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# INTERNATIONAL STANDARD



# 2527

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION · МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ · ORGANISATION INTERNATIONALE DE NORMALISATION

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## Adipate esters for industrial use — Determination of ester content — Volumetric method

*Esters de l'acide adipique à usage industriel — Dosage des esters — Méthode volumétrique*

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**Descriptors** : esters, adipates, chemical analysis, determination of content, volumetric analysis.

## 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.

International Standard ISO 2527 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1971.

It has been approved by the Member Bodies of the following countries :

Austria	Ireland	Switzerland
Belgium	Israel	Thailand
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	Poland	U.S.A.
Germany	Romania	U.S.S.R.
Hungary	South Africa, Rep. of	
India	Spain	

No Member Body expressed disapproval of the document.

# Adipate esters for industrial use – Determination of ester content – Volumetric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of the ester content of adipate esters for industrial use.

The method is applicable to simple and mixed alcohol esters that are liquid at ambient temperature.

## 2 REFERENCE

ISO 2525, *Adipate esters for industrial use – Determination of acidity to phenolphthalein – Volumetric method.*

## 3 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.

## 4 REAGENTS

Distilled water, or water of equivalent purity, freshly boiled and cooled, shall be used in the test.

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

**4.2 Hydrochloric acid**, N standard volumetric solution.

**4.3 Phenolphthalein**, 10 g/l ethanolic solution.

Dissolve 1,0 g of phenolphthalein in 100 ml of 95 % (V/V) ethanol and make faintly pink by the addition of dilute sodium hydroxide solution.

## 5 APPARATUS

Ordinary laboratory apparatus and

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

**5.2 Water-cooled reflux condensers**, with ground glass joints to fit the flasks (5.1).

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

## 6 PROCEDURE

### 6.1 Test portion

Weigh, to the nearest 0,01 g, the mass of the laboratory sample indicated in the following table.

Sample	R	Mass of test portion	M
		g	
dibutyl adipates	C <sub>4</sub> H <sub>9</sub>	3,0 to 3,4	258,4
dioctyl adipates	C <sub>8</sub> H <sub>17</sub>	4,4 to 4,8	370,6
dinonyl adipates	C <sub>9</sub> H <sub>19</sub>	4,8 to 5,2	398,6
didecyl adipates	C <sub>10</sub> H <sub>21</sub>	5,1 to 5,5	426,6
diundecyl adipates	C <sub>11</sub> H <sub>23</sub>	5,4 to 5,8	454,7
didodecyl adipates	C <sub>12</sub> H <sub>25</sub>	5,8 to 6,2	482,7
ditridecyl adipates	C <sub>13</sub> H <sub>27</sub>	6,0 to 6,4	510,8

where

**R** is the alkyl radical present in the adipate;

**M** is the relative molar mass of the adipate.

In the case of mixed alcohol adipate 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

$$\frac{0,025 M}{n} \pm 0,2 \text{ g}$$

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

### 6.2 Blank test

At the same time as the determination, carry out a blank test using the same reagents and following exactly the same procedure as described in 6.3, but omitting the test portion.

### 6.3 Determination

**6.3.1** Introduce 50,0 ml of the potassium hydroxide solution (4.1) into one of the two 250 ml conical flasks (5.1), and add immediately 5 ml of water. By means of the weighing pipette (5.3) transfer the test portion immediately to the flask.

**6.3.2** Attach the flask (5.1) to the condenser (5.2) 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.

**6.3.3** Add 0,5 ml of the phenolphthalein solution (4.3) and titrate immediately with the standard volumetric hydrochloric acid solution (4.2) until the pink colour is discharged.

## 7 EXPRESSION OF RESULTS

### 7.1 Ester content

Ester content, expressed as a percentage by mass of the adipate  $(\text{CH}_2\text{CH}_2\text{COOR})_2$ , where **R** is the alkyl radical present in the adipate, is given by the formula :

$$\frac{M(V_1 - V_2)}{20m} - \frac{M \times A}{146}$$

where

$V_1$  is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (4.2) required for the blank test;

$V_2$  is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (4.2) required for the titration of the test portion;

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

$A$  is the acidity, expressed as a percentage by mass of adipic acid, determined by the method specified in ISO 2525;

$M$  is the relative molar mass of the adipate (see the table in 6.1).

### 7.2 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 milli-equivalents per kilogram, determined by the method specified in ISO 2525.

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

$$\frac{M}{561,0n}$$

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

## 8 TEST REPORT

The test report shall include the following particulars :

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard or the document to which reference is made, or regarded as optional.

ANNEX

This document forms one of a series on methods of test for adipate esters for industrial use.

The list of documents already prepared is as follows :

ISO 2523 – *List of methods of test .*

ISO 2524 – *Measurement of colour after heat treatment .*

ISO 2525 – *Determination of acidity to phenolphthalein – Volumetric method .*

ISO 2526 – *Determination of ash – Gravimetric method .*

ISO 2527 – *Determination of ester content – Volumetric method .*

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