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मानक

IS 13601 (1993): Ethylene vinyl acetate (EVA) copolymers

for its safe use in contact with foodstuffs, pharmaceuticals and drinking water [PCD 12: Plastics]



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## भारतीय मानक

खाद्य सामग्रियों, भेपज सामग्रियों और पेय जल के सम्पर्क में निरापद उपयोग के लिए इथाइलीन विनायल एसीटेट ( इविए ) कोपालिमर — विशिष्टि

Indian Standard

ETHYLENE VINYL ACETATE (EVA) COPOLYMERS FOR ITS SAFE USE IN CONTACT WITH FOODSTUFFS, PHARMACEUTICALS AND DRINKING WATER — SPECIFICATION

UDC 678 : 742-134·442·2 [ 663/664 : 614·8 ]

## BIS 1993

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

**Price Group 4** 

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Plastics are now being used on a large scale for packaging of foodstuffs and pharmaceuticals. Where direct contact occurs between the packed commodity and the plastics, the high molecular mass polymer itself does not pose a toxic hazards being inert and essentially insoluble in food. There is, however, a likelihood that some transfer may occur of polymer additives, adventitious impurities such as monomers, catalyst remnants and residual polymerization solvents and of low molecular mass polymer fractions from the plastics into the packaged material with consequent toxic hazard to the consumers. The occurrence of acute toxicity due to plastics material in contact with food is most unlikely, since only trace quantities of potentially toxic materials are likely to migrate. However, the accumulation of these toxic materials with time may lead to hazards which may be serious.

Ethylene vinyl acetate copolymers, commonly referred to as EVA copolymers or simply as EVA, may be produced to cover the complete range of comonomer ratios form 99: 1 (ethylene: vinyl acetate) to 1:99. However, copolymers containing up to 50 percent vinyl acetate are classified as thermoplastics. EVA copolymers are produced commercially by continuous bulk and solution polymerization process.

EVA copolymer is an inert material and offers good flexibility and toughness even at low temperature, low heat sealing temperature, excellent environmental stress crack resistance, etc.

EVA copolymers are particularly useful in the packaging industry. EVA copolymers may be injection moulded, blow moulded or extruded. EVA copolymers with vinyl acetate content up to 18 percent may also be converted into films by conventional processing methods, such as blown, co-extrusion, cast, lamination and coating.

EVA copolymers may be combined with other polymers in co-extrusion, for example, LDPE/ EVA, HDPE/EVA, BOPP/EVA, Polyester/EVA, etc, giving improved performance in flexible packaging in the form of multilayer films which are widely used for food packaging.

EVA copolymers may also be blended with other polymers like LDPE, HDPE, PP, PVC, etc, to improve properties such as impact, flexibility and easy heat sealability.

EVA copolymers are considered as safe for use as articles or components of articles intended for safe use in contact with foodstuffs, in accordance with the code of Federal Regulation, FDA (USA) (28 CFR 177. 1350).

This standard is intended to be used with the series of Indian Standards on plastics for food contact applications which is given in Annex A.

It is emphasized that these standards need to be used in combination to provide a system of control to the manufacturers of plastics as well as the fabricators of thermoplastic packaging materials to derive maximum benefits. Besides, it may also serve as basis for official agencies to frame suitable legislation to ensure effective safeguards for the safety and health of consumers where thermoplastics for food contact applications are concerned.

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, 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

## ETHYLENE VINYL ACETATE ( EVA ) COPOLYMERS FOR ITS SAFE USE IN CONTACT WITH FOODSTUFFS, PHARMACEUTICALS AND DRINKING WATER — SPECIFICATION

## **1 SCOPE**

1.1 This standard specifies the requirements and methods of sampling and test of ethylene vinyl acetate (EVA) copolymer for the manufacture of plastic items used in contact with foodstuffs, pharmaceuticals and drinking water.

**1.2** This standard does not purport to establish the suitability of the packing media with particular foodstuffs, pharmaceuticals and drinking water, from other than toxicological considerations.

## **2 NORMATIVE REFERENCES**

The following standards 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 editions of the standards indicated below:

IS No. Title

4905 : 1968 Methods for random sampling

- 9833 : 1981 List of pigments and colourants for use in plastics in contact with foodstuffs, pharmaceuticals and drinking water
- 9845: 1986 Methods of analysis for the determination of specific and/ or overall migration of constituents of plastics materials and articles intended to come into contact with foodstuffs, pharmaceuticals and drinking water
- 13449: 1992 Positive list of constituents of ethylene vinyl acetate (EVA) copolymers for safe use in contact with foodstuffs, pharmaceuticals and drinking water

## **3 TERMINOLOGY**

For the purpose of this standard, the definitions given in IS 13449 : 1992 shall apply.

## **4 REQUIREMENTS**

## 4.1 Basic Resins

A basic resin produced by the co-polymerization of ethylene and vinyl acetate.

### 4.1.1 Vinyl Acetate (VA) Content

In the EVA copolymers, the VA content shall be between 3 percent and 50 percent when tested in accordance with the methods prescribed in Annex B.

## 4.1.2 Additive Concentrates

The total level of slip agent and/or anti-block agent added to the copolymer shall not exceed 2.5 percent by mass.

## 4.2 Material

The material shall also comply with the threshold limits of the manufacturing residues, polymerization ingredients, auxiliary items as prescribed in IS 13449 : 1992.

### 4.3 Pigments and Colourants

In case coloured material is used for food packaging applications, it shall comply with the list and limits of the pigments and colourants prescribed in IS 9833 : 1981.

## 4.4 Overall Migration

The material shall also comply with the overall migration limits of 60 mg/1, Max of the simulant and 10 mg/dm<sup>2</sup>, Max of the surface of the material or article, when tested by the method prescribed in IS 9845 : 1986.

#### 4.5 Storage and Control

### 4.5.1 Storage

Plastics materials intended for food contact use shall be stored separately from other materials in closed, properly identified containers.

#### 4.5.2 Control

An authorized person shall supervise and control the issue of plastics materials to the

process or manufacturing area and shall maintain appropriate written records of the issue of such materials.

**4.5.3** Adequate standards of hygiene shall be maintained at all times and plant operators and storemen shall be trained in proper hygiene practices.

## **5 PACKING AND MARKING**

## 5.1 Packing

The material shall be packed in paper/plastics bags with suitable liner, as agreed between the purchaser and the supplier, in a manner so as to ensure that the items do not become contaminated during storage.

## 5.2 Marking

**5.2.1** Each package shall be clearly marked with the following information:

- a) Indication of the source of manufacture, and trade-mark, if any;
- b) Name and type of material;
- c) Month and year of manufacture;
- d) Net mass of the material; and
- e) Lot and batch number.

**5.2.2** The package shall also carry the following symbol clearly embossed/printed on it ( in

accordance with the EEC Directive 80/590/EEC 'Symbol' that shall accompany materials and articles intended to come into contact with foodstuff):



5.2.3 The package may also be marked with the Standard Mark.

## **6** SAMPLING

The method of drawing representative sample of the material and the criteria for conformity shall be as prescribed in Annex C.

## ANNEX A

## (Foreword)

## LIST OF INDIAN STANDARDS ON PLASTICS FOR FOOD CONTACT APPLICATIONS

| IS No.             | Title  | IS No.               | Title   |
|--------------------|--|----------------------|---|
| <b>9833</b> : 1981 | List of pigments and colourants<br>for use in plastics in contact<br>with foodstuffs, pharmaceuti-<br>cals and drinking water  | 10142 : 1 <b>982</b> | Specification for styrene poly-<br>mers for its safe use in contact<br>with foodstuffs, pharmaceuti-<br>cals and drinking water                         |
| 9845:1986          | Method of analysis for the<br>determination of specific and/or<br>overall migrations of consti-<br>tuents of plastic materials and<br>articles intended to come into<br>contact with foodstuffs ( <i>first</i> ) | 10146 : 1982         | Specification for polyethylene<br>for its safe use in contact with<br>foodstuffs, pharmaceuticals and<br>drinking water                                 |
| 10141 : 1982       | revision)<br>Positive list of constituents of<br>polyethylene in contact with<br>foodstuffs, pharmaceuticals and<br>drinking water   | 10148 : 1982         | Positive list of constituents of<br>polyvinyl chloride (PVC) and<br>its copolymers in contact with<br>foodstuffs, pharmaceuticals<br>and drinking water |

| IS No.       | Title   | IS No.       |                            |
|--------------|---|--------------|----------------------------|
| 10149 : 1982 | Positive list of constituents of<br>styrene polymers in contact<br>with foodstuffs, pharmaceuti-<br>cals and drinking water                                   | 12247 : 1988 | in<br>pł<br>wa<br>Sr       |
| 10151 : 1982 | Specification for polyvinyl chlo-<br>ride (PVC) and its copolymers<br>for its safe use in contact with<br>foodstuffs, pharmaceuticals and<br>drinking water   | 12248 : 1988 | m<br>w:<br>ca<br>Pc        |
| 10171 : 1986 | Guide on suitability of plastics<br>for food packaging ( <i>first</i><br><i>revision</i> )  |              | us<br>pl<br>w              |
| 10909 : 1984 | Positive list of constituents of<br>polypropylene and its copoly-<br>mers in contact with foodstuffs,<br>pharmaceuticals and drinking<br>water                | 12252 : 1987 | Si<br>te<br>fo<br>wi<br>ca |
| 10910 : 1984 | Specification for polypropylene<br>and its copolymers for its safe<br>use in contact with foodstuffs,<br>pharmaceuticals and drinking<br>water                | 12229 : 1987 | Po<br>pc<br>(H<br>us<br>ph |
| 11434 : 1985 | Specification for ionomer resins<br>for its safe use in contact with<br>foodstuffs, pharmaceuticals<br>and drinking water                                     | 13449 : 1992 | Pc<br>et<br>co             |
| 10435 : 1985 | Positive list of constituents/<br>ionomer resin for its safe use<br>in contact with foodstuffs,<br>pharmaceuticals and drinking<br>water                      | 13576 : 1992 | dr<br>Et<br>(I<br>te:      |
| 11704 : 1986 | Specification for ethylene/<br>acrylic acid (EAA) copoly-<br>mers for their safe use in con-<br>tact with foodstuffs, pharma-<br>ceuticals and drinking water | 13577 : 1992 | ph<br>wa<br>Pc<br>et       |
| 11705 : 1986 | Positive list of constituents of<br>ethylene/acrylic acid (EAA)<br>copolymers for their safe use  |              | ter<br>fo<br>dr            |

Title

in contact with foodstuffs, pharmaceuticals and drinking water

Specification for Nylon 6 polymer for its safe use in contact with foodstuffs, pharmaceuticals and drinking water

Positive list of constituents of Nylon 6 polymer for its safe use in contact with foodstuffs, pharmaceuticals and drinking water

7 Specification for polyalkylene terephthalates (PET and PBT.) for their safe use in contact with foodstuffs, pharmaceuticals and drinking water

7 Positive list of constituents of polyalkylene terephthalates (PET and PBT) for their safe use in contact with foodstuffs, pharmaceuticals and drinking water

1992 Positive list of constituents of ethylene vinyl acetate (EVA) copolymers in contact with foodstuffs, pharmaceuticals and drinking water

> Ethylene methacrylic acid (EMAA) copolymers and terpolymers for their safe use in contact with foodstuffs, pharmaceutical and drinking water — Specification

2 Positive list of constituents of ethylene methacrylic acid (EMAA) copolymers and terpolymers in contact with foodstuffs, pharmaceuticals and drinking water — Specification

## ANNEX B

(Clause 4.1.1)

## DETERMINATION OF VINYL ACETATE CONTENT IN ETHYLENE VINYL ACETATE ( EVA ) COPOLYMERS

## **B-1 METHOD-I BY PYROLYSIS**

## **B-1.1** Outline of the Method

The polymer is pyrolysed at 400°C. The pyrolysate is bubbled through sodium hydroxide solution where acetic acid formed during pyrolysis is absorbed. Excess sodium hydroxide is then back titrated with standard hydrochloric acid (0.1 N) solution using phenolphthalein as indicator. A nitrogen purge is used to force the vapourized acetic acid into the absorbing solution.

## **B-2 APPARATUS**

**B-2.1** Any suitable combustion furnaces capable of being maintained at a temperature of 400°C.

**B-2.2** Combustion tube with B 29 ground glass joints — Outside diameter 32 mm, length 36 mm.

**B-2.3** Prefabricated ground glass joint to fit the pyrolysis tube (gas disperser).

**B-2.4** Quartz boat to fit inside the combustion tube.

B-2.5 Tall form bubbler, 500 ml capacity.

B-2.6 Pipette, 25-ml.

B-2.7 Burette, 50-ml, with stand and holder.

B-2.8 Regulated nitrogen supply.

## **B-3 REAGENTS**

**B-3.1** Standard hydrochloric acid (HCL) solution, 0.1 N.

**B-3.2** Sodium hydroxide (NaOH) solution, 0.1 N (approx).

**B-3.3** Phenolphthalein indicator solution, 0.1 percent, prepared by dissolving 0.1 g of phenolphthalein in 60 ml neutralized rectified spirit and diluting to 100 ml with distilled water.

B-3.4 Distilled water.

## **B-4 PROCEDURE**

**B-4.1** Depending upon vinyl acetate content, weigh accurately 0.4-0.6 g of EVA sample into a quartz boat.

**B-4.2** Measure 25-ml 0.1 N sodium hydroxide solution into a 500-ml tall form bubbler. Back titrate with standard 0.1 N hydrochloric acid to determine equivalent number of millilitres required to neutralize 25 ml of 0.1 N sodium hydroxide.

**B-4.3** Connect the gas disperser and 500 ml tall form bubbler to the combustion tube, making sure that the gas disperser is well below the level of the absorbing sodium hydroxide solution.

**B-4.4** Adjust the flow of nitrogen to 350-400 ml/min.

**B-4.5** Insert the sample into the combustion tube and quickly connect the nitrogen purge. Heat the sample at  $400^{\circ}$ C for 1 h.

**B-4.6** Sample should be left in the combustion furnace for at least 20-25 minutes.

**B-4.7** After the prescribed time, stop heating and rinse the disperser with distilled water.

**B-4.8** Add 10 drops of phenolphthalcin indicator to the absorbing solution and back titrate with standard 0.1 N hydrochloric acid. (Change of colour — Pink to colourless).

## **B-5 CALCULATION**

Calculate the percent vinyl acetate (VA) content using formula:

$$\% \text{ V.A.} = \frac{(b-a) \times N \times 8.61}{W}$$

where

- b is the amount of HCl consumed for blank determination (B-4.2),
- A is the amount of HCl consumed for black titration ( B-4.8 ),
- N is the normality of HCl solution, and
- W is the weight of the sample taken (B-4.1).

## **B-6 METHOD-II BY INFRA-RED (IR)** SPECTROPHOTOMETRY

## **B-6.1** General

This method describes infra-red spectrophotometric procedure for the determination of combined and free vinyl acetate, when carbon black is absent. The method of determination is applied directly to a hot pressed plate of accurately known thickness.

Two different procedures are used for different ranges of copolymerized vinyl acetate.

i) For the range 5-20 percent combined vinyl acetate, plate of  $0.38 \pm 0.05$  mm thickness is used and measurements are made of the absorption band at 3460 cm<sup>-1</sup>, due to carbonyl overtone of vinyl acetate.

The determination is not affected by the presence of vinyl acetate monomer.

ii) For the higher range of 20-35 percent combined vinyl acetate, plate of  $3.8 \pm 0.25$  mm thickness is used and measurements are made of the absorption band at 4688 cm<sup>-1</sup>, due to carbonyl combination overtone of vinyl acetate.

Up to 5 percent free vinyl acetate monomer can be tolerated in this procedure.

## **B-7 PRINCIPLE**

The determination of free vinyl acetate is based on the measurement of the band at 6210  $cm^{-1}$  due to C=C stretching mode of - CH = CH<sub>2</sub> group in vinyl acetate monomer. The contribution of the end group structure - CH<sub>2</sub> - C-O - CH=CH<sub>2</sub> amounts to only

0.03 percent calculated as vinyl acetate monomer for copolymers containing 30 percent and 40 percent W/W vinyl acetate and can thus be ignored.

## **B-8 APPARATUS**

**B-8.1 IR Spectrophotometer** — Range 350 cm<sup>-1</sup> -7000 cm<sup>-1</sup>.

B-8.2 Hydraulic Press — Capacity 10 tons.

B-8.3 Press plates and spacer.

**B-8.4** Dial gauge micrometer (1 div = 0.002 mm).

B-8.5 Melinex Film — thickness 100 microns.

NOTE — Melinex is a trade-name of a film made of polyethylene terephthalates (PET), supplied by M/s Mehta Enterprise, Bombay. Other equivalent films may also be used.

#### **B-9 PREPARATION OF TEST SPECIMEN**

#### B-9.1 For Samples Having 5-20 percent Combined Vinyl Acetate

Obtain a specimen of the sample  $0.38 \pm 0.05$  mm thickness by pressing between stainless steel plates, melinex film.

Measure accurately the mean thickness of the sample to the nearest 0.002 mm with a dialgauge micrometer, taking the measurements along the centre line of the length of the moulded sample.

Record the average of ten values as thickness of the sample.

**B-9.1.1** Required dimensions of the specimen are:

| 50  | Х | <b>5</b> 0 1 | mm |
|-----|---|--------------|----|
| 75  | × | <b>75</b> 1  | mm |
| 100 | × | 100 i        | mm |
| 150 | × | 150          | mm |

Out of these four dimensions any one size of spacer can be used.

## B-9.2 For Samples Having 20-35 percent W/W Combined Vinyl Acetate

Prepare a specimen of the sample as per the available spacer as given in **B-9.1.1**,  $3\cdot 8 \pm 0.25$  mm in thickness by pressing between stainless steel plates and melinex film. Measure accurately the mean thickness of the sample in accordance with **B-9.1**.

## **B-10 PROCEDURE**

**B-10.1** Obtain a specimen of the standard sample prepared as per **B-9**.

**B-10.2** Take absorbance of more than two different standard plates of some grade, and the said range of vinyl acetate content having nearly the same thicknesses.

**B-10.3** Prepare a calibration curve using these absorbance values.

**B-10.4** Take out absorbance of sample of nearly same thickness and compare the absorbance of the sample with the absorbance of standard plates.

#### NOTES

 For 5-20 percent combined vinyl acetate content take absorption spectra from 3050 cm<sup>-1</sup> to 3800 cm<sup>-1</sup>.
For 20-35 percent combined vinyl acetate content

take absorption spectra from 4 500 cm<sup>-1</sup> to 6 000 cm<sup>-1</sup>.

## **B-11 CALCULATION**

Calculate the percent vinyl acetate content as follows:

|           | % Concentration    | Thickness of |
|-----------|--------------------|--------------|
| 0/ Vinul  | on the screen or   | × Standard   |
| acetate   | calibration cenrte | plate        |
| acciate - | Thickness of sa    | ample plate  |

# B-12 METHOD OF OPERATION OF TYPICAL HYDRAULIC PRESS

#### **B-12.1** Press Details

Upstroke, single acting, electrically heated rubber moulding hydraulic press having the following specification:

| a) | Capacity         |   | Tons  |
|----|------------------|---|---|
| b) | Types            |   | 4 pillar type                               |
| c) | Platen size      |   | 200 mm × 200 mm                             |
| d) | Daylites         |   | Single daylite                              |
| e) | Daylite gap      |   | 75 mm                                       |
| f) | Ram diameter     |   | 75 mm                                       |
| g) | Ram stroke       |   | 75 mm                                       |
| h) | Working pressure | — | 210 kg                                      |
| j) | Mode of heating  |   | Electrical with tem-<br>perature controller |
| k) | Heaters          |   | 6 No. heaters of capacity 1 kW each         |

- m) Cooling Water cooling in arrangement heating plates
- n) Mode of Hand operated by operation D.C. valve
- p) Max temperature 250°C

## **B-12.2 Operation**

**B-12.2.1** Start the main switch, set the required temperature both top and bottom temperature controller.

**B-12.2.2** As soon as the charging temperature is attained, insert the plate. Weigh the EVA granules as per the grade spacer thickness (see **B-12.2**).

**B-12.2.3** Start the motor. Operate the lever downwards direction. Keep the plate so that there is positive pressure, then switch off motor.

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**B-12.2.4** Wait till the set temperature is reached.

**B-12.2.5** Pressurize with help of motor and lever to 5 tons for the scheduled time (see **B-12.2.8**).

**B-12.2.6** Stop heaters and open the water tap for cooling. As soon as the room temperature is reached, release pressure and remove the plates.

B-12.2.7 Clean the spacer by acetone or xylene.

**B-12.2.8** Operating conditions of hydraulic press are as under:

**B-13.2** Type CONFIG, ENTER and then press F1. Then press Esc. Press Y ENTER.

**B-13.3** Type BOMEN ENTER and then press any key to continue. Original menu will be displayed on the screen.

**B-13.4** Press F1 (reference) and feed appropriate range as per the requirement. Press F1 (start acquisition). Once the reference spectrum appears on the screen, then press Esc. Original menu will be displayed.

B-13.5 Keep the sample in the sample compart-

| Vinyl                          | Set Charge Samp |  | High Pressure |                  |  |   | Cooling      |
|--------------------------------|-----------------|--|---------------|------------------|--|---|--------------|
| Acetate<br>Content,<br>Percent | °C<br>℃         | on Contact<br>Pressure at<br>Temperature | Temp<br>°C    | Pressure<br>Tons | Thickness  | <i>Time</i> ,<br>Min                                | Time,<br>Min |
| 07 — 02                        | 140             | 90                                       | 140           | 5                | $\left. \begin{array}{c} 0.3-1.0\\ 2.0-3.0\\ 4.0-8.0 \end{array} \right\}$ | $\left\{\begin{array}{c}2\\3\\4\end{array}\right\}$ | 15           |
| 12 — 02                        | 130             | 80                                       | 130           | 5                | ,,   | ,,  | 15           |
| 14 — 08                        | 125             | 75                                       | 125           | 5                | ••   | ,,  | 15           |
| 18 - 02                        | 120             | 70                                       | 120           | 5                | ••   | ••  | 15           |
| 18 — 10                        | 120             | <b>7</b> 0                               | 120           | 5                | **   | ••  | 15           |
| 28 — 06                        | 100             | 50                                       | 95            | 5                | **   | ••  | 15           |
| <b>28</b> — 25                 | <b>9</b> 0      | 40                                       | 85            | 5                | ••   | ,,  | 15           |
| 28 150                         | 80              | 40                                       | 75            | 5                | "  | "   | 10           |
| <b>28</b> — 350                | 65              | 40                                       | 65            | 5                | **   | ,,  | 10           |
| 28 — 400                       | 65              | 40                                       | 65            | 5                | ••   | "   | 10           |

B-12.2.9 Switch off the heaters at the time of applying high pressure.

**B-12.2.10** For sample containing 28 percent vinyl acetate, put in ice water for 5 minutes after machine cooling.

B-12.2.11 Weight of the sample to be taken as per the size of the spacer as follows:

| Spacer Thickness,<br>mm | Spacer Size,<br>mm | Material<br>Quantity, g | EVA Grade         |
|-------------------------|--------------------|-------------------------|-------------------|
| 0.38                    | $150 \times 150$   | 9                       | 07-02, 12-02,     |
|                         |                    |                         | 14-08, 18-02,     |
|                         |                    |                         | 18-10             |
| 0.38                    | $150 \times 150$   | 75                      | 26 - 06, 28 - 25, |
|                         |                    |                         | 28-25, 28-150,    |
|                         |                    |                         | 28-350, 28-400    |

## **B-13 OPERATION OF TYPICAL FTIR** ( **BOMEN MODEL** — MB 100 )

**B-13.1** Switch on computer and monitor. On the screen C:/> will be displayed.

ment and feed sample details. Then press F3 (absorbance). Press F1 (start acquisition). After few minutes sample spectrum will be displayed on the screen. Then press Esc, till the original menu appears.

**B-13.6** Press F5 (display) to confirm whether it is saved or not. Then press Esc to get original menu.

**B-13.7** For quantitative estimation of vinyl acetate content in plate follow steps mentioned below.

B-13.7.1 Press F6 — Batch analysis

Press F1 — Quantitative analysis Press F2 — Run method **B-13.7.2** Type method name and then press ENTER. If the results are to be printed, then press F1 (*see* that printer is connected). Press F3 ( select files and RUN ).

**B-13.7.3** Mark files to analyse by pressing space bar. Move the cursor from experiment to sub files by pressing arrow keys and then press space bar and press ENTER.

**B-13.7.4** Concentration will be displayed on the screen.

## ANNEX C

## (*Clause* 6.1)

## SAMPLING OF ETHYLENE VINYL ACETATE (EVA) COPOLYMERS

## C-1 GENERAL

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

C-1.2 Sample shall not be taken in an exposed place.

**C-1.3** The sampling instrument, whenever applicable, shall be made of stainless steel or any other suitable material on which the material shall have no action. The instrument shall be clean and dry.

C-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for \_ samples from adventitious contamination.

C-1.5 The samples shall be placed in a suitable, clean, dry, air-tight metal or glass containers on which the material has no action. The sample containers shall be of such a size that they are almost completely filled by the sample.

**C-1.6** Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, such as the date of sampling, the month and year of manufacture of the material, etc.

**C-1.7** Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

## C-2 LOT

C-2.1 In a single consignment all the packages of the same class, same type, same form and belonging to the same batch of manufacture shall be grouped together to constitute a lot. If a consignment is known to consist of packages belonging to the different batches of manufacture of different forms, the packages belonging to the same batch of manufacture and same form shall be grouped together and each such group shall constitute a lot.

C-2.2 For ascertaining the conformity of the material to the requirements of this specification, samples shall be tested from each lot separately. The number of packages to be sampled shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 1.

Table 1 Scale of Sampling

| No. of Packages<br>in the Lot | Sample Size |  |  |
|-------------------------------|-------------|--|--|
| (1)                           | (2)         |  |  |
| Up to 50                      | 3           |  |  |
| 51 to 150                     | 4           |  |  |
| 151 to 300                    | 5           |  |  |
| 301 to 500                    | 7           |  |  |
| 501 and above                 | 10          |  |  |

**C-2.2.1** These packages shall be selected at random from the lot and in order to ensure the randomness of selection, procedures given in IS 4905 : 1968 may be followed.

## **C-3 PREPARATION OF TEST SAMPLES**

C-3.1 From each of the packages of material selected, small portions of material shall be drawn with the help of a suitable sampling instrument. The total quantity of material collected from each package shall be sufficient to test all the requirements given in 4.

C-3.2 In the case of packages consisting of containers vials, rolls or films the number of items to be selected from a package for testing each of the requirements given in 4 shall be one.

## **C-4 NUMBER OF TESTS**

The tests for determining all the requirements given in 4 shall be carried out on the individual test samples.

## **C-5 CRITERIA FOR CONFORMITY**

From the individual test results calculate the average (X) and the range (R)

where

 $X = \frac{\text{Sum of the test results}}{\text{Number of tests}}$ 

R = difference between the maximum and the minimum values of the test results The lot shall be declared as conforming to the requirements of various characteristics if:

 $X + KR \leq$  the maximum value specified; and where the value of K shall be chosen from table given below:

Value of K for Various Sizes and AQL

| Sample<br>Size |               | AÇ            | 2L    |               |                |
|----------------|---------------|---------------|-------|---------------|----------------|
|                | 0.65          | 1.00          | 1.50  | 2.50          | <b>4</b> ·00   |
| 3              |               |               |       | 0.587         | 0.502          |
| 4              |               | 0.651         | 0.598 | 0.525         | 0.450          |
| 5              | 0 <b>·663</b> | 0.614         | 0.565 | 0· <b>496</b> | 0 <b>·43</b> 1 |
| 7              | 0.613         | 0 <b>·696</b> | 0.525 | 0.465         | 0.405          |
| 10             | 0.755         | 0·70 <b>3</b> | 0.650 | 0·579         | 0.507          |

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