# INTERNATIONAL STANDARD

Fourth edition 2013-07-15

# Animal and vegetable fats and oils — Determination of saponification value

*Corps gras d'origines animale et végétale — Détermination de l'indice de saponification* 

# iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>ISO 3657:2013</u> https://standards.iteh.ai/catalog/standards/sist/ef2a435d-910a-4c92-94ed-4a8d67739852/iso-3657-2013



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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2, www.iso.org/directives.

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The committee responsible for this document is ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*. **Teh STANDARD PREVIEW** 

This fourth edition cancels and replaces the third edition (150 3657:2002), which has been technically revised.

<u>ISO 3657:2013</u> https://standards.iteh.ai/catalog/standards/sist/ef2a435d-910a-4c92-94ed-4a8d67739852/iso-3657-2013

# Animal and vegetable fats and oils — Determination of saponification value

#### 1 Scope

This International Standard specifies a method for the determination of the saponification value of animal and vegetable fats and oils. The saponification value is a measure of the free and esterified acids present in fats and fatty acids.

The method is applicable to refined and crude vegetable and animal fats.

If mineral acids are present, the results given by this method are not interpretable unless the mineral acids are determined separately.

The saponification value can also be calculated from fatty acid data obtained by gas liquid chromatography analysis as given in <u>Annex B</u>. For this calculation, it is necessary to be sure that the sample does not contain major impurities or is thermally degraded.

#### 2 Normative references

# The following documents, in whole or in part, are normatively referenced in this document and are

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 661, Animal and vegetable fats and oils Separation of test sample

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#### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

#### 3.1

#### saponification value

number of milligrams of potassium hydroxide required for the saponification of 1 g of the product tested

Note 1 to entry: The saponification value is a dimensionless quantity. The usual abbreviation is SV.

#### 4 Principle

The test sample is saponified by boiling under reflux with an excess of ethanolic potassium hydroxide, followed by titration of the excess potassium hydroxide with standard volumetric hydrochloric acid solution.

#### **5** Reagents

Use only reagents of recognized analytical grade, and distilled or demineralized water of equivalent purity.

**5.1 Ethanol**, volume fraction  $\varphi = 95$  %.

#### **5.2 Potassium hydroxide**, *c*(KOH) = 0,5 mol/l solution in ethanol.

This solution shall be colourless or straw yellow. A stable colourless solution can be prepared by either of the following procedures.

- a) Reflux 1 l of ethanol (5.1) with 8 g of potassium hydroxide and 5 g of aluminium pellets for 1 h, then distil immediately. Dissolve the required amount of potassium hydroxide (approximately 35 g) in the distillate. Allow to stand for several days, then decant the clear supernatant liquid from the precipitated potassium carbonate into a brown-glass stock bottle.
- b) Add 4 g of aluminium *tert*-butylate to 1 l of ethanol and allow the mixture to stand for several days. Decant the supernatant liquid and dissolve in it the required amount of potassium hydroxide. Allow to stand for several days, and then decant the clear supernatant liquid from the precipitated potassium carbonate into a brown-glass stock bottle.
- **5.3 Hydrochloric acid**, standard volumetric solution, *c*(HCl) = 0,5 mol/l.
- **5.4 Phenolphthalein solution**,  $\rho = 0,1$  g/100 ml in ethanol (5.1).
- **5.5** Alkali blue 6B solution,  $\rho = 2,5$  g/100 ml in ethanol (5.1).
- 5.6 Boiling aids.

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## Apparatus iTeh STANDARD PREVIEW

Usual laboratory apparatus and, in particular, the ford mg. teh.ai)

- 6.1 Conical flask, of 250 ml capacity, made of alkali resistant glass and having a ground neck. https://standards.iteh.ai/catalog/standards/sist/ef2a435d-910a-4c92-94ed-
- **6.2 Reflux condenser**, with a ground glass joint which fits the conical flask (<u>6.1</u>).

**6.3** Heating device (e.g. a water-bath, electric hot-plate or other suitable apparatus). A naked flame is not suitable.

- 6.4 Burette, capacity 50 ml, graduated in 0,1 ml divisions or automatic burette.
- 6.5 **Pipette**, capacity 25 ml or an **automatic pipette**.
- **6.6 Analytical balance,** readability 0,000 1 g, weighing precision 0,001 g.

#### 7 Sampling

Sampling is not part of this method specified in this International Standard. A recommended sampling method is given in ISO 5555.

It is important that the laboratory receive a truly representative sample which has not been damaged or changed during transport or storage.

#### 8 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

The test samples are carefully mixed and filtered if visible impurities are present; if filtration is necessary, this shall be mentioned in the test report.

#### 9 Procedure

#### 9.1 Test portion

Weigh, to the nearest 5 mg, approximately 2 g of the test sample (<u>Clause 8</u>) into a conical flask (<u>6.1</u>).

The test portion of 2 g has been determined on the basis of saponification values of 170 to 200. For other saponification values, the mass should be altered accordingly so that approximately half the ethanolic potassium hydroxide solution is neutralized. Recommendations for the mass of the test portion are given in Table 1.

Expected saponification value	Mass of test portion		
150 to 200	2,2 to 1,8 g		
200 to 250	1,7 to 1,4 g		
250 to 300	1,3 to 1,2 g		
>300	1,1 to 1,0 g		

Table 1 — Mass of test portion

#### 9.2 Determination

**9.2.1** Using a pipette (6.5), add to the test portion 25,0 ml of the ethanolic potassium hydroxide solution (5.2) and some boiling aids (5.6). Connect the reflux condenser (6.2) to the flask, place the flask on the heating device (6.3) and boil gently, shaking from time to time, for 60 min or for 2 h in the case of oils and fats having a high melting point and which are difficult to saponify.

**9.2.2** Add to the hot solution 0.5 ml to 1 ml of the phenolphthalein solution (5.4) and titrate with the standard volumetric hydrochloric acid solution (5.3) until the pink colour of the indicator just disappears. If the solution is strongly coloured, use 0.5 ml to 1 ml of alkali blue 6 B solution (5.4).

#### 9.3 Blank test

Carry out a blank test following the procedure specified in 9.2, using another 25,0 ml of the ethanolic potassium hydroxide solution (5.2) but omitting the test portion.

#### **10 Expression of results**

The saponification value  $I_s$  is given by the equation

$$I_{\rm s} = \frac{\left(V_0 - V_1\right) \times c \times 56,1}{m}$$

where

- $V_0$  is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (5.3) used for the blank test;
- $V_1$  is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (5.3) used for the determination;
- *c* is the exact concentration, in moles per litre, of the standard volumetric hydrochloric acid solution (5.3);
- m is the mass, in grams, of the test portion (9.1).

Take as the result the arithmetic mean of the two determinations, provided that the requirement for repeatability (<u>Clause 11</u>) is satisfied.

Express the result as a whole number.

#### **11 Precision**

#### 11.1 Results of interlaboratory test

An interlaboratory test, carried out at the international level in 2000 by DIN, in which 22 laboratories participated, each of which carried out two determinations on each sample, gave the statistical results (evaluated in accordance with ISO 5725-1<sup>[2]</sup> and ISO 5725-2<sup>[3]</sup>) shown in <u>Table A.1</u>.

#### **11.2 Repeatability**

The absolute difference between two independent single test results, obtained using the same method in identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than the repeatability limit r given in Table A.1.

#### **11.3 Reproducibility**

The absolute difference between two single test results, obtained using the same method in identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than the reproducibility limit R given in Table A.1.

#### **12 Test report**

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The test report shall contain at least the following information -4c92-94ed-

- a) all information necessary for the complete identification of the sample;
- b) the sampling method used, if known;
- c) whether filtration of test sample(s) was necessary;
- d) the test method used, with reference to this International Standard (ISO 3657:2013);
- e) a statement of which indicator has been used, <u>5.4</u> or <u>5.5</u>;
- f) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- g) the test result(s) obtained, or if the repeatability has been checked, the final result obtained.

# Annex A (informative)

## **Results of an interlaboratory test**

An international collaborative test involving 22 laboratories in eight countries was carried out on five samples.

A: Coconut oil B: Palm oil C: Rapeseed oil D: medium chain triglyceride (MCT) oil E: Mixture by volume of 60 % A and 40 % D

The test was organized by DIN in 2000 and the results obtained were subjected to statistical analysis in accordance with ISO 5725-2<sup>[3]</sup> to give the precision data shown in <u>Table A.1</u>.

Parameter	Rapeseed oil	Palm oil	Coconut oil	60 % A + 40 % D	MCT oil
Number of participating laboratories (N)	22	22	22	22	20
Number of laboratories retained after eliminating outliers ( <i>n</i> ) <b>TANDAR</b>	D PRI	EV <sup>17</sup> EV	20	18	16
Number of individual test results of all laboratories on each sample (z)	.iteh.a	<b>i)</b> 34	40	36	32
Mean value ( $\overline{I}_{S}$ ) ISO 3657:2	<u>.013</u> 190,2	199,5	256,8	287,5	334,1
Repeatability standard deviation $(s_r)$	/sist/ef2a4350	-910a-4c92- 0,6	94ed-0,7	0,7	1,4
Repeatability coefficient of variation ( $C_{V,r}$ ) %	0,4	0,3	0,3	0,2	0,4
Repeatability limit (r)	1,1	0,8	0,8	0,6	1,6
Reproducibility standard deviation ( <i>s</i> <sub><i>R</i></sub> )	1,8	2,0	4,2	2,4	2,9
Reproducibility coefficient of variation ( $C_{V,R}$ ), %	0,9	1,0	1,6	0,8	0,9
Reproducibility limit (R)	2,5	2,8	4,5	2,2	2,5

#### Table A.1 — Summary of statistical results