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सुघट्यकारी ईस्टर — नमूने लेने तथा परीक्षण की पद्धतियाँ
(पहला पुनरीक्षण)

Indian Standard

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PLASTICIZER ESTERS — METHODS OF
SAMPLING AND TESTS

(*First Revision*)

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (First Revision) 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.

This standard was first published in 1980 in order to avoid any ambiguity and also to unify the various methods of tests on plasticizers which though common were, however, covered in the individual specifications. Subsequently with the publication of this standard the methods of test covered in the various individual product standards on plasticizers were not included when their revisions were published. The present revision has been taken up as a result of the review of this standard by Plastics Sectional Committee, PCD 12 to align the various methods of test on plasticizers with the corresponding overseas standards. In this revision, additional test methods for determining volatile loss of plasticizers, determination of colour value by Lovibond Scale (Sucrose Test) and determination of matter oxidizable by potassium dichromate have been included.

Looking at the market needs the Committee felt to include Gas chromatographic analysis to check the purity of the plasticizers which are becoming more and more popular. However, the Committee observed that the standardization of the method is yet to take some more time and it was thought appropriate to include the same at a later date as and when they are standardized.

Considerable assistances have been derived from the following publications while preparing this standard:

ISO 2211 : 1973(E) Liquid chemical products — Measurement of colour in Hazen units (Platinum — Cobalt scale), issued by the International Organization for Standardization (ISO).

ISO 2524 : 1974(E) Adipate esters for industrial use — Measurement of colour after heat treatment, issued by the International Organization for Standardization (ISO).

ISO 2525 : 1974(E) Adipate esters for industrial use — Determination of acidity to phenolphthalein — Volumetric method, issued by the International Organization for Standardization (ISO).

ISO 2526 : 1974 (E) Adipate esters for industrial use — Determination of ash — Gravimetric method, issued by the International Organization for Standardization (ISO).

ISO 2527 : 1974 (E) Adipate esters for industrial use — Determination of ester content — Volumetric method, issued by the International Organization for Standardization (ISO).

BS 4835 : 1973 Methods of test for plasticizer esters, issued by the British Standards Institution (BSI).

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PLASTICIZER ESTERS — METHODS OF SAMPLING AND TESTS

(*First Revision*)

1 SCOPE

This standard prescribes methods of sampling and tests for plasticizer esters which are organic esters of phthalic acid and alcohols and are liquids at ambient temperature.

2 NORMATIVE REFERENCES

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

<i>IS No.</i>	<i>Title</i>
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
323 : 1959	Rectified spirit (<i>revised</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
2362 : 1973	Determination of water by the Karl Fischer method (<i>first revision</i>)
2828 : 1964	Glossary of terms used in the plastics industry
4825 : 1982	Specification for liquid-in-glass solidstem reference thermometers (<i>first revision</i>)
4905 : 1968	Methods for random sampling

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 2828 : 1964 and the following shall apply.

3.1 Hazen Colour Unit

The colour of a solution containing 1 mg of platinum per litre in the form of chloroplatinic acid (H_2PtCl_6), in the presence of 2 mg of cobalt (II) chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) per litre.

4 SAMPLING**4.1 General Requirements of Sampling**

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

4.1.1 The sampling instrument shall be clean and dry.

4.1.2 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

4.1.3 The samples shall be placed in suitable, clean, dry and airtight glass containers.

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

4.1.5 Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

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

4.2 Sampling Instrument

It shall be made of thick glass or metal on which the material has no action and shall be 20 to 40 mm in diameter and 400 to 800 mm in length (Fig. 1). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing samples, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

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

4.3 Scale of Sampling**4.3.1 Lot**

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate

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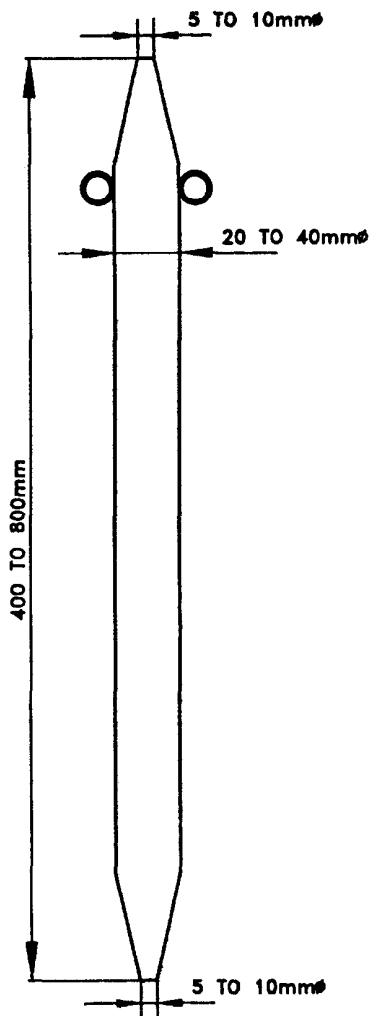


FIG. 1 SAMPLING TUBE

lots. In the case of a consignment drawn from a continuous process, 1 000 containers shall constitute a lot.

4.3.2 The number of containers to be drawn from a lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 1.

Table 1 Number of Containers to be Selected for Sampling

Lot Size	No. of Containers to be Selected
<i>N</i>	<i>n</i>
(1)	(2)
3 to 50	3
51 " 100	4
101 " 300	5
301 " 500	6
501 " 1 000	7
1 001 and above	8

NOTE — When the lot size is less than 3, number of containers to be selected shall be as agreed to between the purchaser and the supplier.

4.3.2.1 These containers shall be selected at random from the lot. To ensure the randomness of selection reference may be made to IS 4905 : 1968. Alternately the following procedure shall be adopted:

Starting from any container in the lot count them 1, 2, 3 up to *r* and so on in one order. Every *r*th container thus counted shall be withdrawn to give a sample for test where *r* is the integral part of N/n .

4.4 Test Sample and Referee Sample

4.4.1 Preparation of Test Sample

4.4.1.1 From each of the containers selected according to 4.3.2.1, a representative portion of the material, not less than 200 g, shall be drawn with the help of a sampling instrument.

4.4.1.2 Out of these portions, an equal quantity of the material shall be taken and mixed thoroughly to form a composite sample of about 400 g. 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.

4.4.1.3 The remaining portions of the material from each container shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried containers which shall be suitably secured against contamination and labelled with all the particulars of sampling given under 4.1.5. The material in each such container shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be for the purchaser, another for the supplier and the third to be used as a referee sample.

4.4.2 Referee Sample

The referee sample shall consist of the composite sample (4.4.1.2) and a set of individual samples constituting the set of test samples (4.4.1.3). These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

4.5 Number of Tests

4.5.1 Tests for the determination of ester value shall be conducted on each of the individual samples constituting the set of test samples.

4.5.2 Tests for the remaining characteristics shall be conducted on the composite sample.

4.6 Criteria for Conformity

4.6.1 For Individual Samples

The mean and the range for the test results for the determination of ester value shall be calculated as follows:

Mean (X) = The sum of test results divided by the number of test results, and

Range (R) = The difference between the maximum and minimum values of the test results.

4.6.1.1 If the value of the expression ($X - 0.6 R$) and ($X + 0.6 R$) as calculated from the test results lies between the value stipulated, the lot shall be declared to have satisfied the requirement of ester value.

4.6.2 For Composite Sample

The test results on the composite sample shall meet the corresponding requirements.

4.6.3 A lot shall be declared as conforming to the specification, if it satisfies the requirements for each of the characteristics prescribed.

4.6.4 If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of the specification.

5 QUALITY OF REAGENTS

Unless specified otherwise pure chemicals and distilled water (see IS 1070 : 1992) shall be used in all tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

6 DETERMINATION OF COLOUR

6.1 General

This method is applicable only to clear, slightly coloured liquids for which the colour characteristics are close to those of the reference platinum-cobalt scale. Such colour characteristics are generally describable as 'brownish-yellow'.

6.2 Principle

Visual comparison of the colour of a sample with that of colour standards, and expression of the result in terms of Hazen (platinum-cobalt) colour units.

For routine control purposes an instrument such as a comparator, colorimeter or spectrophotometer may be used, provided that it has first been established that the results so obtained are identical with those obtained by visual comparison.

6.3 Reagents

Distilled water, or water of equivalent purity, shall be used in the test (see IS 1070 : 1992).

6.3.1 Cobalt (II) Chloride Hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$)

6.3.2 Hydrochloric Acid

Approximately 1.19 g/ml, about 38 percent (m/m) solution, or approximately 12 N solution (see IS 265 : 1993).

6.3.3 Chloroplatinic Acid or Potassium Chloroplatinate (K_2PtCl_6)

Dissolve 1.00 g of platinum in a sufficient quantity of *aqua regia* in a glass or porcelain dish by heating on a boiling-water bath. When the metal has dissolved, evaporate the solution to dryness. Add 4 ml of the hydrochloric acid solution (6.3.2) and again evaporate to dryness. Repeat this operation twice more. In this way 2.10 g of chloroplatinic acid (H_2PtCl_6) are obtained.

6.4 Apparatus

Two colorimetric tubes, flat based if possible, with a graduation mark at least 100 mm above the base and matched especially with respect to colour of glass and height of graduation mark above the base. Suitable tubes are available commercially as 50 ml or 100 ml Nessler cylinders.

For the measurement of low colorations (less than 50 Hazen units), the height of the graduation mark above the base must be greater than for the measurement of deeper colours and must be sufficient that, on looking through this greater depth of liquid, a clear distinction between the standard Hazen matching solutions can be observed.

6.5 Preparation of Standard Colorimetric Solutions

6.5.1 Standard Colorimetric Solution, 500 Hazen Units

Dissolve 2.00 g of cobalt chloride (6.3.1) and the equivalent of 1.00 g of platinum, that is, either:

- 2.10 g of the chloroplatinic acid, or 2.49 g of the potassium chloroplatinate (6.3.3),

in water in a 2 000 ml one-mark volumetric flask, add 200 ml of the hydrochloric acid solution (6.3.2), dilute to the mark and mix.

This solution has a colour of 500 Hazen colour units.

6.5.2 Standard Hazen Matching Solutions (Diluted Solutions)

Into two series of ten 500-ml and fourteen 250-ml one-mark volumetric flasks, place the volumes of standard colorimetric solution (6.5.1) shown in Table 2, dilute to the mark and mix.

Table 2 Volumes of Standard Colorimetric Solution

500-ml Volumetric Flasks		250-ml Volumetric Flasks	
Volumes of Standard Colorimetric Solution (6.5.1)	Corresponding Colour	Volumes of Standard Colorimetric Solution (6.5.1)	Corresponding Colour
(1)	(2)	(3)	(4)
ml	Hazen units	ml	Hazen units
0	0	30	60
5	5	35	70
10	10	40	80
15	15	45	90
20	20	50	100
25	25	62.5	125
30	30	75	150
35	35	87.5	175
40	40	100	200
50	50	125	250
		150	300
		175	350
		200	400
		225	450

6.5.3 Storage

Store these solutions (6.5.1 and 6.5.2) in the dark in stoppered glass bottles. Under these conditions the colour standard solution (6.5.1) is stable for 1 year. The standard Hazen matching solution (6.5.2), although stable for at least 1 month, shall preferably be prepared fresh.

6.6 Procedure

First check visually that the sample has colour characteristics close to those of the standard Hazen matching solutions (6.5.2). If not, follow the instructions given in the second paragraph of 6.7.

Pour into one of the colorimetric tubes (6.4.1) a quantity of the sample sufficient to fill it to the graduation mark. Similarly pour the standard Hazen matching solution (6.5.2) which appears to have a similar colour into the other tube, to the mark.

Compare the colour of the sample with that of the standard, by looking down the tubes from top to bottom against a white background strongly illuminated by daylight or by an electric 'daylight' lamp, taking care to avoid any side illumination.

Repeat, if necessary, with other standard Hazen matching solutions until the closest match is obtained.

NOTES

1 Instruments are available which permit visual comparison of a liquid of a given depth, with a moving tinted glass disc

corresponding to the different standard Hazen matching solutions. The use of such instruments, whose standards are very stable, is permissible provided that it has been established that using these tinted glass discs gives the same results as do the corresponding standard Hazen matching solutions.

2 For routine control purpose, a colorimeter or spectrophotometer may be used, the instrument being standardized by means of the standard colorimetric solutions (6.5.1 and 6.5.2), provided that it has been confirmed that the use of that instrument gives the same results as does visual comparison.

3 In case of solid sample, make the measurements at a temperature 10°C above its melting point.

6.7 Expression of Results

Express the colour of the sample as the number of Hazen colour units corresponding to the standard Hazen matching solution having the closest match to the sample.

If the colour of the sample does not correspond with that of any of the standard hazen matching solutions (brownish-yellow) give if possible, an estimate of the colour and a description of the observed colour.

7 DETERMINATION OF RELATIVE DENSITY

7.0 Outline of the Method

The relative density of the material is determined with a specific gravity bottle or a pycnometer.

7.1 Apparatus

7.1.1 Specific Gravity Bottle or Pycnometer, with well-fitting ground-glass joints. To calibrate, clean and dry the relative density bottle or pycnometer thoroughly, weigh and then fill with freshly boiled and cooled water at $27 \pm 2^\circ\text{C}$ after removing the cap of the side arm. Fill to over-flowing by holding the bottle or pycnometer on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper and immerse pycnometer/bottle in a water-bath maintained at $27.0 \pm 0.2^\circ\text{C}$. Keep the entire bulb of pycnometer/bottle completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove pycnometer/bottle from the bath, wipe completely dry, replace the cap. Bring back to $27.0 \pm 0.2^\circ\text{C}$ and weigh. Calculate the mass of water. This is a constant for the bottle or pycnometer, but should be checked periodically.

7.1.2 Water Bath, maintained at the desired test temperature.

7.1.3 Thermometer, any convenient, thermometer of a suitable range with 0.1 or 0.2°C sub-divisions. The thermometer should be checked against a standard thermometer, which has been calibrated and certified by the National Physical Laboratory,

New Delhi, or any other laboratory recognized for such work.

7.2 Procedure

Fill the relative density bottle or pycnometer with the material previously maintained at $27.0 \pm 0.2^\circ\text{C}$. Hold the bottle or pycnometer on its side in such a manner as to prevent the entrapment of air bubbles after removing the cap of the side arm. Insert the stopper and immerse in the water-bath maintained at $27.0 \pm 0.2^\circ\text{C}$ for a period of at least 30 minutes. Carefully wipe off any material that has come through the capillary opening. Remove the bottle or pycnometer from the bath, clean and dry it thoroughly. Replace the cap of the side arm, bring back to $27.0 \pm 0.2^\circ\text{C}$ and weigh.

7.3 Calculation

$$\text{Relative density at } 27/27^\circ\text{C} = \frac{M_1 - M}{M_2 - M}$$

where

M_1 = mass in g of the specific gravity bottle or Pycnometer with material at the test temperature,

M = mass in g of the specific gravity bottle or Pycnometer, and

M_2 = mass in g of the specific gravity bottle or Pycnometer with water at the test temperature.

8 DETERMINATION OF REFRACTIVE INDEX

8.0 Outline of the Method

The refractive index is determined by Abbe Refractometer at $27.0 \pm 0.1^\circ\text{C}$ using diffused daylight or any convenient light as illuminant.

8.1 Apparatus

Refractometer, An Abbe Refractometer with scale graduated directly in terms of refractive index of the D-line of sodium at a temperature $27.0 \pm 0.1^\circ\text{C}$ is used. Water supply is used for maintaining this temperature.

8.2 Procedure

8.2.1 Place the refractometer in front of a suitable source of a light (either daylight or electric light). Adjust the temperature of refractometer to $27.0 \pm 0.2^\circ\text{C}$ by circulation of thermostated water. Clean the prisms with alcohol and wipe them dry. Spread a drop of liquid to be tested on the lower prism and clamp it. Adjust the mirror so that the light enters the telescope. Focus the eye-piece on the cross hair and the reading lens of the scale by moving the prism-arm and find a position where the lower part of the fields is dark and the upper part is

bright. In general, the border line, which is coloured, shall be corrected by turning the milled head on the right of the telescope until a sharp line of contrast between black and white areas is obtained.

8.2.2 Move the prism-arm until this black edge just crosses the inter-section of the cross hair. Read the refractive index on the scale to the fourth decimal place. The accuracy of the instrument should be checked by a small test piece of known refractive index which is supplied with the refractometer. This test piece is attached to the upper prism with a liquid of high refractive index (usually monobromonaphthalene).

9 DETERMINATION OF WATER CONTENT

9.0 Moisture is determined by the Karl Fischer method.

9.1 Take about 20 g of the material, weigh accurately and determine the water content by the procedure given in IS 2362 : 1973.

10 DETERMINATION OF ASH

10.1 Principle

Ignition of a test portion at $600 \pm 30^\circ\text{C}$ and weighing of the residue.

10.2 Apparatus

Furnace, capable of being controlled at $600 \pm 30^\circ\text{C}$.

10.3 Procedure

10.3.1 Test Portion

Weigh to the nearest 1 g about 50 g of the laboratory sample.

10.3.2 Determination

Slowly burn the test portion, in several portions, in a tared platinum or silica basin previously heated to $600 \pm 30^\circ\text{C}$, cooled in a desiccator and weighed to the nearest 0.1 mg. Ignite in the furnace (10.2.1), controlled at $600 \pm 30^\circ\text{C}$, until all carbonaceous matter has disappeared. Cool in a desiccator and weigh to the nearest 0.1 mg. Repeat this series of operations of ignition, cooling and weighing until the mass recorded is constant.

10.4 Expression of Results

Ash is given, as percentage by mass, by the formula:

$$\frac{100 m_1}{m_0}$$

where

m_1 = mass, in g, of the residue; and

m_0 = mass, in g, of the test portion.

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11 DETERMINATION OF ACIDITY

11.1 Principle

Titration of the acidity using a standard volumetric solution of sodium hydroxide in the presence of phenolphthalein.

11.2 Reagents

Distilled water, or water of equivalent purity, shall be used in the test (see IS 1070 : 1992).

11.2.1 Sodium Hydroxide

0.1 N standard volumetric solution.

11.2.2 Ethanol

95 percent (v/v) (see IS 323 : 1959).

11.2.3 Phenolphthalein

10 g/l ethanolic solution. Dissolve 1.0 g of phenolphthalein in 100 ml of the ethanol (11.2.2) and make faintly pink by the addition of dilute sodium hydroxide solution.

11.3 Apparatus

11.3.1 Conical Flask, of borosilicate glass, capacity 250 ml, fitted with a stopper carrying a soda-lime tube.

11.3.2 Burette, graduated in 0.02 ml or smaller divisions.

11.4 Procedure

11.4.1 Test Portion

Weigh, to the nearest 0.5 g, about 50 g of the laboratory sample.

11.4.2 Determination

Place 50 ml of the ethanol (11.2.2) in the conical flask (11.3.1), add 0.5 ml of the phenolphthalein solution (11.2.3) and make faintly pink by the addition of the sodium hydroxide solution (11.2.1). Add the test portion (11.4.1) and titrate the mixture with the sodium hydroxide solution (11.2.1) until the pink colour remains for 5 s.

11.5 Expression of Results

Acidity, expressed as a percentage by mass of phthalic acid, is given by the formula:

$$A = \frac{0.83 V}{m}$$

or expressed as milli-equivalents per kilogram, by the formula:

$$B = \frac{100 V}{m}$$

where

V = volume, in millilitres, of the sodium hydroxide solution (11.2.1) used for the titration; and

m = mass in g, of the test portion.

12 DETERMINATION OF ESTER VALUE

12.1 Principle

Saponification of the ester, using potassium hydroxide in ethanolic solution, followed by titration with standard volumetric hydrochloric acid solution in the presence of phenolphthalein as indicator.

12.2 Reagents

Distilled water, or water of equivalent purity, freshly boiled and cooled, shall be used in the test (see IS 1070 : 1992).

12.2.1 Potassium Hydroxide, approximately 1 N solution in 95 percent (v/v) ethanol. (see IS 323 : 1959).

12.2.2 Hydrochloric Acid, 1 N standard volumetric solution.

12.2.3 Phenolphthalein, 10 g/l ethanolic solution. Dissolve 1.0 g of phenolphthalein in 100 ml of 95 percent (v/v) ethanol and make faintly pink by the addition of dilute sodium hydroxide solution.

12.3 Apparatus

12.3.1 Two conical flasks, capacity 250 ml, fitted with ground glass stoppers.

12.3.2 Water-cooled reflux condensers, with ground glass joints to fit the flasks (12.3.1).

12.3.3 Weighing pipette, capable of delivering up to 10 g of sample.

12.4 Procedure

12.4.1 Test Portion

Weigh, to the nearest 0.01 g, the mass of the laboratory sample indicated in Table 3.

Table 3 Mass of the Sample

Ester	Relative Molar Mass, <i>M</i> (Hitherto Known as Molecular Weight)	Mass of the Sample, g
Dimethyl phthalates	194	2.4±0.2
Diethyl phthalates	222	2.8±0.2
Dibutyl phthalates	278	3.5±0.2
Dimethoxyethyl phthalates	280	3.5±0.2
Benzyl butyl phthalates	312	3.9±0.2
Diheptyl phthalates	362	4.5±0.2
Dibutoxyethyl phthalates	366	4.6±0.2
Diocetyl phthalates	390	4.9±0.2
Dinonyl phthalates	418	5.2±0.2
Didecyl phthalates	447	5.6±0.2
Ditridecyl phthalates	531	6.6±0.2

In the case of mixed alcohol phthalate esters, for which the relative molar mass can only, at best, be estimated, the mass of the test portion shall be calculated from the formula:

$$\frac{0.025 M}{n} \pm 0.2 g$$

where

- M = relative molar mass of the phthalate esters, and
- n = basicity of the acid (2 in the case of phthalate esters).

12.4.2 Blank Test

At the same time as the determination, carry out a blank test using the same reagents and following exactly the same procedures as described in 12.4.3, but omitting the test portion.

12.4.3 Determination

12.4.3.1 Introduce 50.0 ml of the potassium hydroxide solution (12.2.1) into one of the two 250 ml conical flasks (12.3.1), and add immediately 5 ml of water. By means of the weighing pipette (12.3.3) transfer the test portion immediately to the flask.

12.4.3.2 Attach the flask (12.3.1) to the condenser (12.3.2) and heat for 1 h in a boiling-water bath. Withdraw the flask, still carrying its condenser, and immerse it in cold running water. When cold, wash down the inside of the condenser with two 20 ml portions of water. Disconnect the flask and wash the joint with a further 20 ml of water.

12.4.3.3 Add 0.5 ml of the phenolphthalein solution (12.2.3) and titrate immediately with the standard volumetric hydrochloric acid solution (12.2.2) until the pink colour is discharged.

12.5 Expression of Results

12.5.1 Ester Content

Ester content, expressed as a percentage by mass of the phthalates is given by the formula:

$$\frac{M(v_1 - v_2)}{20 m} - \frac{M \times A}{146}$$

where

- M = relative molar mass of the phthalates (see Table 3);
- v_1 = volume in ml, of the standard volumetric hydrochloric acid solution (12.2.2) required for the blank test;
- v_2 = volume, in ml, of the standard volumetric hydrochloric acid solution (12.2.2) required for the titration of the test portion;

A = acidity, expressed as a percentage by mass of phthalic acid, determined by the method specified in 11; and

m = mass, in g, of the test portion.

12.5.2 Saponification and Ester Value

In the case of mixed alcohol esters, for which the relative molar mass can only, at best, be estimated, saponification or ester values are used to express the result.

Saponification value, expressed as milligrams of potassium hydroxide per gram, is given by the formula:

$$\frac{56.10 (v_1 - v_2)}{m}$$

where 56.10 is the relative molar mass of potassium hydroxide.

Ester value, expressed as milligrams of potassium hydroxide per gram, is given by the formula

$$\frac{56.10 (v_1 - v_2)}{m} - 0.561 B$$

where B is the acidity, expressed as milliequivalents per kilogram, determined by the method specified in 11.

The ester content, as a percentage by mass of the ester concerned, may be estimated from the ester value by multiplication by the factor:

$$\frac{M}{561.0 n}$$

where n is the basicity of the acid (2 in the case of phthalate esters).

13 MEASUREMENT OF COLOUR AFTER HEAT TREATMENT

13.1 Principle

Measurement of the colour of the sample after a specified heat treatment.

13.2 Apparatus

Apparatus specified in 6.

13.2.1 Tube of borosilicate glass, about 200 mm long, with an external diameter of 38 ± 1 mm, walls 1.0 ± 0.1 mm thick, and provided with a vented ground glass stopper.

13.2.2 Oil bath, capable of being thermostatically controlled at $180 \pm 2^\circ\text{C}$ and of a size such that this temperature is maintained when the glass tube (13.2.1) is introduced. A volume of about 2 000 ml of oil is suitable for testing three samples at the same time.

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13.3 Procedure

13.3.1 Transfer 70 ml of the laboratory sample to the glass tube (13.2.1) and close it with its stopper. Place it in the oil bath (13.2.2), controlled at $180 \pm 2^\circ\text{C}$, making sure that the temperature of the bath remains within the specified limits, and support the tube so that the surface of the test portion is 25 mm below the surface of the oil.

13.3.2 At the end of 120 ± 2 min, remove the tube containing the sample from the oil bath and allow it to cool, in air, to room temperature. (see Note). Measure the colour of the heat-treated sample by the method specified in 6 using equal volumes of sample and standard Hazen matching solutions in, for example, 50 ml Nessler cylinders.

NOTE — If the sample is normally solid at room temperature, cool it to a temperature 10°C above its melting point.

13.4 Expression of Results

See 6.7, but quote also the colour of the untreated sample, determined by the method specified in 6.

14 DETERMINATION OF VOLATILE LOSS

14.1 Outline of the Method

Volatile loss is determined from loss of weight of the plasticizers when kept at $130 \pm 2^\circ\text{C}$ for 3 h.

14.2 Apparatus

14.2.1 Oven, convection type, capable of being controlled at $130 \pm 2^\circ\text{C}$.

14.2.2 Petridish, glass, 75 mm dia.

14.3 Procedure

14.3.1 Weigh accurately 10 g of the sample in the petridish.

14.3.2 Keep the petridish in the oven controlled at $130 \pm 2^\circ\text{C}$ for 3 h.

14.3.3 Remove the petridish and cool in a desiccator.

14.3.4 Weigh to the nearest 0.1 mg.

14.4 Expression of Results

Volatile loss, as percentage by mass, is given by the formula:

$$\frac{100 (m_0 - m_1)}{m_0}$$

where

m_0 = mass, in g of the test portion; and

m_1 = mass, in g of the residue.

15 DETERMINATION OF COLOUR VALUE BY LOVIBOND SCALE (SUCROSE TEST)

15.1 General

This empirical test is very sensitive to variations in procedure, even those apparently trivial, and these

instructions should therefore be followed as closely as possible. It is essential that all operations be performed in an atmosphere free from acid fumes and from sulphur compounds, and that all apparatus be thoroughly clean and free from traces of sulphur compounds.

15.2 Apparatus and Reagent

The following apparatus and reagent are required:

15.2.1 Two Test Tubes, of borosilicate glass, size 150 mm \times 25 mm.

15.2.2 Oil Bath, of 2 litres capacity, containing 1 750 ml of oil, measured at room temperature, and fitted with an efficient stirrer, a thermometer (one of the two quoted below), a support for the test tubes and a means of heating the oil.

15.2.3 Drying Oven, electrically or steam heated, maintained at $100 \pm 2^\circ\text{C}$ and suitably ventilated.

15.2.4 Two Thermometers any convenient, thermometer of a suitable range with 0.1°C subdivisions (see IS 4825 : 1982).

15.2.5 Trough, about 350 mm to 375 mm long, 50 mm wide and semi-circular in section. The trough shall be made of copper or other material non-reactive to sucrose solution.

15.2.6 Sucrose Paper, strips 75 mm \times 50 mm, prepared from sucrose (analytical reagent quality) and ashless, acid-washed filter paper (Whatman No. 40 or equivalent).

Sucrose paper may be conveniently prepared in sheets 350 mm \times 280 mm as follows:

Dissolve 30 g of sucrose in 150 ml of distilled water and pour the solution into the trough. Draw the sheets of filter paper one at a time through the solution grasping one end by a suitable clip (leaving about 25 mm of the paper dry at the end), and laying a glass rod about 10 mm in diameter over the paper in the trough so as to submerge it in the solution. Allow the paper to drain with the lower edge in contact with the trough for about 10 s. Transfer the paper to the drying oven and allow it to dry, hanging freely from the undipped edge, for 30 minutes.

When dry, cut off a border 38 mm in depth from the top edge of the sheet, and 12 mm wide from each of the other edges. Finally, cut the trimmed sheets into pieces of 75 mm \times 50 mm. Avoid handling the paper with the fingers, or with anything other than perfectly clean forceps and scissors. Store the cut pieces in a clean, dry glass-stoppered bottle and use them within one month of preparation.

15.3 Procedure

Wash both the test tubes thoroughly, rinse with hot distilled water, dry in the oven and allow to cool.

Into each tube insert a piece of sucrose paper rolled into the form of a cylinder about 25 mm in diameter and 50 mm high, pushing it down with clean forceps so as to line the lower end of the tube.

Place 25 ml of the sample into each tube, and transfer the unstoppered tubes into the oil bath already heated to 182°C, as indicated by the thermometer in the oil. Support the tubes so that the surface of the sample is approximately 25 mm below the surface of the oil, and support a thermometer in the sample in one of them.

Adjust the rate of heating of the oil bath so that the temperature, as indicated by the thermometer in the sample, reaches 180°C in not less than 8 minutes and not more than 10 minutes. Maintain the temperature of the sample thereafter at 180 ± 1°C.

At the end of 70 minutes from the time of immersion of the tubes in the oil, withdraw the tubes and allow them to cool in air to room temperature. When cool, pour off the sample from each tube into a clean vessel, mix it thoroughly and measure its colour in a 25 mm cell in a Lovibond Tintometer. If the sample is turbid or contains suspended matter, filter it through a dry filter paper (Whatman No. 1 or equivalent) before measuring the colour.

15.4 Expression of Results

Express the result of the test by adding the red, yellow and blue colour units for each tube separately.

16 DETERMINATION OF MATTER OXIDIZABLE BY POTASSIUM DICHROMATE

16.1 Reagents

16.1.1 Unless specified otherwise pure chemicals (A.R. Grade) and distilled water (see IS 1070 : 1992) shall be used throughout.

16.1.2 Oxidizing Solution

Dissolve approximately 1 g of potassium dichromate in 100 ml of water. Then continuously add 150 ml of concentrated nitric acid, 70 percent (m/m) (16 N). Cool the flask during the addition of the nitric acid. Store the solution in a dark-coloured bottle.

16.1.3 Potassium Iodide (50 g/l Solution)

Dissolve 50 g of potassium iodide in 1 litre of water.

16.1.4 Sodium Thiosulphate

0.1 N standard volumetric solution.

16.1.5 Cyclohexane

Wash 1 litre of cyclohexane with the 100 ml portions of water.

16.1.6 Starch Indicator Solution

16.2 Procedure

16.2.1 Calibration

Weigh, to the nearest 1 mg, 500 mg of the pure alcohol (corresponding to the alcohol component of the ester) into a 50 ml one-mark volumetric flask. Dilute to the mark with the cyclohexane and mix.

1 ml of solution = 10.0 mg of alcohol

To four 100-ml stoppered separating funnels add 20, 19, 18 and 17 ml of the cyclohexane respectively, followed by 0, 1, 2 and 3 ml of the alcohol solution (corresponding to 0, 10, 20 and 30 mg of alcohol). Add, by pipette, 25.0 ml of the oxidizing solution into each separating funnel.

NOTE — On no account pipette the oxidizing solution by mouth.

Shake each funnel vigorously for exactly 5 minutes. Allow to settle and then run off the (lower) potassium dichromate layer into a 250 ml conical flask. Wash the cyclohexane layer with two 25 ml portion of water and add the washings (lower layer) to the dichromate solution. Add 100 ml of the potassium iodide solution, swirl to mix and allow the flask to stand in the dark for 10 minutes. Titrate the liberated iodine with 0.1 N sodium thiosulphate solution using starch as indicator. The end-point is the discharge of the dark blue of the starch to the light bluish-green of the resultant solution.

From the difference in titre between the blank and the calibration standards, draw a graph of the volume of the 0.1 N sodium thiosulphate solution against free alcohol content, in milligrams.

16.2.2 Test Portions

Use the following volumes of sample depending upon the expected oxidizable matter content:

Oxidizable matter, percent	Volume of sample, ml
< 0.2	20
0.2 to 0.5	10
0.5 to 1.0	5

16.2.3 Determination

Place 20 ml of the washed cyclohexane in a 100 ml stoppered separating funnel. Add the test portion (16.2.2) and then pipette 25.0 ml of the oxidizing

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solution into the funnel. Then follow the procedure given in 16.2.1.

$$\frac{M}{10V}$$

16.2.4 Blank Test

where

Carry out a blank determination as described in 16.2.3, but omitting the test portion.

M = mass, in mg, of alcohol corresponding to the difference between the volumes of standard volumetric sodium thiosulphate solution used for the determination and for the blank test; and

16.3 Calculation

Content of oxidizable matter, expressed as a percentage by mass/volume of the free alcohol

V = volume, in ml, of the test portions.

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