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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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Foreword

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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).

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This document was prepared by Technical Committee ISO/TC 35, Paints and varnishes. 16-4ed5-81ab-55c5be7a176c/iso-

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- sampling in accordance with ISO 15528 has been replaced by sampling in accordance with ISO 5555: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

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

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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

Characte	ristic		Requir	rement		Test
		Raw linseed oil	Alkali-refined linseed oil	Acid-refined linseed oil	Boiled linseed oil	method
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		_	b	_	_	_

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max.					
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	9e	<u>8</u> 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)	175	175	175	-	_ISO 3961 <u>f</u> .
Phosphoric acid test (PAT value) max. % (mass, fraction)	0,25	(STAN	(DARL dards.)	PRE teh.ai)	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)

- 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.
- $^{\rm f}\,$ The iodine value and saponification value can also be obtained from the composition of fatty acids.
- 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 $65\,^{\circ}\text{C}$ 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 $15\,^\circ\text{C}$ to $20\,^\circ\text{C}$ for $24\,^\text{h}$ 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