
Electronic	: For computation of results integration

A-2.1.1 Instrument Condition

Detector	: TCD
Oven temperature	: 110°C
Injector, temperature	: 280°C
Detector, temperature	: 200°C
Carrier gas flow rate	: H ₂ , 30 ml/min

NOTES

1 TCD is preferred for acetone purity and impurities along with moisture content because moisture estimated by Karl Fischer is not applicable to acetone.

2 The above gas chromatographic conditions are suggestive. However, any GC with different column may be used provided standardization/calibration are done after setting up chromatographic conditions for the required resolution.

A-2.1.2 Identification, Calibration and Standardization

Install the column in the chromatograph. Set the conditions given above, of column temperature and carrier gas flow that gives the necessary resolution of the components in the sample being analyzed. Set the conditions as such which gives a minimum 10 percent

recorder deflection for a 0.1 percent concentration of purity at the most sensitive setting of instrument. Allow sufficient time for the instrument to reach equilibrium as indicated by stable base line. Adjust carrier gas flow rate to a constant value.

A-2.1.2.1 Identification

Determine the retention time of each component by injecting small amount either separately or in known mixture.

A-2.1.2.2 Calibration and standardization

The response of any given detector varies from one chemical to another. Therefore, calibration is must. Calibration allows the computation of response factor which express the relative response of different components to the detector.

Calibration standard is prepared containing the impurities present in acetone. During standard preparation the exact amounts of all the components is noted. Inject 0.2 µl of calibration standard in the chromatographic column in accordance with the specified conditions.

Calculate response factor to four decimal places for all components relative to acetone using following equation:

$$Rf_x = \frac{W_x \times AM}{A_x \times WM}$$

where

Rf_x = response factor for component x relative to acetone;

W_x = weight of component x, in the calibration standard;

A_x = area of component x, in the calibration run;
 WM = weight of acetone in the calibration standard;
 and

AM = area of acetone in the calibration standard run, which is selected as reference component.

The component which is unknown in the sample is assigned the relative response factor 1.000 0. It is advisable that the determination of response factors be made on the basis of duplicate analysis.

Response factor should be re-checked after any perceptible change in column or instruments performance.

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A-3 PROCEDURE

Inject 0.2 µl of sample into the chromatographic column, using the same conditions as for components identification and standardization. Record and integrate the chromatogram using appropriate attenuation setting that provide optimum peak heights. Measure the area of all the peaks.

R_x = peak response of compound x; and

f_x = relative response factor of compound x; and

$R_x \times f_x$ = sum of the individual component peak responses (R_x) multiplied by their relative response factors (f_x).

A-4 CALCULATION

Calculate the concentrations of sample components using the following equation:

$$C_x, \text{ percent} = \frac{f_x \times R_x}{R_x \times f_x} \times 100$$

where

C_x = compound x, weight, percent;

A-5 PRECISION AND BIAS

A-5.1 Repeatability

Two results, should be considered suspect if they differ by more than 0.000 6.

A-5.2 Reproducibility

Two results should be considered suspect, if they differ by more than 0.002 7.

ANNEX B

[Table 1, Sl No. (iv)]

REQUIREMENTS FOR THE THERMOMETER

B-1 THERMOMETER

B-1.1 Thermometer (Schedule Mark 22 of IS 4825) with the following requirements shall be used:

- Range : 48 to 102°C
- Immersion : 100 mm
- Graduations: :
 - 1) Subdivisions : 0.2°C
 - 2) Long lines at each : 1°C
 - 3) Number at each : 2°C
- Scale error, Max : 0.2°C
- Total length : 395 ± 5 mm

Outer diameter of stem : 6.0 to 7.0 mm

Bulb: :

a) Length : 15 to 20 mm

b) Outer diameter : Not greater than that of stem

Scale location: *Distance*

a) Bottom of bulb to line at 48°C : 125 to 145 mm

b) Bottom of bulb to line at 102°C : 335 to 360 mm

B-1.1.1 Any other thermometer of similar range and accuracy may also be used.

ANNEX C

[Table 1, Sl No. (vii)]

TEST FOR ACIDITY

C-1 REAGENTS

C-1.1 Phenolphthalein Indicator — prepared by dissolving 0.1 g of phenolphthalein in 100 ml of 60 percent ethyl alcohol.

C-1.2 Standard Sodium Hydroxide Solution — 0.01N.

C-2 PROCEDURE

C-2.1 Fill 100 ml of water and put a few pieces of clean porous porcelain in a 500-ml conical flask (non-alkaline glass) and boil gently for 5 min to eliminate carbon dioxide. Cool slightly and add 100 ml of the material. Boil gently for a further period of 5 min. At the end of this period, close the neck of the flask with a stopper carrying a sodalime tube, allow to cool. When cool, remove the stopper, add 0.5 ml of

phenolphthalein indicator and examine for alkalinity; if not alkaline, titrate with standard sodium hydroxide solution using a microburette.

C-3 CALCULATION

Acidity (as CH_3COOH), percent by mass = $\frac{6VN}{100D}$

where

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

N = normality of standard sodium hydroxide solution; and

D = relative density of the material at the temperature of determination.

ANNEX D

[Table 1, Sl No. (viii)]

TEST FOR ALKALINITY

D-1 REAGENTS

D-1.1 Hydrochloric Acid — approximately 0.02N by volume.

D-1.2 Sodium Hydroxide Solution — approximately 0.02N.

D-1.3 Bromothymol Blue Indicator Solution — prepared by dissolving 0.1 g of bromothymol blue in 100 ml of 50 percent rectified spirit (*see* IS 2263).

D-2 PROCEDURE

D-2.1 Take 200 ml of freshly boiled and cooled distilled water in a 500-ml conical flask (non-alkaline

glass). Add 2 drops (approximately 0.1 ml) of bromothymol blue indicator solution. Adjust to the neutral colour of the indicator by adding hydrochloric acid or sodium hydroxide solution as necessary. Transfer 100 ml of the neutralized water to each of two 250-ml cylinders. To one of these, add 100 ml of the sample and shake well. Compare the colour of the sample with that of the blank, viewing both downward to compensate for dilution.

D-2.2 The material shall be considered to have passed the test, if no change of colour towards blue, indicating the presence of alkalinity, takes place.

ANNEX E

[Table 1, Sl No. (ix)]

PERMANGANATE TEST

E-1 SCOPE

This method covers the detection of impurities in acetone, which reduce potassium permanganate.

E-2 SUMMARY OF METHOD

Substances reacting with potassium permanganate in neutral solutions reduce it to manganese dioxide and change the colour of solution. In the permanganate test, the time required for the colour of the test solution to change to that of a standard solution is measured. The colour of the test solution changes from pink to cobalt (II) chloride and uranyl nitrate colour standard.

E-3 APPARATUS

E-3.1 Two Matched Cylinders — Two matched cylinders, of capacity 100 ml, of colourless transparent glass, graduated at 50 ml and fitted with ground glass stoppers.

E-3.2 Constant Temperature Bath — Capable of maintaining a temperature of $25 \pm 0.2^\circ\text{C}$. It is important that the constant temperature bath be protected from direct light. If a glass constant temperature bath is employed, it should be wrapped or coated with an opaque material.

E-3.3 Pipette — Capable of delivering 2.0 ml solution.

E-3.4 Interval Timer or Clock — Capable of measuring a time interval of 120 min or more. An alarm arrangement is desirable.

E-4 REAGENTS

E-4.1 Potassium Permanganate Solution (0.2 g/l) — Use water previously boiled for 30 min with sufficient dilute potassium permanganate solution to give a stable faint colouration. Cool the water to ambient temperature

before preparation of the solution. Prepare this solution immediately before use and protect it from light.

E-4.2 Cobalt (II) Chloride and Uranyl Nitrate, Colour Standard Solution — To 5 ml of a 50 g/l solution of cobalt (II) chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), add 7 ml of a 40 g/l solution of uranyl nitrate hexahydrate [$\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], and dilute with water to 50 ml. (Prepare this solution on the day of use.)

E-5 PROCEDURE

Rinse one of the cylinders (*see E-3.1*), first with 15 to 20 ml of hydrochloric acid, of density approximately 1.19 g/ml, about 38 percent (*m/m*) solution, then six times with tap water, twice with distilled water and finally with sample. Immediately fill the cylinder to the mark with the sample at a temperature of about 25°C .

Rinse the second cylinder (*see E-3.1*), as specified above but omitting the last rinse with the sample. Fill the cylinder to the mark with the colour standard solution (*see E-4.2*). Place the cylinder containing test sample in the water bath maintained at $25 \pm 0.2^\circ\text{C}$, so that the water level in the bath is approximately 25 mm below the neck of the cylinder. After 15 min, remove the sample cylinder from the water bath and using 2 ml pipette add 2.0 ml of potassium permanganate solution. Note the time. Immediately stopper the cylinder, shake and replace in the water bath. Remove the cylinder from water bath, from time to time and compare the colour with standard colour solution, viewing vertically downwards against a white background.

E-6 REPORT

Report the time, in minutes, from the addition of the potassium permanganate solution, for the colour of the test solution to match that of the colour standard.

ANNEX F

[Table 1, Sl No. (x)]

TEST FOR ALCOHOLIC IMPURITIES

F-1 REAGENTS

F-1.1 Agulhon's Reagent — Dissolve 0.5 g of potassium bichromate, analytical reagent grade (conforming to IS 250) in 100 ml of nitric acid (relative density 1.33).

F-2 PROCEDURE

F-2.1 Take 1 ml of acetone in a test tube. Add 3 ml of

Agulhon's reagent. Mix and allow to stand at $27 \pm 1^\circ\text{C}$ for 5 min. Note if any blue or violet colour, indicating the presence of alcoholic impurities, is produced.

F-2.1.1 The material shall be deemed to have passed the test, if the test solution shows no blue or violet colouration.

ANNEX G

(Clauses 5.1, 5.2.1 and 5.2.2)

SAMPLING OF ACETONE

G-1 GENERAL REQUIREMENTS OF SAMPLING

G-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

G-1.2 Samples shall be taken in a protected area with good ventilation. Keep samples away from flame.

G-1.3 The sampling instrument shall be clean and dry.

G-1.4 The samples, the material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.

G-1.5 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.

G-1.6 The samples shall be placed in suitable, clean, dry and air-tight glass containers.

G-1.7 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

G-1.8 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with the manufacturer's name or trade-mark; month and year of manufacture of the material; batch number (if available); and other details of sampling, such as the date of sampling and sampler's name.

G-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

G-2 SAMPLING INSTRUMENTS

G-2.1 The following forms of sampling instrument may be used:

- Weighted sampling can for taking samples from various depths in large tanks, and
- Sampling tube.

G-2.2 Weighted Sampling Can, of suitable capacity, 500 to 1 000 ml and to 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. 1). 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 (*see* IS 82).

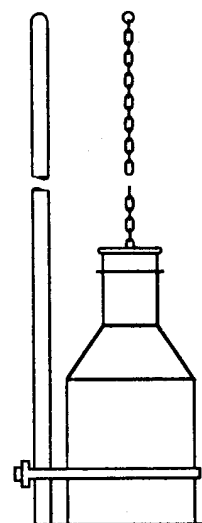
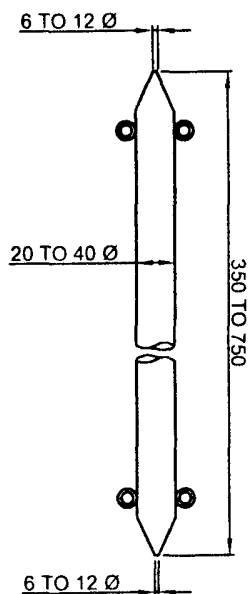


FIG. 1 SAMPLING BOTTLE OR CAN

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G-2.3 Sampling Tube, is made of metal or thick glass 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 internal diameter at the narrow ends. Handling is facilitated by two rings at the upper end. 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 while to admit the material and finally closed and withdrawn (see IS 82).



All dimensions in millimetres.

FIG. 2 SAMPLING TUBE FOR HOMOGENEOUS LIQUIDS

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

G-3 SCALE OF SAMPLING

G-3.1 Lot

In a single consignment, all the containers of the same size 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.

G-3.2 For ascertaining the conformity of the material in a lot to the requirements of this standard tests shall be carried out for each lot separately. For this purpose the number of containers to be selected from a lot shall be as given below:

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

G-3.2.1 The containers shall be chosen at random from the lot to ensure the randomness in selection. The procedure given in IS 4905 may be followed.

G-3.3 Sampling from Tanks or Vessels

Each of the tanks/vessels in the lot shall be sampled separately for determining the conformity of the lot to the requirements of the specification.

G-4 TEST SAMPLES AND REFEREE SAMPLES

G-4.1 Preparation of Sets of Samples for Testing Individually

Draw with an appropriate sampling instrument (see G-2), material from each freshly opened container and selected as in G-3.2. Keep these representative samples from different containers 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 clean 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 supplier. Keep the referee sample at a place agreed to between the purchaser and the supplier.

G-4.2 Preparation of Composite Sample

Draw with an appropriate sampling instrument (see G-2), equal portions of material from each container selected as in G-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 a thoroughly clean and dry sample container. 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 as agreed to between the purchaser and the supplier.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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AMENDMENT NO. 1 SEPTEMBER 2018
TO
IS 170 : 2004 ACETONE — SPECIFICATION

(Fourth Revision)

(Page 1, clause 2, line 10) — Substitute 'IS 82 : 1973' for 'IS 82 : 1992'.

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