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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

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<u>ISO 150:2006</u> https://standards.iteh.ai/catalog/standards/sist/c21a7cdf-6498-44b9-8055-cabbd0da27b2/iso-150-2006



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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:

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- The requirements for turbidity (clarity) have been changed
- The maximum acid value for alkali-refined inseed oil has been changed to 1,0 mg KOH/q.
- 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- The determination of volatile-matter content has been replaced by the determination of water content.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 https://standards.iteh.ai/catalog/standards/sist/c21a7cdf-6498-44b9-8055-

ISO 3681, Binders for paints and varnishes and 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

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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.

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5 Sampling

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Take a representative sample of the oil in accordance with 180 15528.

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6 Density

Determine the density at 23 $^{\circ}$ 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 $^{\circ}$ 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 $^{\circ}$ C to 20 $^{\circ}$ 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

	Requirement				
Characteristic	Raw linseed oil	Alkali-refined linseed oil	Acid-refined linseed oil	Boiled linseed oil	Test method
Density ^a , $ ho_{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 , $n_{\rm D}^{\rm 23}$	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
lodine value, min. (Wijs method) ^h	175	175 standards	175 itoh ai)	_	ISO 3961 ^g
Phosphoric acid test (PAT) value, max., % (by mass)	0,25			I	Clause 9
Drying time, max.	https://standards.ite —	h.ai/catalog/standards/s cabbd0da27b2/iso-		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.

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)

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

^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.

 $^{^{9}}$ 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 μ m to 16 μ m 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.

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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.

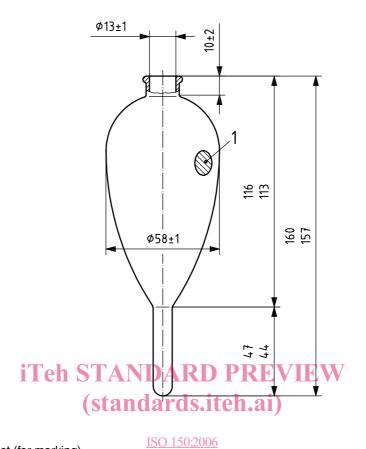
- 9.3.3 Centrifuge tubes, of capacity 100 ml, pear-shaped as shown in Figure 1, fitted with a stopper.
- 9.3.4 Centrifuge, capable of holding two or more tubes.

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

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Figure Pear-shaped centrifuge tube

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

Diameter of swing	Rotational frequency corresponding to a relative centrifugal acceleration of $500g$	Rotational frequency corresponding to a relative centrifugal acceleration of $800g$
mm	r/min	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

$$n = 1346\sqrt{\frac{c}{d}}$$

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.