
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

Phthalates 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

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).

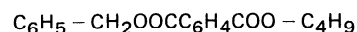
4.3 Weighing pipette, capable of delivering up to 10 g of the sample.

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	Relative molar mass M
		g	
Dimethyl phthalate	CH ₃	2,2 to 2,6	194,2
Diethyl phthalate	C ₂ H ₅	2,6 to 3,0	222,2
Di-isopropyl phthalate	C ₃ H ₇	2,9 to 3,3	250,3
Diallyl phthalate	C ₃ H ₅	2,9 to 3,3	246,3
Di-isobutyl phthalate	C ₄ H ₉	3,3 to 3,7	278,3
Di-n-butyl phthalate			
Di-hexyl phthalates	C ₆ H ₁₃	4,0 to 4,4	334,4
Di-heptyl phthalates	C ₇ H ₁₅	4,3 to 4,7	362,5
Di-octyl phthalates	C ₈ H ₁₇	4,7 to 5,1	390,5
Dinonyl phthalates	C ₉ H 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 ₆ H 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



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.