

REAFFIRMED १०.०३

IS 14421 : 1997
(Superseding IS 3371 : 1980,
IS 3672 : 1980, IS 6627 : 1980
and IS 9572 : 1980)

भारतीय मानक

सुघट्टयकारी ईस्टर — विशिष्टि (Reaffirmed 2013)

Indian Standard

PLASTICIZER ESTERS — SPECIFICATION

ICS 71.080.70; 83.040.30

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

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Price Group 2

Plastics Sectional Committee, PCD 12

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.

A plasticizer has been defined as a material incorporated in a plastic to increase its workability and its flexibility or distensibility. The addition of the plasticizer may lower the melt-viscosity, the second-order transition temperature or the elastic modulus of the plastics. For a plasticizer to be effective with any polymeric material, the two must be intimately mixed. The use of any plasticizer requires a compromise of desirable properties in every case and as such the choice of a specific plasticizer for a given use is not easy. The efficiency of a plasticizer refers to its ability to bring about a desired effect. The more plasticizer required to produce the effect, the less efficient it is. Many attempts have been made to compare the efficiency of one plasticizer with that of another by one-number efficiency factor. Results are highly dependent on the test method and use property of interest. Usually more viscous plasticizers are less efficient than less viscous ones; however, there are minor exceptions. A high proportion of compatibility inducing groups in the molecule lowers efficiency. The principal exception is with linear polyester plasticizers; their high molecular weight leads to high viscosity but, because of predominantly aliphatic linear structures, they react almost segment by segment and are far more efficient as plasticizers than otherwise would be predicted.

The principal use of the plasticizer is with the thermoplastic resins, which they may modify slightly only for easier processing or which they may modify into radically different materials. Polyvinyl chloride especially suited for beneficial modification by plasticizers and accounts for the greatest use of organic plasticizers. Organic plasticizers are usually moderately high molecular weight liquids or occasionally low-melting solids. Most commonly, they are esters of carboxylic acids or phosphoric acids, for example, phthalates, adipates, sebacates, azelates, etc. Other types include hydrocarbons, halogenated hydrocarbons, ethers, polyglycols and sulfonamides.

Phthalate esters, particularly dialkyl phthalates are considered as general purpose plasticizers and are widely used in view of their all-round performance, for example, compatibility with the resin, efficiency in flexibilizing, low volatility and resistance to extraction. Dialkyl phthalate plasticizers have dominated the plasticizer technology since 1930s. For many years, di-2-ethyl hexyl phthalate (commonly known as DOP) was the accepted industry standard for a general purpose plasticizer for PVC and is a bench mark for comparison of other plasticizers. Its all-round performances are so good that it alone has accounted for a fourth of the total plasticizer production.

In this standard only the phthalate esters, particularly dialkyl phthalates, have been covered amongst the wide range of plasticizer esters considering their major applications as plasticizer with resin like polyvinyl chloride and other vinyl polymers; cellulosic plastics such as cellulose acetate, cellulose acetobutyrate, cellulose nitrate; synthetic rubbers; shallac; alkydes and phenolic resins. Other uses include good film formation properties in paint industry, imparting good tack in adhesives and emulsions, preparations of insect repellants, fixatives, etc.

Indian Standard on phthalate plasticizer ester was first published in 1965, namely, 'Specification for Di-*n*-butyl phthalate' (IS 3371 : 1965) followed by formulation of standards on 'Di-octyl phthalate' (IS 3672 : 1966) and 'Dimethyl phthalate plasticizer' (IS 6627 : 1972), respectively. All these standards were revised in 1980 incorporating additional test requirements and aligning the various methods of tests with the corresponding overseas standards. Subsequently, IS 9572 : 1980 'Benzyl butyl phthalate plasticizer' was formulated considering its importance amongst the phthalate plasticizer.

Considering the applicability of the various changes and the advancements those have taken place in the field of plasticizers, the requirements of both the manufacturers and users and also to effect a possible merger of the existing four Indian Standards on phthalate plasticizers, the concerned Technical Committee had critically reviewed the existing Indian Standards and decided to formulate this Indian Standard in line with the practices followed overseas. With the publication of this standard the existing standards

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**AMENDMENT NO. 1 MARCH 2003
TO
IS 14421 : 1997 PLASTICIZER ESTERS —
SPECIFICATION**

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CD 12)

Reprography Unit, BIS, New Delhi, India

Indian Standard

PLASTICIZER ESTERS — SPECIFICATION

1 SCOPE

This standard specifies the requirements, methods of sampling and tests for various types of plasticizer esters (phthalates only) for use as plasticizers for plastics and for other purposes requiring similar quality of materials.

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 editions of the standards indicated below:

IS No.	Title
2828 : 1964	Glossary of terms used in plastics industry
9591 : 1996	Plasticizer esters—Methods of sampling and tests

3 TERMINOLOGY

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

4 TYPES

There shall be eight types of the materials as indicated below:

- Type 1** Di-*n*-butyl phthalate, $[C_6H_4(COOC_4H_9)_2]$, CAS Registry No. (84-74-2), consisting essentially of the di-ester of phthalic acid and butane-1-ol (*n*-butanol) commonly known as *DBP*.
- Type 2** Di-(2-ethylhexyl) phthalate, $[C_6H_4(COOCH_2CH(C_2H_5)C_4H_9)_2]$, CAS Registry No. (117-81-7), consisting essentially of the diester of phthalic acid and 2-ethylhexanol, commonly known as *DOP*.
- Type 3** Di-isooctyl phthalate $[C_6H_4(COOCH_2CH(C_2H_5)C_4H_9)_2]$, CAS Registry No. (27554-26-3), consisting essentially of the diester of phthalic acid and iso octanol, commonly known as *DIOP*.
- Type 4** Dimethyl phthalate $[C_6H_4(COOCH_3)_2]$, CAS Registry No.

(131-11-3), consisting essentially of the diester of phthalic acid and methanol, commonly known as *DMP*.

- Type 5** Diethyl phthalate, $[C_6H_4(COOC_2H_5)_2]$, CAS Registry No. (84-66-2), consisting essentially of the diester of phthalic acid and ethanol, commonly known as *DEP*.
- Type 6** Di-(2-methyl propyl) phthalate, $\{C_6H_4[COOCH_2CH(CH)_3CH_3]_2\}$, CAS Registry No. (84-69-5) consisting essentially of the diester of phthalic acid and 2-methylpropan-1-ol (iso-butanol), commonly known as *DIBP*.
- Type 7** Di-isodecyl phthalate, $[C_6H_4(COOC_{10}H_{21})_2]$ CAS Registry No. (2761-42-0), consisting essentially of the diester of phthalic acid and iso decanol, commonly known as *DIDP*.
- Type 8** Benzyl butyl phthalate $[C_6H_4(COOC_4H_9.COOCH_2C_6H_5)]$ CAS Registry No. (85-68-7), consisting essentially of the diester of phthalic acid and butyl benzyl alcohol, commonly known as *BBP*.

5 REQUIREMENTS

5.1 Description

The materials shall be clear liquids and free from sediments and matter in suspension, as determined by visual inspection.

5.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Col 11 of Table 1.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in well-closed containers as agreed to between the purchaser and the supplier.

6.2 Marking

The containers shall be securely closed and legibly and indelibly marked with the following information:

- a) Indication of the source of manufacture and recognized trade-mark, if any;

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- b) Name and type of the material;
- c) Tare, gross and net mass;
- d) Date of manufacture; and
- e) Batch No. or Code No.

6.3 BIS Certification Marking

The containers may also be marked with the Standard Mark.

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

7 SAMPLING AND CRITERIA FOR CONFORMITY

7.1 The method of drawing representative samples of the material shall be as prescribed in 4 of IS 9591 : 1996 using appropriate instruments.

7.2 Number of Tests

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

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

7.3 Criteria for Conformity

7.3.1 For Individual Samples

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

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

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

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

7.3.2 For Composite Sample

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

7.3.3 A lot shall be declared as conforming to the specification, if it satisfies the requirements for each of the characteristics as prescribed under 5.

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

Table 1 Requirements for Plasticizer Esters
(Clause 5.2)

SI No.	Characteristics	Requirements for Types								Methods of Test Ref to Cl No. of IS 9591 : 1996
		1(DBP)	2(DOP)	3(DIOP)	4(DMP)	5(DEP)	6(DIBP)	7(DIDP)	8(BBP)	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
i)	Colour, in Hazen units, <i>Max</i>	100	100	100	100	100	100	75	150	6
ii)	Relative density at 27/27°C	1.041 to 1.045	0.980 to 0.986	0.980 to 0.986	1.186 to 1.191	1.113 to 1.118	1.034 to 1.041	0.963 to 0.968	1.114 to 1.120	7
iii)	Refractive index at 27°C	1.489 to 1.492	1.484 to 1.488	1.484 to 1.488	1.511 to 1.514	1.499 to 1.502	1.483 to 1.487	1.482 to 1.486	1.538 to 1.542	8
iv)	Water content, percent by mass, <i>Max</i>	0.10	0.10	0.10	0.15	0.15	0.10	0.10	0.10	9
v)	Ash, percent by mass, <i>Max</i>	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	10
vi)	Acidity (as phthalic acid), percent by mass, <i>Max</i>	0.01	0.02	0.02	0.01	0.01	0.01	0.02	0.025	11
vii)	Ester value (as mg KOH/g)	399 to 405	284 to 290	284 to 290	571 to 580	499 to 507	399 to 405	248 to 254	355 to 363	12
viii)	Colour after heat treatment, in Hazen units, <i>Max</i>	150	75	150	150	150	100	200	250	13
ix)	Volatile loss, percent by mass, <i>Max</i> (130 ± 2°C for 3 h)	0.4	0.1	0.1	1.0	1.0	0.4	0.1	0.2	14
x)	Sucrose test ¹⁾ (Total colour value), Lovibond scale, <i>Max</i>	—	—	—	15	15	—	—	—	15
xi)	Matter oxidizable by ²⁾ potassium dichromate, percent by mass, <i>Max</i>	—	0.07	0.10	—	—	—	—	—	16

¹⁾ This test is designed to detect trace impurities that would affect the suitability of the material for plasticizing cellulose acetate.

²⁾ Results are calculated as free alcohol content of the esters like 2-ethylhexanol and octanol in case of *DOP* and *DIOP*, respectively.

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on phthalate plasticizers, namely, IS 3371 : 1980; IS 3672 : 1980; IS 6627 : 1980 and IS 9572 : 1980 respectively would become redundant.

Considerable assistance has been derived from the following publications while preparing this standard:

BS 573, 574, 1995, 1996, 2535, 2536, 3647 : 1973 Specification for plasticizer esters. British Standards Institution.

BS 4968-70 : 1973 Specifications for Di-isobutyl phthalate, Di-isooctyl phthalate, Di-Isooctyl sebacates. British Standards Institution.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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Amendments Issued Since Publication

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