INTERNATIONAL STANDARD 1385/V

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Phthalate esters for industrial use — Methods of test — Part V : Determination of ester content — Titrimetric method after saponification

Phtalates à usage industriel — Méthodes d'essai —
Partie V : Dosage des esters — Méthode titrimétrique après saponification

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1385-1970 and found it technically suitable for transformation. The technical committee, however, divided the Recommendation into five parts (ISO 1385 Parts I to V), which therefore replace ISO Recommendation R 1385-1970, to which they are technically identical.

ISO Recommendation R 1385 had been approved by the member bodies of the following countries :

Austria	Iran	Romania
Belgium	Ireland	South Africa, Rep. of
Brazil	Italy	Spain
Cuba	Japan	Sweden
Czechoslovakia	Korea, Rep. of	Switzerland
France	Netherlands	Thailand
Germany	New Zealand	Turkey
Hungary	Poland	United Kingdom
India	Portugal	U.S.S.R.

No member body had expressed disapproval of the Recommendation.

The member bodies of the following countries disapproved the transformation of the Recommendation into an International Standard :

France
Netherlands

Printed in Switzerland

Phthalate esters for industrial use — Methods of test — Part V : Determination of ester content — Titrimetric method after saponification

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1385 specifies a titrimetric method, after saponification, for the determination of the ester content of phthalate esters for industrial use.

This document should be read in conjunction with part I (see the annex).

2 PRINCIPLE

Saponification of the ester in a test portion, using a measured excess of a potassium hydroxide ethanolic solution, followed by back-titration with standard volumetric hydrochloric acid solution, in the presence of phenolphthalein as indicator.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, freshly boiled and cooled.

3.1 Potassium hydroxide, approximately 1 N solution in 95 % (*V/V*) ethanol.

3.2 Hydrochloric acid, 1 N standard volumetric solution.

3.3 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (*V/V*) ethanol and make faintly pink by addition of dilute sodium hydroxide solution.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Two conical flasks, of borosilicate glass, of capacity 250 ml, fitted with ground glass stoppers.

4.2 Water-cooled reflux condensers, with ground glass joints to fit the flasks (4.1).

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

5 PROCEDURE

5.1 Introduce, successively from the same one-mark pipette, 50 ml of the potassium hydroxide solution (3.1) into each of the two conical flasks (4.1) and add immediately 5 ml of water to each flask. By means of the weighing pipette (4.3), transfer immediately a test portion of the laboratory sample, in accordance with the table, into one of the flasks.

Ester	Alkyl radical R	Mass of test portion g	Relative molar mass *M*
Dimethyl phthalate	CH_3	2,2 to 2,6	194,2
Diethyl phthalate	C_2H_5	2,6 to 3,0	222,2
Di-isopropyl phthalate	C_3H_7	2,9 to 3,3	250,3
Diallyl phthalate	C_3H_5	2,9 to 3,3	246,3
Di-isobutyl phthalate Di-*n*-butyl phthalate	C_4H_9	3,3 to 3,7	278,3
Dihexyl phthalates	C_6H_{13}	4,0 to 4,4	334,4
Diheptyl phthalates	C_7H_{15}	4,3 to 4,7	362,5
Dioctyl phthalates	C_8H_{17}	4,7 to 5,1	390,5
Dinonyl phthalates	C_9H_{19}	5,0 to 5,4	418,6
Didecyl phthalates	$C_{10}H_{21}$	5,4 to 5,8	446,6
Ditridecyl phthalates	$C_{13}H_{27}$	6,2 to 6,6	530,8
Dibutoxyethyl phthalate	$C_6H_{13}O$	4,4 to 4,8	366,4
Benzyl butyl phthalate	*	3,7 to 4,1	312,3

* Benzyl butyl phthalate is represented by the formula

$$C_6H_5 - CH_2OOCC_6H_4COO - C_4H_9$$

5.2 Attach the flasks to reflux condensers (4.2), and heat for 1 h in a boiling water bath. Withdraw the flasks, still carrying their condensers, and immerse them in cold running water. When cold, wash down the inside of each condenser with two 20 ml portions of water. Disconnect the flasks and wash each joint with a further 20 ml of water.

5.3 Add 0,5 ml of the phenolphthalein solution (3.3) to each flask and immediately titrate the mixture and the blank successively with the standard volumetric hydrochloric acid solution (3.2) until the pink colour disappears.

6 EXPRESSION OF RESULTS

The ester content, expressed as a percentage by mass of the phthalate $C_6H_4(COOR)_2$, where **R** is the alkyl radical present in the phthalate (see the table in 5.1), is given by the formula

$$\frac{\frac{M}{2}}{1\,000}\left[(V_0 - V_1) - \frac{\left(\frac{A \times m}{100}\right)}{\frac{166}{2}} \times 1\,000\right] \times \frac{100}{m}$$

$$= \frac{M\,(V_0 - V_1)}{20\,m} - \frac{M \times A}{166}$$

where

M is the relative molar mass of the phthalate (see the table in 5.1);

V_0 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (3.2) used for the blank;

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (3.2) required by the unreacted potassium hydroxide after saponification of the test portion;

A is the acidity to phenolphthalein, expressed as a percentage by mass of phthalic acid (see part IV);

m is the mass, in grams, of the test portion;

$\frac{166}{2}$ is the mass, in grams, of phthalic acid corresponding to 1 000 ml of exactly 1 N potassium hydroxide solution.

NOTE – If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

ANNEX

ISO PUBLICATIONS RELATING TO PHTHALATE ESTERS FOR INDUSTRIAL USE

ISO 1385/I – General.*

ISO 1385/II – Measurement of colour after heat treatment (Diallyl phthalate excluded).

ISO 1385/III – Determination of ash.

ISO 1385/IV – Determination of acidity to phenolphthalein – Titrimetric method.

ISO 1385/V – Determination of ester content – Titrimetric method after saponification.

* The determination of the iodine value specified in ISO 1385/I is applicable only to diallyl phthalate.
The determination of the viscosity specified in ISO 1385/I is not applicable to diallyl phthalate.