INTERNATIONAL STANDARD

ISO 150

Third edition
2018-11

Raw, refined and boiled linseed oil for paints and varnishes — Specifications and methods of test

Huiles de lin brutes, raffinées et cuites, pour peintures et vernis — Spécifications et méthodes d'essai

Reference number
ISO 150:2018(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

This third edition cancels and replaces the second edition (ISO 150:2006), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the determination of the composition of fatty acids in accordance with ISO 12966 has been specified;

— ISO 2811-3 (oscillation method) has been added for determination of density;

— the determination of drying time has been deleted;

— reference to the use of the Lovibond colour system as an alternative to the Gardner colour scale has been deleted because the visual method is too inaccurate;

— sampling in accordance with ISO 15528 has been replaced by sampling in accordance with ISO 5555;

— the preparation of samples in accordance with ISO 661 has been added;

— the standard values for the composition of fatty acids have been revised;

— the normative references in Clause 2 have been updated and the text has been editorially revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Raw, refined and boiled linseed oil for paints and varnishes — Specifications and methods of test

1 Scope

This document specifies the requirements and the corresponding methods of test for raw, refined and boiled linseed oils for paints and varnishes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 660, *Animal and vegetable fats and oils — Determination of acid value and acidity*

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

ISO 835, *Laboratory glassware — Graduated pipettes*

ISO 2114, *Plastics (polyester resins) and paints and varnishes (binders) — Determination of partial acid value and total acid value*

ISO 2811-1, *Paints and varnishes — Determination of density — Part 1: Pycnometer method*

ISO 2811-3, *Paints and varnishes — Determination of density — Part 3: Oscillation method*

ISO 3681, *Binders for paints and varnishes — Determination of saponification value — Titrimetric method*

ISO 3961, *Animal and vegetable fats and oils — Determination of iodine value*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 4630, *Clear liquids — Estimation of colour by the Gardner colour scale*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

ISO 5555, *Animal and vegetable fats and oils — Sampling*

ISO 5661, *Petroleum products — Hydrocarbon liquids — Determination of refractive index*

ISO 6320, *Animal and vegetable fats and oils — Determination of refractive index*

ISO 8534, *Animal and vegetable fats and oils — Determination of water content — Karl Fischer method (pyridine free)*

ISO 12966 (all parts), *Animal and vegetable fats and oils — Gas chromatography of fatty acid methyl esters*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
raw linseed oil
oil obtained solely from mature seeds of linseed (*Linum usitatissimum* L.)

3.2
acid-refined linseed oil
oil obtained by refining raw linseed oil with acid

3.3
alkali-refined linseed oil
oil obtained by refining raw linseed oil with sodium hydroxide or other alkali solution

3.4
boiled linseed oil
oil obtained by incorporating driers in raw linseed oil or refined linseed oil and heating either alone or while blowing through air or oxygen

3.5
break
separation of an (insoluble) mucilaginous product which occurs when certain unrefined vegetable oils are heated

Note 1 to entry: When separation occurs, the oil is said to "break". The insoluble matter is also referred to as the "break".

4 Required characteristics and their tolerances

Raw, refined and boiled linseed oils shall have the characteristics specified in Table 1.

Table 1 — Required characteristics and their tolerances

Characteristic	Requirement				Test method
	Raw linseed oil	Alkali-refined linseed oil	Acid-refined linseed oil	Boiled linseed oil	
Density[a] ρ_{23} g/ml	0,924 to 0,931	0,924 to 0,931	0,924 to 0,931	0,926 to 0,948	ISO 2811-1 or ISO 2811-3
Gardner colour max.	13	4	6	To be agreed between the interested parties	ISO 4630
Gardner colour after heating max.	—	—[b]	—	—	—

[a] 23 °C is the standard temperature unless otherwise agreed: for example 20 °C, 25 °C, or 27 °C for tropical countries.

[b] If the acid value of neutral oil has been increased by the addition of fatty acids, then the requirement for colour after heating should be agreed upon between the interested parties, as the limits for neutral oil are not necessarily applicable.

[c] Stricter requirements may be agreed between the interested parties.

[d] Alkali-refined oil may have its acid value adjusted to other limits for specific uses. In such cases the value should be agreed upon by the interested parties.

[e] Or to be agreed between the interested parties.

[f] The iodine value and saponification value can also be obtained from the composition of fatty acids.

[g] Raw or refined linseed oil with an iodine value over 190 should be designated "High iodine value linseed oil". The Hanus method, sometimes used for this test, gives different results to the Wijs method; if it is used by agreement between the interested parties, prior agreement on specification limits is essential.

Table 1 *(continued)*

Characteristic	Requirement				Test method
	Raw linseed oil	Alkali-refined linseed oil	Acid-refined linseed oil	Boiled linseed oil	
Clarity	No sediment[c] at 65 °C	A slight turbidity is allowed; after shortly heating to 45 °C the turbidity shall disappear and the oil shall stay clear after cooling to 20 °C		—	Clause 6
Refractive index[a] n_D^{23}	1,478 0 to 1,483 0	1,478 0 to 1,483 0	1,478 0 to 1,483 0	—	ISO 5661 or ISO 6320
Water max. % (mass fraction)	0,20	0,10	0,10	0,30	ISO 8534
Acid value max. mg KOH/g	4	1[d]	9[e]	8[e]	ISO 2114 or ISO 660
Saponification value mg KOH/g	188 to 195	188 to 195	188 to 195	188 to 200	ISO 3681[f]
Iodine value, min. (Wijs method)[g]	175	175	175	—	ISO 3961[f]
Phosphoric acid test (PAT value) max. % (mass fraction)	0,25	—	—	—	Clause 7
Break	—	Non-visible	—	—	Clause 8
Composition of fatty acids	see Annex A	see Annex A	see Annex A	see Annex A	ISO 12966 (all parts)

a 23 °C is the standard temperature unless otherwise agreed: for example 20 °C, 25 °C, or 27 °C for tropical countries.

b If the acid value of neutral oil has been increased by the addition of fatty acids, then the requirement for colour after heating should be agreed upon between the interested parties, as the limits for neutral oil are not necessarily applicable.

c Stricter requirements may be agreed between the interested parties.

d Alkali-refined oil may have its acid value adjusted to other limits for specific uses. In such cases the value should be agreed upon by the interested parties.

e Or to be agreed between the interested parties.

f The iodine value and saponification value can also be obtained from the composition of fatty acids.

g Raw or refined linseed oil with an iodine value over 190 should be designated “High iodine value linseed oil”. The Hanus method, sometimes used for this test, gives different results to the Wijs method; if it is used by agreement between the interested parties, prior agreement on specification limits is essential.

5 Sampling

Take a representative sample of the oil in accordance with ISO 5555.

Prepare the sample in accordance with ISO 661.

6 Clarity

6.1 Raw oil

Heat a well-mixed test portion to 65 °C and examine it immediately for the presence of insoluble impurities.

6.2 Alkali-refined, acid-refined and boiled oil

Keep a well-mixed test portion at 15 °C to 20 °C for 24 h and then examine it for the presence of sediment and for other insoluble matter.

7 Phosphoric acid test (PAT value) (for raw linseed oil only)

7.1 Principle

Thorough mixing of a test portion with 85 % (mass fraction) orthophosphoric acid. Separation of the precipitated material by centrifuging and washing free of oil with acetone, drying and weighing. Reporting the percentage by mass as the PAT value.

7.2 Reagents

7.2.1 Orthophosphoric acid, 85 % (mass fraction), ρ = 1,7 g/ml.

7.2.2 Acetone.

7.2.3 Filter aid, of the diatomaceous type.

7.3 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

7.3.1 Sintered glass filter crucibles, of porosity grade P 16 (pore size index 10 µm to 16 µm in accordance with ISO 4793) and of capacity 30 ml.

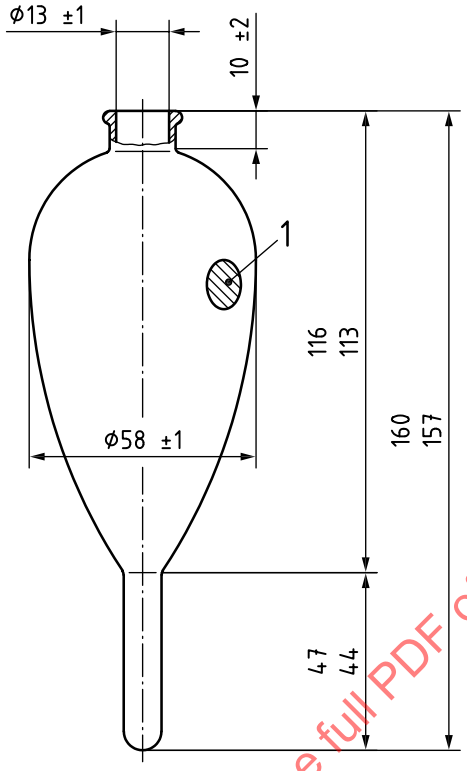
The crucibles shall be cleaned periodically with cleaning solution. It is desirable to test the filtration rate of each crucible with pure acetone and discard any that cannot be cleaned to give satisfactory filtration rates.

7.3.2 Agitator, consisting of a horizontal shaft suitably supported and fitted with clamps or a clamping device for holding the pear-shaped centrifuge tubes.

The tubes are held in such a manner that, when the shaft rotates, the tubes are tipped end over end, thus allowing the liquid content of the tube to mix as it flows from one end of the tube to the other. The shaft is rotated mechanically by any means which allows a frequency of (16 ± 2) min^{-1}.

7.3.3 Centrifuge tubes, of capacity 100 ml, pear-shaped as shown in Figure 1, fitted with a stopper.

Dimensions in millimetres



Key

1 sandblasted spot (for marking)

Figure 1 — Pear-shaped centrifuge tube

7.3.4 Centrifuge, capable of holding two or more tubes.

It should be possible to control the rotational frequency of the centrifuge so as to give a relative centrifugal acceleration of 500 g_n to 800 g_n at the tips of the tube (see Table 2), where g_n is the standard acceleration due to gravity.

Table 2 — Rotational frequencies applicable to centrifuges of various diameters of swing[a]

Diameter of swing mm	Rotational frequency corresponding to a relative centrifugal acceleration of 500 g_n min^{-1}	Rotational frequency corresponding to a relative centrifugal acceleration of 800 g_n min^{-1}
300	1 727	2 184
320	1 672	2 115
340	1 622	2 052
360	1 576	1 994

[a] The rotational frequency is calculated from the formula

$$n = 1\,346\sqrt{\frac{c}{d}}$$

where

c is the relative centrifugal acceleration, expressed as a multiple of the standard acceleration of free fall, g;

d is the diameter of swing, in millimetres;

n is the rotational frequency, expressed in revolutions per minute.

Table 2 *(continued)*

Diameter of swing mm	Rotational frequency corresponding to a relative centrifugal acceleration of 500 g_n min^{-1}	Rotational frequency corresponding to a relative centrifugal acceleration of 800 g_n min^{-1}
380	1 534	1 941
400	1 496	1 892
420	1 460	1 846
440	1 426	1 804
460	1 395	1 764
480	1 365	1 727
500	1 338	1 692

[a] The rotational frequency is calculated from the formula

$$n = 1\,346\sqrt{\frac{c}{d}}$$

where

c is the relative centrifugal acceleration, expressed as a multiple of the standard acceleration of free fall, g;

d is the diameter of swing, in millimetres;

n is the rotational frequency, expressed in revolutions per minute.

7.3.5 **Pipette**, of capacity 1 ml, graduated in 0,01 ml, complying with the requirements of ISO 835.

7.3.6 **Desiccator**, containing an efficient desiccant.

Anhydrous calcium sulfate, anhydrous calcium chloride and silica gel are satisfactory.

7.4 Preparation of the sample

Allow the sample to reach room temperature (23 ± 2) °C and then shake or mix thoroughly to ensure that all the sediment has been completely dispersed. If the volatile matter content of the sample is greater than 0,25 % (mass fraction), dry the sample by heating it at 100 °C under vacuum or by bubbling dry carbon dioxide or nitrogen at (100 ± 5) °C through it for 30 min. Cool the sample to (23 ± 2) °C.

7.5 Procedure

7.5.1 Weigh into a centrifuge tube (7.3.3), (50 ± 0,01) g of the prepared sample and then add (0,50 ± 0,05) ml of orthophosphoric acid (7.2.1) using the pipette (7.3.5).

Stopper the tube and tilt it so that the acid runs out of the tip and into the oil. Shake the tube vigorously for a few seconds. Repeat the tilting and shaking twice more.

7.5.2 Place the tube on the agitator (7.3.2) and mix for 5 min at such a rotational frequency that the whole of the acid disperses throughout the oil and the tip of the tube empties of oil at each revolution (a rotational frequency of 16 min^{-1} is adequate). Adjust the rotational frequency of the agitator so that intimate mixing without separation takes place. Mix at this rate for 25 min.

Place the tube in the centrifuge (7.3.4) and spin it for 1 h with a relative centrifugal acceleration of at least 500 g_n at the tip or until the deposit stays in position as a compact mass when the tube is inverted. The temperature should be maintained at approximately (23 ± 2) °C. This may be done by admitting air to the centrifuge casing.

7.5.3 Decant or siphon the supernatant oil as completely as possible into a clean centrifuge tube and allow time for it to drain. If the sediment layer is liquid, take care to remove the oil without disturbing the layer. To this end a modified siphon can be used to advantage.

Add 25 ml of acetone (7.2.2) to the precipitate in the first tube and mix until any gummy material is dispersed. Use a wire to loosen such material from the tip of the tube if necessary, then make up the volume to 100 ml with acetone and shake the tube.

7.5.4 Prepare the sintered glass crucibles (7.3.1) by adding 0,3 g to 0,6 g of the filter aid (7.2.3) to the empty crucibles. With experience, this quantity can be measured on the tip of a spatula. Mix the filter aid into a slurry with approximately 15 ml of acetone. Remove the acetone by applying a vacuum to the filter. Dry the crucibles in an oven at (100 ± 5) °C for 1 h. Allow to cool for 1 h in the desiccator (7.3.6) and weigh to the nearest 0,1 mg. Check that the mass is constant. Store the prepared crucibles in the desiccator until they are to be used.

7.5.5 Filter the acetone dispersion of the precipitate through a prepared sintered glass crucible. Use a moderate vacuum and always maintain some acetone in the crucible.

Thoroughly wash the centrifuge tube and the precipitate in the crucible with four 15 ml portions of acetone using a wash-bottle.

NOTE Since oil tends to creep up the sides of the crucible, care should be exercised.

7.5.6 After washing, continue applying suction until the crucible is free from acetone, dry it at (100 ± 5) °C, allow it to cool to room temperature in the desiccator and weigh it to the nearest 0,1 mg.

Repeat the whole procedure for the supernatant oil obtained after centrifuging (see 7.5.3) in the same way as for the original oil. Weigh any additional sediment obtained as before.

7.6 Expression of results

The PAT value, expressed as a percentage mass fraction, is given by Formula (1):

$$\text{PAT} = 2\left(m_1 + m_2\right) \tag{1}$$

where

m_1 is the mass, in grams, of sediment from 50 g of the original oil on first phosphoric acid treatment;

m_2 is the mass, in grams, of sediment from supernatant oil on second phosphoric acid treatment.

Report the results to two decimal places.

7.7 Precision

7.7.1 Repeatability

The value below which the absolute difference between two single results on identical test material, obtained by one operator within a short time interval with the same apparatus under constant operating conditions, can be expected to lie with a 95 % probability, is 0,03 % (mass fraction).

7.7.2 Reproducibility

No data are currently available.

8 Assessment of break in alkali-refined linseed oils

8.1 General

This method describes a test to assess the presence or absence of break in alkali-refined linseed oils. It is applicable to refined oils that have not been substantially polymerized, oxidized or chemically modified.

8.2 Principle

Treatment of a test portion with hydrochloric acid, heating to 290 °C, allowing to cool, and examination with transmitted light for insoluble break.

8.3 Reagent

8.3.1 Hydrochloric acid, $\rho \approx 1{,}19$ g/ml.

8.4 Apparatus

8.4.1 Beakers, of capacity 150 ml, squat form.

8.4.2 Thermometer, having a range of approximately –6 °C to +400 °C, with subdivisions of 2 °C, and 25 mm immersion.

8.4.3 Heating equipment, gas or electric, capable of heating the test portion to 290 °C in 3 min to 3,5 min.

8.5 Procedure

Pour 65 ml to 75 ml of well-mixed sample into the beaker (8.4.1). Add 6 to 8 drops of hydrochloric acid (8.3.1) and stir thoroughly with the thermometer (8.4.2). Suspend the thermometer in the centre of the mixture so that the bulb is completely immersed in the liquid but not touching the bottom of the beaker. Using the heating equipment (8.4.3), apply heat so that the test portion reaches (290 ± 3) °C in 3 min to 3,5 min.

The mixture has a tendency to bump. It is therefore advisable to hold the beaker firmly on the heating equipment with a clamp until the temperature has reached 160 °C.

After a temperature of (290 ± 3) °C has been reached, remove the beaker from the heating equipment, allow to cool and observe against transmitted light.

8.6 Expression of results

Report observations as either:

a) non-visible: oil is brilliant or very slightly hazy when viewed as stated;

b) poor: oil is quite hazy, signifying it is not completely refined; or

c) breaks: oil forms a suspension of insoluble matter (“break”) which can coagulate on standing.

9 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 150;