# विनाइल एसीटेट मोनोमर — विशिष्टि

( पहला पुनरीक्षण )

# Vinyl Acetate Monomer — Specification

(First Revision)

ICS 71.080.70

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May 2024

Price Group 8

## FOREWORD

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

Vinyl acetate (CH<sub>3</sub>COOCH=CH<sub>2</sub>) monomer is obtained by the reaction of acetylene or ethylene and acetic acid in the presence of a catalyst. Another commercial route involves reactions of acetaldehyde and acetic anhydride to give ethylene diacetate (CH<sub>3</sub>COOCH<sub>2</sub>)<sub>2</sub> which, on thermolysis, gives vinyl acetate monomer. It is a colourless mobile liquid which spontaneously undergoes polymerization during shipment or in storage due to the catalytic effect of heat, light or oxygen. The commercial material is, therefore, stabilized (inhibited) with chemicals such as hydroquinone and/or diphenylamine, which may be separated by simple distillation. It is miscible in most organic solvents including chlorinated solvents but immiscible in water.

This chemical is a major raw material for vinyl plastics. Polyvinyl acetate (PVA) is obtained by polymerization of vinyl acetate with peroxide catalysts which is used in adhesives, protectives films, paper, lacquers and inks, plastic wood and emulsion paints. Partial hydrolysis of polyvinyl acetate yields polyvinyl alcohol which is used for production of polyvinyl acetal resins by condensation with aldehydes.

The standard was originally published in 1988. In this revision, alternate method for determination of aldehyde, distillation range and specific gravity have been incorporated. The amendment no. 1 and 2 issued to 1988 version, have also been incorporated in this revision.

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

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.

# Indian Standard

# VINYLY ACETATE MONOMER — SPECIFICATION

(First Revision)

## **1 SCOPE**

This standard prescribes the requirements, methods of sampling and testing for vinyl acetate monomer.

## **2 REFERENCES**

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The standards given below 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 edition of these standards:

**m**• .1

IS No.	Title
IS 82 : 1973	Methods of sampling and test for thinners and solvents for paints ( <i>first revision</i> )
IS 1070 : 2023	ReagentGradeWaterSpecification (fourth revision)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
IS 1448 (Part 18) : 2020	Methods of test for petroleum and its products: Part 18 Distillation of petroleum products ( <i>third revision</i> )
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (second revision)
IS 2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification ( <i>third revision</i> )
IS 4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures ( <i>first revision</i> )
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 ( <i>second revision</i> )
IS 15464 : 2022	Anhydrous ethanol for use as blending component in motor gasoline — Specification ( <i>first</i> <i>revision</i> )

## **3 REQUIREMENTS**

**3.1** The material shall be clear, colourless liquid, free from any suspended matter. The material may contain suitable quantities of stabilizer.

NOTE — Hydroquinone (5 ppm to 20 ppm) and/or diphenylamine (up to 250 ppm) are generally added as stabilizers to avoid polymerization in quantities according to length of storage desired.

**3.2** The material shall also comply with the requirements given in <u>Table 1</u>, when tested according to the methods referred in col (4) and col (5) of Table 1.

#### 3.3 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 PACKING AND MARKING**

#### 4.1 Packing

The material shall be packed in mild steel drums (*see* IS 2552) or in any other suitable containers as agreed to between the purchaser and the supplier. Each container shall be securely closed.

## 4.2 Marking

**4.2.1** Each container shall be marked with the following:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Gross, tare and net mass;
- d) Month and year of manufacture.
- e) Shelf life of the material; and
- f) Any other statutory requirements.

**4.2.2** Each container shall bear the caution label '**FLAMMABLE**' together with the corresponding symbol for labelling of dangerous goods in

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accordance to IS 1260 (Part 1). The container shall also be labelled with words as under:

## VAPOURS AND LIQUID DANGEROUS TO EYES

NOTE — Packing and marking for vinyl acetate monomer shall also comply with the provisions of law currently in force.

#### 4.2.3 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.

## **5 SAMPLING**

The procedure for sampling and criteria for conformity of the material shall be as prescribed in Annex G.

## **Table 1 Requirements for Vinyl Acetate Monomer**

## (*Clauses* <u>3.2</u>, <u>G-4.3</u>, <u>G-5.1</u> and <u>G-5.2</u>)

Sl No.	Characteristic	Requirement	Method of test, Ref to	
(1)	(2)	(3)	Annex (4)	Indian Standard (5)
i)	Acidity (as acetic acid), percent by mass, <i>Max</i>	0.01	<u>A</u>	
ii)	Vinyl acetate content (as ester), percent by mass, <i>Min</i>	99.8	<u>B</u>	—
iii)	Aldehyde (as acetaldehyde), percent by mass, <i>Max</i>	0.03	Method B of Annex $\underline{B}^{1}/\underline{C}$	_
iv)	Moisture, percent by mass, Max	0.05	—	IS 2362
v)	Distillation range at 760 mm of Hg	Not less than 97 ml shall distil in the range of 71 °C to 73.5 °C	_	IS 1448 (Part 18)/ IS 5298 <sup>1)</sup>
vi)	Colour, Pt-Co, Max	5		IS 8768
vii)	Specific gravity, 20 °C/20 °C	0.933 5 to 0.934 5	—	6 of IS 82/A-3 of IS 15464
viii)	Inhibitor content:			
	a) Hydroquinone, ppm and/or	5 to 20	<u>D</u>	—
	b) Diphenylamine, ppm	up to 250	<u>E</u>	
ix)	Polymer content, ppm, Max	5	<u>F</u>	

<sup>&</sup>lt;sup>1)</sup> In case of disputes, Method B of Annex B for determination of aldehyde, IS 5298 for determination of distillation range and A-3 of IS 15464 for determination of specific gravity shall be the referee methods.

## ANNEX A

## [*Table 1, Sl No. i*] and <u>B-2.5</u>]

## DETERMINATION OF ACIDITY (AS ACETIC ACID)

#### **A-1 OUTLINE OF THE METHOD**

This method describes a procedure for determining the acidity in vinyl acetate. The method is applicable to acidity determination up to 0.04 percent by mass. In this procedure, the sample is mixed with methanol and titrated at a reduced temperature with standard sodium hydroxide solution using bromothymol blue indicator.

#### **A-2 REAGENTS**

A-2.1 Bromothymol Blue Indicator — 0.1 percent

Dissolve 1 g of bromothymol blue indicator in methanol and make it up to 1 litre.

A-2.2 Methanol — reagent grade

A-2.3 Standard Sodium Hydroxide Solution — 0.05 N

## **A-3 PROCEDURE**

A-3.1 Place 50 ml of methanol in a 250 ml Erlenmeyer flask containing a small amount of

crushed ice. Add 1 ml of bromothymol blue indicator and titrate with standard sodium hydroxide solution to a dark green end point.

NOTE — Sufficient ice should be used so that some ice remains in the flask after titration.

**A-3.2** Add 100 ml of the sample and titrate rapidly with standard sodium hydroxide solution until the first green end point.

## A-4 CALCULATION

Calculate the acidity (as acetic acid) as follows:

Acidity (as CH<sub>3</sub>COOH ), percent by mass = 
$$\frac{V \times N \times 0.060 \text{ 05}}{S}$$

where

- V = volume, in ml, of standard sodium hydroxide;
- N = normality of standard sodium hydroxide solution; and

S = specific gravity of the sample.

#### ANNEX B

## [*Table 1, Sl No. ii*)]

## DETERMINATION OF VINYL ACETATE CONTENT (AS ESTER)

#### **B-1 GENERAL**

Two methods have been prescribed, namely, Method A ester value method and Method B gas chromatography method. In case of dispute, Method B shall be the referee method.

#### **B-2 METHOD A — ESTER VALUE METHOD**

#### **B-2.1** Outline of the Method

A known amount of the material is digested with excess standard sodium hydroxide solution. From the amount of the standard sodium hydroxide solution consumed, the percent vinyl acetate equivalent in the sample is calculated.

#### **B-2.2** Apparatus

**B-2.2.1** *Lung-ray Pipette* — of the shape and dimensions shown in Fig. 1.

#### **B-2.3 Reagents**

**B-2.3.1** Standard Sodium Hydroxide Solution — 1 N

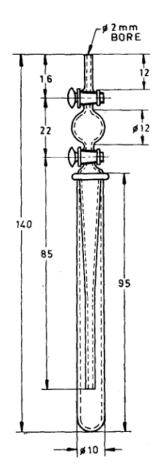
B-2.3.2 Standard Sulphuric Acid Solution - 1 N

#### B-2.3.3 Phenolphthalein Indicator Solution

Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent ethyl alcohol and add solution of sodium hydroxide until the colour turns faint pink.

#### **B-2.4 Procedure**

Take 30 ml of standard sodium hydroxide solution (1.0 N) accurately from a burette in a 250 ml iodine flask. Add about 50 g of crushed ice to the flask. Then add approximately 2 g (accurately weighed) sample with the help of lung-ray pipette to the contents of the flask and stopper the flask immediately. Shake the flask for 20 min and wash the sides and the stopper of the flask with distilled water. Titrate the excess sodium hydroxide solution (*see* <u>B-2.3.2</u>) using phenolphthalein as indicator. Run a blank in exactly a similar way without taking the sample.



All dimensions in millimeters. FIG. 1 LUNG-RAY PIPETTE

#### **B-2.5** Calculation

Calculate the percentage of vinyl acetate content in the sample using the formula and deduct the vinyl acetate equivalent for any acidity present as determined in <u>Annex A</u>.

Vinyl acetate content, percent by mass

$$=\frac{(V_1 - V_2) \times N \times 8.609}{M} - (1.434 \times P)$$

where

- $V_I$  = volume, in ml, of standard sulphuric acid solution required for the blank;
- $V_2$  = volume, in ml, of standard sulphuric acid solution required for the test;

- N = normality of standard sulphuric acid solution;
- M = mass, in g, of the material taken forthe test; and
- P = percentage of acetic acid as determined in Annex A.

# **B-3 METHOD B — GAS CHROMATOGRAPHY METHOD**

## **B-3.1** Outline of the Method

A known amount of internal standard is added to the sample and an aliquot portion of this mixture is introduced into the separating column of a gas chromatograph by means of a syringe. The vaporized sample is swept through a column by a flow of carrier gas and as each component emerges, it is detected by a flame ionization detector and recorded as a peak in a chromatogram. Under the operating conditions, the impurities, such as acetaldehyde, acetone, methyl acetate, ethyl acetate and acrolein are identified by their relative retentions and are quantitatively determined from their peak areas relative to the peak area of the internal standard.

#### **B-3.2 Reagents**

B-3.2.1 Acetaldehyde

B-3.2.2 Acrolein

B-3.2.3 Acetone

B-3.2.4 Methyl Acetate

B-3.2.5 Vinyl Acetate

B-3.2.6 Ethyl Acetate

#### **B-3.3** Apparatus

#### B-3.3.1 Gas Chromatograph

Any commercially available gas chromatograph with a flame ionization detector, a split/splitless

injector and a suitable electronic integrator/software may be used.

NOTE — It is recommended that maximum resolution should be obtained in the injection column. If an inlet splitter is fitted to the chromatograph, poor resolution may result from blocking it at the vent.

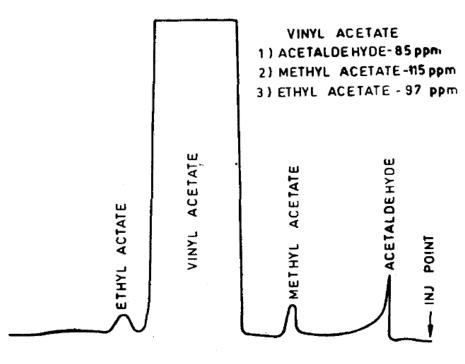
#### A-3.4 Procedure

**A-3.4.1** A typical set of operating conditions in the column is given below:

i)	Column	
	a) Length	400 mm
	b) Outer diameter	3 mm
	c) Material	Stainless steel
	d) Packing	Porapak QS of
	material	50/80 mesh
ii)	Carrier gas	Nitrogen
iii)	Carrier gas flow rate,	50
	ml/min	
iv)	Air flow rate, ml/min	400 to 500
v)	Hydrogen flow rate,	40
	ml/min	
vi)	Sensitivity	$1 imes 10^{-10}$
vii)	Sample size, µl	1
viii)	Temperature, °C	
	a) Oven,	150
	isothermal	
	b) Injection port	155
	c) Detector block	140

NOTE — The above gas chromatographic conditions are suggestive. However, any GC having difference in detector, column packing material and type (packed/capillary having different length/diameter/film thickness etc) 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.

**A-3.4.2** The chromatographic conditions given above are for guidance only. Variations in conditions are possible to achieve separation of constituents by this method. A typical chromatogram obtained with the above operating conditions is shown in Fig. 2.





## ANNEX C

## DETERMINATION OF ALDEHYDE CONTENT (AS ACETALDEHYDE)

#### **C-1 GENERAL**

This method covers determination of aldehyde (as acetaldehyde) present in the sample by reacting with excess sodium metabisulphite and titrating it with sodium iodide solution.

## **C-2 APPARATUS**

C-2.1 Burette — 50 ml, graduated in 0.1 ml subdivisions

C-2.2 Erlenmeyer Flask — 500 ml, glass stoppered

C-2.3 Pipette — 50 ml and 100 ml capacity

#### **C-3 REAGENTS**

#### C-3.1 Iodine Standard Solution — 0.1 N

Dissolve 35.0 g of potassium iodide and 13 g iodide in water and dilute it up to 1 litre with water. The solution as prepared is stored in dark bottle and standardized, as required, against 0.1 N standard sodium thiosulfate solution by the procedure as given below:

Take approximately 40 ml 0.1 N iodine solution, which is to be standardized, in 250 ml Erlenmeyer flask. Add 50 ml water to the flask and titrate it with standardized 0.1 N standard sodium thiosulphate. Calculate normality of iodine solution as follows:

Normality of iodine solution = 
$$\frac{V \times N}{V_1}$$

where

- *V* = volume, in ml, of sodium thiosulfate solution used for titrating of iodine solution;
- N = normality of sodium thiosulfate solution; and
- $V_1$  = volume, in ml, of iodine solution used, in ml.

C-3.2 Sodium metabisulfite Solution — 0.44 percent

Dissolve 4.4 g of sodium metabisulfite in 1 litre of water.

NOTE — Sodium metabisulfite solution is to be prepared fresh daily or before using.

#### **C-3.3 Starch Indicator**

Take 6 g of powdered soluble starch in water and dilute it up to 1 litre with water. Stir it and while stirring, add 20 g of potassium hydroxide (KOH) pellets. Continue stirring until the potassium hydroxide is dissolved. Leave the solution for 2 h and add 27.5 ml of hydrochloric acid and maintain the *p*H of mixture to  $6.0 \pm 0.1$ . Add 6 ml of glacial acetic acid as a preservative.

#### **C-4 PROCEDURE**

**C-4.1** Take 50 ml sodium metabisulfite solution (C-3.2) into each of two 500 ml Erlenmeyer flask. To each flask add 25 g clean ice. Now to one flask, add 100 ml of sample and the other to be used as blank. Close the flasks with stoppers and place the flasks in mechanical shaker for 10 min  $\pm$  1 min.

**C-4.2** After 10 min, add 0.5 ml of starch indicator to both solutions, sample solution as well as blank. Titrate the excess sodium metabisulfite with the standard iodine solution. The end point is when the first permanent blue colour is obtained.

#### **C-5 CALCULATION**

Aldehyde (as acetaldehyde), percent by mass

$$=\frac{(B-V_2) \times N_1 \times 0.022\ 02}{(D \times V_3)} \times 100$$

where

- B = volume, in ml, of iodine used for the titrating blank solution;
- $V_2$  = volume, in ml of iodine used for the titrating sample solution;
- $N_1$  = normality of the iodine solution;
- D = specific gravity of sample; and
- $V_3$  = volume of sample used, in ml.

#### ANNEX D

## [*Table 1, Sl No.* viii), a)]

## DETERMINATION OF HYDROQUINONE CONTENT

#### **D-1 OUTLINE OF THE METHOD**

This method describes a procedure for determining the hydroquinone content in the range of 1 ppm to 20 ppm. In this procedure, the vinyl acetate is evaporated either by air-drying at room temperature or vacuum evaporation at 50 °C. The resulting hydroquinone residue is dissolved in water and titrated with dilute standardized ceric acid sulphite using diphenylamine indicator.

#### **D-2 APPARATUS**

#### **D-2.1 Rotary Film Evaporator**

## **D-3 REAGENTS**

## **D-3.1 Standard Solution of Ceric Acid Sulphite** - 0.002 N

Dissolve 1.09 g of ceric ammonium nitrate  $[(NH_4)_2Ce(NO_3)_6]$  in 28 ml concentrated sulphuric acid in a 50 ml beaker. Slowly pour the ceric solution, while stirring, into 200 ml water contained in a 500 ml beaker. When the solution is complete, transfer this mixture to a 1 litre volumetric flask and dilute it to mark with water.

#### **D-3.2 Diphenylamine Indicator**

Dissolve 0.1 g of diphenylamine in 100 ml concentrated sulphuric acid. Store this solution in a brown bottle.

#### **D-3.3 Standard Hydroquinone Solution**

Dissolve 0.2 g of hydroquinone weighed nearest to 0.1 mg in water and dilute to 1 litre in a volumetric flask. This solution is unstable and should be discarded after one week of normal use.

#### **D-4 PROCEDURE**

D-4.1 Pipette 50 ml of vinyl acetate into each of two

125 ml flasks. Connect the flasks to rotary film evaporator and apply vacuum. Keep the flask in a hot water bath maintained at 50 °C  $\pm$  5 °C. Evaporate the sample to dryness. Do not exceed a temperature of 55 °C or 30 min of evaporation time. Carefully repressure the flask and disconnect. Displace any remaining vinyl acetate vapours by carefully purging the flask with dry air. Dissolve the residual hydroquinone in 25 ml water. Add 3 drops of diphenylamine indicator using the same dropper every time. Titrate each solution with 0.002 N standard ceric acid sulphite to a faint blue end point which persists for 15 s.

**D-4.2** Pipette 10 ml of standard hydroquinone solution (D-3.3) into each of two 125 ml Erlenmeyer flasks. Add 3 drops of diphenylamine indicator using the same dropper as used for the test and titrate with 0.002 N standard ceric acid sulphite solution to a faint blue end point that persists for 15 s.

NOTE — Duplicate titrations should agree within 0.05 ml. Use the average of the two values.

#### **D-5 CALCULATION**

The hydroquinone content in ppm is calculated as follows:

Hydroquinone content, ppm =  $\frac{V \times F}{M} \times 1000$ 

where

V	=	volume, in ml, of ceric acid
		sulphite solution used to titrate
		the sample;
F (Factor)	=	(mg of hydroquinone
(		$\frac{\text{in 10 ml of sample}}{(\text{volume of corig acid sulphito to})}$ ; and
		(volume of ceric acid sulphite to , and
		titrate the standard)
М	=	mass, in g, of sample taken.

#### **D-6 REPORT**

Report the hydroquinone content to nearest 0.1 ppm.

## ANNEX E

### [*Table* 1, *Sl No.* viii), b)]

## DETERMINATION OF DIPHENYLAMINE CONTENT

## **E-1 OUTLINE OF THE METHOD**

This method describes a procedure for determining the diphenylamine content in vinyl acetate by measuring the absorbance of diphenylamine in methanol.

## **E-2 APPARATUS**

**E-2.1 Spectrophotometer** — equipped with hydrogen lamp and matched 10 ml silica cells.

#### E-2.2 Rotary Film Evaporator

#### E-3 REAGENTS

#### E-3.1 Diphenylamine Stock Solution

Weigh approximately 0.09 g of inhibitor grade diphenylamine to the nearest 0.1 mg and dissolve in 1 litre of methanol. Calculate the parts per million of diphenylamine in the stock solution. This stock solution will contain approximately 100 ppm of diphenylamine.

#### E-3.2 Methanol — reagent grade

#### **E-4 PROCEDURE**

**E-4.1** Pipette 10 ml of sample into a 125 ml Erlenmeyer flask. Connect the flask to a rotary film

evaporator and evaporate to dryness under vacuum in a hot water bath maintained at  $(50 \pm 5)$  °C. Carefully repressurize and disconnect the flask. Displace any remaining vinyl acetate vapours by carefully purging the flask with dry air. Dissolve the residual diphenylamine in 50 ml of methanol. Transfer the methanol solution of diphenylamine to one of the two matched 10 ml silica cells. Determine the optical density by spectrophotometer at a wavelength of 315 nm using methanol as a blank.

NOTE — Do not exceed temperature of 55  $^{\circ}\mathrm{C}$  or 30 min of evaporation.

**E-4.2** Prepare standards by placing 10 ml, 20 ml, 40 ml, 60 ml and 80 ml of the diphenylamine stock solution in 100 ml volumetric flasks. Make up to the mark with methanol stopper and mix well. Calculate the parts per million for each standard. Determine the optical density of the standards at a wavelength of 315 nm using methanol as blank.

NOTE — The data, when plotted on a graph paper, should be a straight line.

#### **E-5 CALCULATION**

Read the parts per million of diphenylamine present in the sample from the working curve and multiply by five or calculate from the slope of the curve.

Diphenylamine, 
$$ppm = \frac{optical density \times 5}{slope of the curve}$$

## ANNEX F

## [*Table 1, Sl No.* ix)]

## **DETERMINATION OF POLYMER CONTENT**

#### **F-1 OUTLINE OF THE METHOD**

This method describes a procedure for determining the polymer content of vinyl acetate by evaporating the sample to dryness under vacuum at 50 °C. The non-volatile residue is polymer content. Any inhibitor present is included in the value measured for polymer content and thus to be subtracted during calculation.

#### **F-2 APPARATUS**

#### F-2.1 Rotary Film Evaporator

## **F-3 PROCEDURE**

**F-3.1** Determine the mass of a 125 ml Erlenmeyer flask nearest to 0.1 mg. The flask must be dried in an oven at 110 °C  $\pm$  5 °C and then cooled in a desiccator before weighing. Pipette 50 ml of material into the flask, connect the flask to a rotary film evaporator before placing it in a hot water bath maintained at 50 °C  $\pm$  5 °C. Apply vacuum, and

evaporate to dryness at a temperature not exceeding 55 °C and within 30 min. Repressurize the flask carefully and disconnect.

**F-3.2** Displace any remaining vinyl acetate vapours by carefully purging with dry air. Cool the flask in a desiccator and weigh to the nearest 0.1 mg.

## **F-4 CALCULATION**

Calculate the polymer content by using the following formula:

Polymer content, percent by mass

$$= \left[\frac{M_2 - M_1}{S} \times 2\right] - \frac{P}{10\,000}$$

where

- $M_1$  = mass, in g, of the flask;
- $M_2$  = mass, in g, of the flask and residue after evaporation;
- S = relative density of the material; and
- P = hydroquinone or diphenylamine content, in ppm, present in the material.

## ANNEX G

## (<u>Clause 5</u>)

## SAMPLING OF VINYL ACETATE MONOMER

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

**G-1.3** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**G-1.4** The samples shall be placed in clean, dry and air tight amber glass bottle or other suitable containers on which the material has no action.

**G-1.5** The sample containers shall be of such a size that they are almost completely filled by sample.

**G-1.6** Each sample container shall be sealed airtight after filling, and marked with full details of sampling, date of sampling and year of manufacture of the material.

## **G-2 SCALE OF SAMPLING**

#### G-2.1 Lot

All the containers in a single consignment of the material 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 drums, the drums belonging to the same batch and size shall be grouped together and such group shall constitute a separate lot.

**G-2.2** Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of drums to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with co1 (1) and (2) of Table 2.

**G-2.3** The drums shall be chosen at random from the lot and in order to ensure the randomness of selection, the procedure given in IS 4905 may be followed.

## G-3 PREPARATION OF TEST SAMPLES AND REFEREE SAMPLES

**G-3.1** A small representative portion of the material from different parts of the drums (G-2.2) shall be drawn with the help of a suitable sampling instrument. These portions of the material shall be thoroughly mixed. The quantity of the material taken from a sampled drum shall be Sufficient for making triplicate determinations for all the characteristics given in the specification.

**G-3.2** Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to form a composite sample. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

**G-3.3** the remaining portion of the material from each of the drums shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the drums sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

## Table 2 Scale of Sampling

## (<u>Clause G-2.2</u>)

Sl No.	Lot Size	No. of Drums to be Selected
	N	n
(1)	(2)	(3)
i)	Up to 15	3
ii)	16 to 25	4
iii)	26 to 50	5
iv)	51 and above	6

**G-3.4** All the individual and composite samples shall be transferred to separate containers and shall be sealed and marked with full identification, vinyl acetate content if the following condition is particulars given in **G-1.5** satisfied.

**G-3.5** The referee test samples consisting of a composite sample and a test of individual samples shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

## **G-4 NUMBER OF TESTS**

**G-4.1** The material from all the drums shall be visually examined for colourlessness and for any suspended matter.

**G-4.2** Tests for determination of vinyl acetate content, percent by mass, shall be conducted on individual sample.

**G-4.3** Test for the determination of all the remaining characteristics given in <u>Table 1</u> shall be conducted on the composite sample.

## **G-5 CRITERIA FOR CONFORMITY**

## **G-5.1 For Individual Samples**

The lot shall be declared as conforming to the

requirements of vinyl acetate content if the following condition is satisfied:

From the test results of individual sample, average  $(\bar{x})$  and range (*R*) shall be calculated.

Average 
$$(\bar{x}) = \frac{\text{sum of the test result}}{\text{number of test}}$$

where

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

The lot shall be declared as conforming to the requirements of vinyl acetate content, if the expression.

 $\bar{x} - 0.6 R \ge$  the minimum requirement given in Table 1.

#### **G-5.2 For Composite Samples**

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results for each of characteristics shall satisfy the relevant requirements given in <u>Table 1</u>.

## ANNEX H

## (<u>Foreword</u>)

## COMMITTEE COMPOSITION

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

Organization	Representative(s)
National Chemical Laboratory (NCL), Pune	DR C. V. RODE ( <i>Chairperson</i> )
Alkyl Amines Chemicals Limited, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE ( <i>Alternate</i> )
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (Alternate)
All India Distillers Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Limited, Mumbai	SHRI KIRAN BHAT SHRI HEMAL ( <i>Alternate</i> )
Chemical and Petrochemicals Manufacturers Association (CPMA), New Delhi	Shri Uday Chand
CSIR - Central Drug Research Institute (CDRI), Lucknow	Dr Sanjeev Kanojiya
Deepak Fertilizer, New Delhi	DR L. B. YADAWA Shri Suresh Amle ( <i>Alternate</i> )
Deepak Phenolics Limited, Vadodara	Shri Dharmesh Siddhapuria Shri Sandip Kumar Pandya ( <i>Alternate</i> )
Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizers, New Delhi	SHRI O. P. SHARMA SHRI VARUN SINGH POONIA ( <i>Alternate</i> )
Dow Chemical International Private Limited, Mumbai	SHRI V. MOHANDOSS SHRI GOVIND GUPTA ( <i>Alternate</i> )
Godavari Biorefineries, Mumbai	SHRI SHANUL LAXMANRAO PAGAR Shri Appasaheb J. Wani ( <i>Alternate</i> )
Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad	DR R. M. PATEL SHRI C. S. PATEL (Alternate)
Hindustan Organic Chemicals Limited (HOCL), Mumbai	SHRI DELEEP KUMAR K. DR B. RAJEEV (Alternate)
India Glycols Limited, Uttarakhand	DR R. K. SHARMA SHRI ALOK SINGHAL ( <i>Alternate</i> )
Indian Chemical Council (ICC), Mumbai	Dr Mritunjay Chaubey Shri J. Sevak ( <i>Alternate</i> )

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Organization	Representative(s)
Indian Oil Corporation Limited, Panipat	DR Y. S. JHALA
Jubilant Agri and Consumer Products Limited, Gurugram	Dr Kanak Baran Dass
Jubilant Life Sciences Limited, Noida	Shri Mansukh D. Kanzariya
Laxmi Organic Industries, Mumbai	SHRI KRISHNA A. RAO SHRI KAMLESH FULCHAND SHINDE ( <i>Alternate</i> )
National Chemical Laboratory (NCL), Pune	Dr Ravindar Kontham Dr Udaya Kiran Marelli (Alternate)
Reliance India Limited (RIL), Mumbai	SHRI SREERAMACHANDRAN KARTHA SHRI VASANT WARKE ( <i>Alternate</i> )
United Phosphorus Limited (UPL), Mumbai	SHRI M. D. VACHHANI
In Personal Capacity (37 Nandanvan Society, Near GNFC Township, Narmadanagar, Bharuch – 392015)	Dr Mayur J. Kapadia
BIS Directorate General	SHRIMATI MEENAL PASSI, SCIENTIST 'F'/ SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS) [REPRESENTING DIRECTOR GENERAL ( <i>Ex-officio</i> )]

Member Secretary MS ADITI CHOUDHARY SCIENTIST 'B'/ASSISTANT DIRECTOR (PETROLEUM, COAL AND RELATED PRODUCTS), BIS this Page has been intertionally left blank

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This Indian Standard has been developed from Doc No.: PCD 09 (22405).

## **Amendments Issued Since Publication**

Amend No.	Date of Issue	Text Affected

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