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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 150:2006(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 150 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 150:1980), which has been technically revised.

The main changes are:

— The requirements for turbidity (clarity) have been changed.

— The maximum acid value for alkali-refined linseed oil has been changed to 1,0 mg KOH/g.

— The determination of unsaponifiable-matter content, foots, colophony (rosin), fish oil and mineral acid have been deleted because they are no longer necessary.

— The determination of volatile-matter content has been replaced by the determination of water content.

— The determination of ash has been deleted because it is not required very often.

— Standard values for the composition of fatty acids of raw linseed oil have been added (see Annex A).

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

1 Scope

This International Standard 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 referenced documents are indispensable for the application 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 835-1, *Laboratory glassware — Graduated pipettes — Part 1: General requirements*

ISO 1517:1973, *Paints and varnishes — Surface-drying test — Ballotini method*

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: Pyknometer 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 4630-1, *Clear liquids — Estimation of colour by the Gardner colour scale — Part 1: Visual method*

ISO 4630-2, *Clear liquids — Estimation of colour by the Gardner colour scale — Part 2: Spectrophotometric method*

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

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

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

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

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

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 air or oxygen through the oil

3.5

break

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

NOTE 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.

5 Sampling

Take a representative sample of the oil in accordance with ISO 15528.

6 Density

Determine the density at 23 °C or another agreed temperature by the method specified in ISO 2811-1. (See Footnote “a” to Table 1.)

7 Refractive index

Determine the refractive index at 23 °C or another agreed temperature by the method specified in ISO 5661. (See Footnote “a” to Table 1)

8 Clarity

8.1 Raw oil

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

8.2 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.

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	Clause 6 and ISO 2811-1
Colour[b], max. (Gardner)	13	4	6	To be agreed between purchaser and vendor	ISO 4630-1 ISO 4630-2
Colour after heating[b], max. (Gardner)	—	—[c]	—	—	—
Clarity	No sediment[d] at 65 °C	Slight turbidity is allowed. After heating briefly to 45 °C the turbidity shall disappear and the oil shall stay clear after cooling to 20 °C.		—	Clause 8
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	—	Clause 7 and ISO 5661
Water, max., % (by mass)	0,20	0,10	0,10	0,30	ISO 8534
Acid value, max., mg KOH/g	4	1[f]	9[e]	8[e]	ISO 2114
Saponification value, mg KOH/g	188 to 195	188 to 195	188 to 195	188 to 200	ISO 3681[g]
Iodine value, min. (Wijs method)[h]	175	175	175	—	ISO 3961[g]
Phosphoric acid test (PAT) value, max., % (by mass)	0,25	—	—	—	Clause 9
Drying time, max.	—	—	—	24 h at 15 °C to 20 °C or 15 h at 25 °C to 30 °C	ISO 1517 and Clause 10
Break	—	Non-visible	—	—	Clause 11

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

[b] By agreement between the interested parties, the Lovibond colour system may be substituted for the Gardner with the following limits being recommended:

Raw: 70Y 6R (25 mm cell)

Alkali-refined: 15Y 1,5R (25 mm cell)

Alkali-refined, heated: 20Y 2,0R (133 mm cell)

Acid-refined: 20Y 1,5R (25 mm cell)

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

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

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

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

[g] The iodine value and saponification value can also be obtained from the fatty acid contents.

[h] 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.

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

9.1 Principle

Mix a test portion thoroughly with 85 % (by mass) orthophosphoric acid. Separate the precipitated material by centrifuging, wash the precipitate free of oil with acetone and then dry and weigh. Report the percentage of precipitated material by mass as the PAT value.

9.2 Reagents and materials

9.2.1 Orthophosphoric acid, 85 % (by mass), $\rho = 1{,}7$ g/ml.

9.2.2 Acetone.

9.2.3 Filter aid, of the diatomaceous type.

9.3 Apparatus

Ordinary laboratory apparatus, together with the following:

9.3.1 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.

9.3.2 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 will give a frequency of (16 ± 2) r/min.

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

9.3.4 Centrifuge, capable of holding two or more tubes.

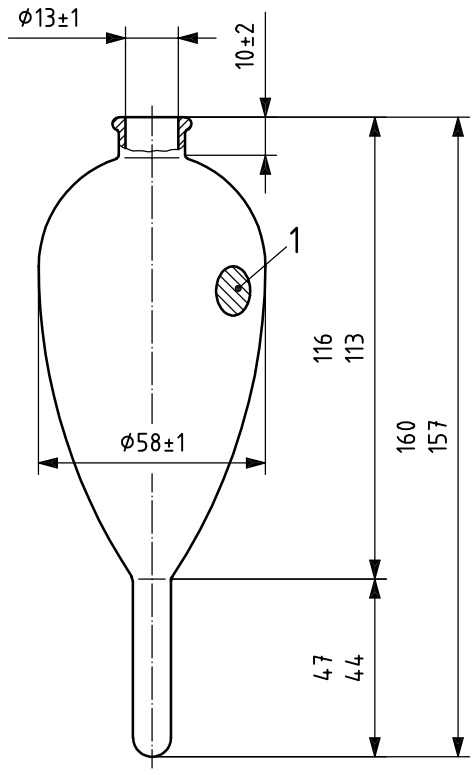
It should be possible to control the rotational frequency of the centrifuge so as to give a relative centrifugal acceleration of 500g to 800g at the tips of the tube (see Table 2), where g is the standard acceleration due to gravity.

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

9.3.6 Desiccator, containing an efficient desiccant.

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

Dimensions in millimetres



Key

1 sandblasted spot (for marking)

Figure 1 — Pear-shaped centrifuge tube

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** $500g$ r/min	**Rotational frequency corresponding to a relative centrifugal acceleration of** $800g$ r/min
300	1 727	2 184
320	1 672	2 115
340	1 622	2 052
360	1 576	1 994
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.

9.4 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 % (by mass), 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.

9.5 Procedure

9.5.1 Weigh into a centrifuge tube (9.3.3), (50 ± 0,01) g of the prepared sample and then add (0,5 ± 0,05) ml of orthophosphoric acid (9.2.1) using the pipette (9.3.5).

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.

9.5.2 Place the tube on the agitator (9.3.2) 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 r/min 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 (9.3.4) and spin it for 1 h with a relative centrifugal acceleration of at least 500g at the tip or until the deposit stays in position as a compact mass when the tube is inverted. The temperature shall be maintained at approximately (23 ± 2) °C. This may be done by admitting air to the centrifuge casing.

9.5.3 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. A modified siphon is suitable for this operation.

Add 25 ml of acetone (9.2.2) 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.

9.5.4 Prepare the sintered-glass crucibles (9.3.1) by adding 0,3 g to 0,6 g of the filter aid (9.2.3) 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 (9.3.6) 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.

9.5.5 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.

Since oil tends to creep up the sides of the crucible, care is necessary when doing this.

9.5.6 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 9.5.3) in the same way as for the original oil. Weigh any additional sediment obtained as before.

9.6 Expression of results

9.6.1 Calculation

The PAT value, expressed as a percentage by mass, is given by the formula

$$2(m_1 + m_2)$$

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 the supernatant oil on second phosphoric acid treatment.

Report the result to two decimal places.

9.6.2 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, may be expected to lie with a 95 % probability is 0,03 % (by mass).

10 Drying time (for boiled linseed oil only)

Determine the time by the method specified in ISO 1517, incorporating the following modifications appropriate for drying oils:

a) Substrate (see 6.1 of ISO 1517:1973)

The test panel shall be a polished glass plate that has been cleaned to prevent the oils to be tested from crawling. This may be carried out by washing the panel thoroughly with a suitable solvent such as xylene, followed by complete removal of solvent, or by washing thoroughly in an aqueous non-ionic detergent solution, followed by thoroughly rinsing with distilled water.

b) Coating the panel (see 6.2 of ISO 1517:1973)

Coat the test panel by spreading oil evenly over the whole surface with a brush or finger. Excess oil will drain off when the panel is placed in the vertical position.

11 Assessment of break in alkali-refined linseed oils

11.1 General

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

11.2 Principle

A test portion is treated with hydrochloric acid, heated to 290 °C, allowed to cool and then examined against transmitted light for insoluble break.

11.3 Reagent

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

11.4 Apparatus

11.4.1 Beaker, of capacity 150 ml, squat form.

11.4.2 Thermometer, having a range of approximately $-6\ ^{\circ}C$ to $+400\ ^{\circ}C$, with subdivisions of $2\ ^{\circ}C$ and an immersion depth of 25 mm.

11.4.3 Heating equipment, gas or electric, capable of heating the test portion to $290\ ^{\circ}C$ in 3 min to 3,5 min.

11.5 Procedure

Pour 65 ml to 75 ml of well-mixed sample into the beaker (11.4.1). Add 6 to 8 drops of hydrochloric acid (11.3.1) and stir thoroughly with the thermometer (11.4.2). 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 (11.4.3), apply heat so that the test portion reaches $(290 \pm 3)\ ^{\circ}C$ in 3 min to 3,5 min.

NOTE 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 $160\ ^{\circ}C$.

After $(290 \pm 3)\ ^{\circ}C$ has been reached, remove the beaker from the heating equipment, allow to cool and observe against transmitted light.

11.6 Expression of results

Report observations as one of the following:

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

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

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

12 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard (ISO 150);

b) the type and identification of the product tested;

c) any deviation, by agreement or otherwise, from the procedures specified;

d) the results of the tests, and whether or not the product complies with the relevant specification limits;

e) the dates of the tests.

Annex A
(informative)

Standard values for the composition of fatty acids of raw linseed oil

Standard values for the composition of fatty acids of raw linseed oil as obtained by gas chromatographic analysis are shown in Table A.1.

Table A.1 — Standard values for the contents of fatty acids in raw linseed oil

Fatty acid		Content % (by mass)
Myristic acid	C14:0	0 to 0,4
Palmitic acid	C16:0	4,5 to 7,1
Palmitoleic acid	C16:1	0,1 to 0,3
Stearic acid	C18:0	2,3 to 5,8
Oleic acid	C18:1 *cis*	17 to 23,5
Elaidic acid	C18:1 *trans*	0,6 to 1
Linoleic acid	C18:2	13,8 to 17,5
Linolenic acid	C18:3	50 to 60
Arachidic acid	C20:0	0,2 to 0,6

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