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# International Standard



# 150

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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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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**Descriptors** : linseed oil, paints, varnishes, specifications, physical properties, chemical properties, tests, density measurement, refractivity, chemical analysis, impurities.

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 150 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in July 1978.

It has been approved by the member bodies of the following countries :

|                      |                |                       |
|----------------------|----------------|-----------------------|
| Australia            | Italy          | South Africa, Rep. of |
| Brazil               | Kenya          | Sweden                |
| Czechoslovakia       | Korea, Rep. of | Switzerland           |
| Egypt, Arab. Rep. of | Mexico         | United Kingdom        |
| France               | Netherlands    | USSR                  |
| Germany, F. R.       | Norway         | Yugoslavia            |
| India                | Poland         |                       |
| Israel               | Romania        |                       |

The member body of the following country expressed disapproval of the document on technical grounds :

Canada

This International Standard cancels and replaces ISO Recommendation R 150-1960 of which it constitutes a technical revision.

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

## 1 Scope and field of application

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

## 2 References

ISO 835, *Laboratory glassware — Graduated pipettes*.<sup>1)</sup>

ISO 842, *Raw materials for paints and varnishes — Sampling*.

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

ISO 1773, *Laboratory glassware — Boiling flasks (narrow-necked)*.

ISO 3681, *Paint media — Determination of saponification value — Titrimetric method*.

ISO 3682, *Paint media — Determination of acid value — Titrimetric method*.

ISO 3838, *Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pycnometer method*.<sup>2)</sup>

ISO 3961, *Fats and oils — Determination of iodine value*.<sup>2)</sup>

ISO 4630, *Binders for paints and varnishes — Estimation of colour of clear liquids by the Gardner colour scale*.<sup>2)</sup>

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*.<sup>2)</sup>

ISO 5661, *Petroleum products — Hydrocarbon liquids — Determination of refractive index, refractive dispersion and specific optical dispersion*.<sup>2)</sup>

## 3 Definitions

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

**3.2 acid-refined linseed oil** : The oil obtained by refining raw linseed oil (3.1) with acid.

**3.3 alkali-refined linseed oil** : The oil obtained by refining raw linseed oil (3.1) with sodium hydroxide or other alkali solution.

**3.4 boiled linseed oil** : The oil obtained by incorporating driers in raw linseed oil (3.1) or refined linseed oil (3.2 and 3.3) and heating either alone or while blowing in air or oxygen.

**3.5 break** : The separation of an (insoluble) mucilaginous product which occurs when certain unrefined vegetable oils are heated. 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 shown in table 1.

## 5 Sampling

Take a representative sample of the oil in accordance with ISO 842.

1) At present at the stage of draft. (Revision of ISO/R 835).

2) At present at the stage of draft.

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 <sup>1)</sup> $\rho_{23}$ 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 3838  |
| Colour <sup>2)</sup> max. (Gardner)                         | 13                                 | 6  | 6  | To be agreed between purchaser and vendor  | ISO 4630               |
| Colour after heating <sup>2)</sup> , max. (Gardner)         | —                                  | 4 <sup>3)</sup>                          | —  | —  | Clause 18              |
| Clarity   | No sediment <sup>4)</sup> at 65 °C | No sediment <sup>4)</sup> at 15 to 20 °C | No sediment <sup>4)</sup> at 15 to 20 °C | No sediment <sup>4)</sup> at 15 to 20 °C   | Clause 8               |
| Refractive index <sup>1)</sup> $n_D^{23}$                   | 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  |
| Volatile matter, max. % (m/m)                               | 0,20                               | 0,10                                     | 0,10                                     | 0,30                                       | Clause 9               |
| Ash, max. <sup>5)</sup> % (m/m)                             | 0,15                               | 0,02                                     | 0,02                                     | 0,50                                       | Clause 10              |
| Acid value, max.  | 4                                  | 0,50 <sup>6)</sup>                       | 9 <sup>5)</sup>                          | 8 <sup>5)</sup>                            | ISO 3682               |
| Saponification value  | 188 to 195                         | 188 to 195                               | 188 to 195                               | 188 to 200                                 | ISO 3681               |
| Unsaponifiable matter, max. % (m/m)                         | 1,5                                | 1,5                                      | 1,5                                      | 2,0  | Annex                  |
| Iodine value, min. (Wijs method) <sup>7)</sup>              | 175                                | 175                                      | 175                                      | —  | ISO 3961               |
| Foots <sup>8)</sup> , max. % (V/V)                          | 1,0                                | —  | Nil                                      | —  | Clause 11              |
| Phosphoric acid test (PAT value) <sup>8)</sup> max. % (m/m) | 0,25                               | —  | —  | —  | Clause 12              |
| Drying time, max.   | —                                  | —  | —  | 24 h at 15 to 20 °C or 15 h at 25 to 30 °C | ISO 1517 and clause 13 |
| Colophony (rosin)   | Negative result                    | Negative result                          | Negative result                          | Negative result                            | Clause 14              |
| Fish oil  | Negative result                    | Negative result                          | Negative result                          | Negative result                            | Clause 15              |
| Mineral acid  | —                                  | —  | Negative result                          | —  | Clause 16              |
| Break   | —                                  | Non-visible                              | —  | —  | Clause 17              |

1) 23 °C is the standard temperature unless otherwise agreed : for example 20 °C, 25 °C, or 27 °C for tropical countries.

2) 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)  |

3) 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.

4) Stricter requirements may be agreed between the interested parties. See also clause 11, "Foots".

5) Or to be agreed between the interested parties.

6) 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.

7) Raw or refined linseed oil with an iodine value over 190 should be designed "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.

8) By agreement between the interested parties one of these tests may be omitted.

## Methods of test

During the analyses, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

### 6 Density

Determine the density at 23 °C or another agreed temperature by the methods specified in ISO 3838. (See footnote 1 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 1 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 to 20 °C for 24 h and then examine it for the presence of sediment and for other insoluble matter.

### 9 Volatile matter

#### 9.1 Procedure

Weigh, to the nearest 1 mg, about 10 g of the sample into a tared 250 ml conical flask complying with the requirements of ISO 1773. Heat the flask for 30 min in a heating bath at 105 to 110 °C, blowing a dry inert gas over the surface of the oil at a rate of 10 l/h. Remove the flask from the bath, cool the flask to room temperature, clean it and weigh it to the nearest 1 mg.

#### 9.2 Expression of results

The volatile matter, expressed as a percentage by mass, is given by the formula

$$\frac{m_0 - m_1}{m_0} \times 100$$

where

$m_0$  is the mass, in grams, of the original test portion;

$m_1$  is the mass, in grams, of the test portion after heating.

### 10 Ash

#### 10.1 Apparatus

**10.1.1 Crucible**, of porcelain or vitreous silica (see note), of capacity 50 ml.

NOTE — Platinum is not recommended for boiled oils, as lead from driers may alloy with the platinum.

**10.1.2 Electric muffle furnace**, capable of being maintained at  $600 \pm 50$  °C.

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

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

#### 10.2 Procedure

Heat the crucible (10.1.1) in the furnace (10.1.2) to  $600 \pm 50$  °C. Cool slightly, place in the desiccator (10.1.3) for 1 h, and weigh to the nearest 0,1 mg. Weigh, to the nearest 10 mg, about 10 g of the well-mixed sample into the crucible. Support the filled crucible on a pipe-clay triangle. Heat gently by moving a flame on the bottom and sides of the crucible until the oil ignites.

Reduce the size of the flame until the heat is just sufficient to keep the test portion burning. Continue heating the test portion to a black char and transfer to the furnace. Heat at  $600 \pm 50$  °C for 1 h. Remove from the furnace, cool slightly, place in the desiccator and cool to room temperature; weigh to the nearest 0,1 mg and repeat the above operations until the difference between successive weighings is no more than 1 mg.

#### 10.3 Expression of results

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

$$\frac{m_1}{m_0} \times 100$$

where

$m_0$  is the mass, in grams, of the test portion;

$m_1$  is the mass, in grams, of the residue.

**11 Foots** (for raw and acid-refined linseed oil)

**11.1 Definition**

For the purposes of this International Standard, the following definition applies :

**foots** : Material which has settled from the oil after 4 days under the specified test conditions.

NOTE — “Foots” is a term that originally was used to describe those impurities that precipitate from raw linseed oil during storage and subsequently settle to the bottom or “foot” of a storage tank.

**11.2 Apparatus**

**Sediment tube**, pear-shaped or cone-shaped. (See figures 1 and 2.)

**11.3 Procedure**

Place 100 ml of the oil in the sediment tube (11.2) and allow it to stand there in a vertical position for 4 days (96 h) at a temperature between 15 and 20 °C. During this time, the tube should remain undisturbed. At the end of the 4 days (96 h), read the volume of sediment or foots at the bottom of the tube.

**11.4 Expression of results**

Report the value of foots as a percentage by volume.

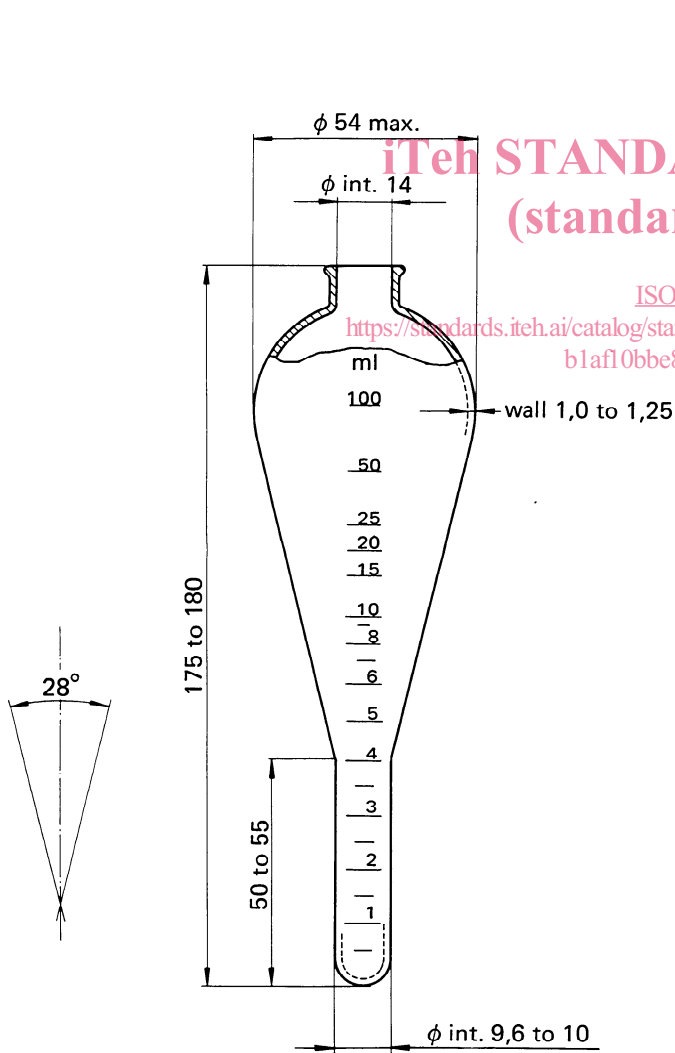


Figure 1 — Pear-shaped sediment tube

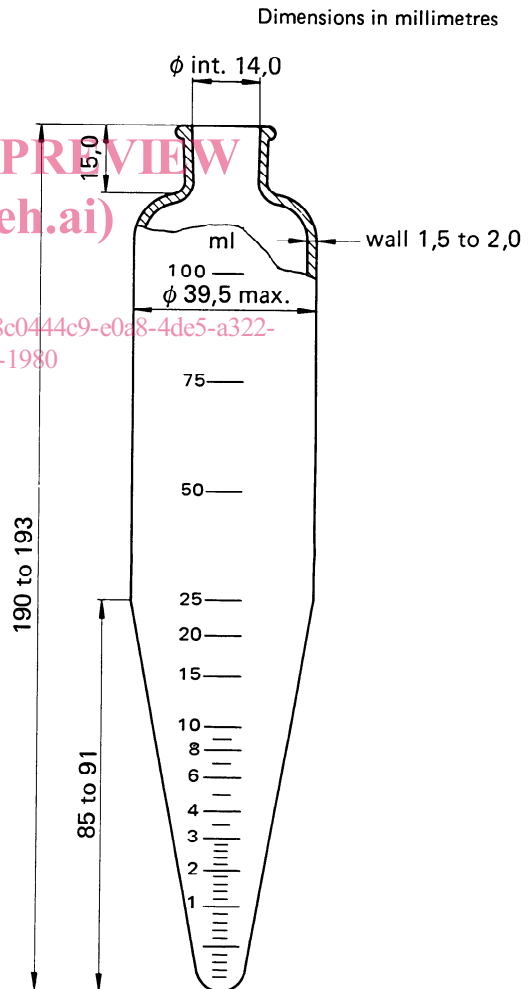


Figure 2 — Cone-shaped sediment tube

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

### 12.1 Principle

Thorough mixing of a test portion with 85 % (*m/m*) ortho-phosphoric 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.

### 12.2 Reagents

12.2.1 Orthophosphoric acid, 85 % (*m/m*),  $\rho$  1,7 g/ml.

12.2.2 Acetone.

12.2.3 Filter aid, of the diatomaceous type.

### 12.3 Apparatus

Ordinary laboratory apparatus and

12.3.1 Sintered glass filter crucibles, of porosity grade P 16 (pore size index 10 to 16  $\mu\text{m}$  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.

12.3.2 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 be other. The shaft is rotated mechanically by any means which allows the choice of two fixed rotational frequencies, a low frequency  $16 \pm 2$  r/min and a higher frequency of  $32 \pm 2$  r/min.

12.3.3 Centrifuge tubes, of capacity 100 ml, pear-shaped as shown in figure 3, fitted with a stopper.

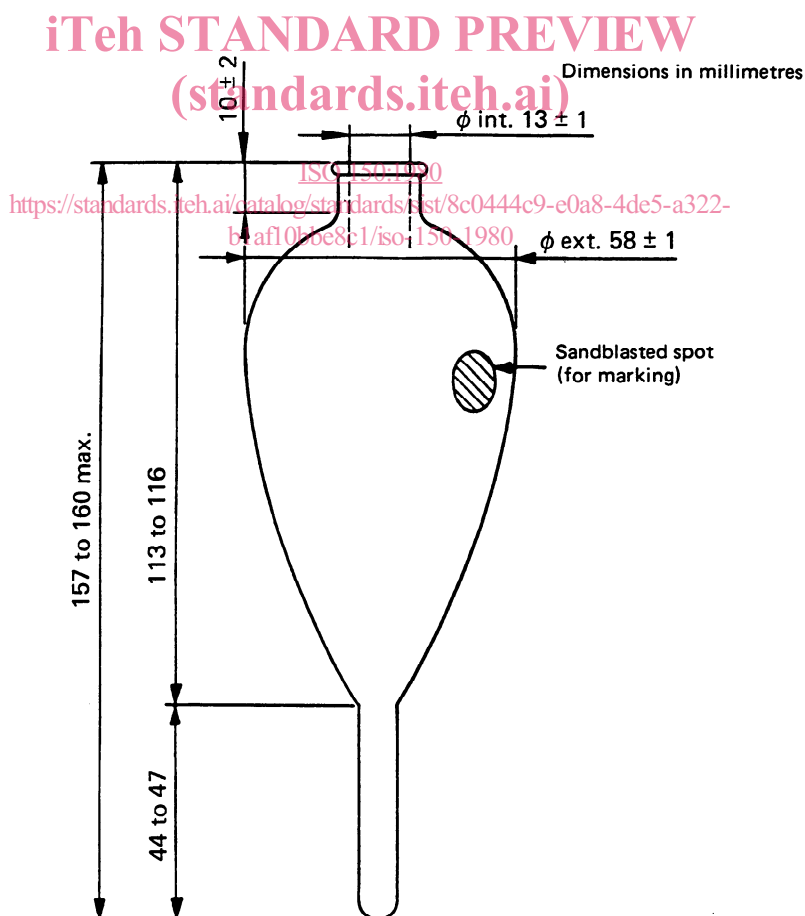


Figure 3 — Pear-shaped centrifuge tube

### 12.3.4 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<sup>1)</sup>**

| Diameter of swing | Rotational frequency corresponding to a relative centrifugal acceleration of $500 g_n$ | Rotational frequency corresponding to a relative centrifugal acceleration of $800 g_n$ |
|-------------------|--|--|
| 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  |

1) 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,  $g_n$ ;

$d$  is the diameter of swing, in millimetres;

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

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

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

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

## 12.4 Preparation of the sample

Allow the sample to reach room temperature ( $23 \pm 2$  °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 % ( $m/m$ ), dry the sample by heating it at 100 °C under vacuum or by bubbling dry carbon dioxide or nitrogen at  $100 \pm 5$  °C through it for 30 min. Cool the sample to  $23 \pm 2$  °C.

## 12.5 Procedure

**12.5.1** Weigh into a centrifuge tube (12.3.3),  $50 \pm 0,01$  g of

the prepared sample and then add  $0,5 \pm 0,05$  ml of the orthophosphoric acid (12.2.1) using the pipette (12.3.5).

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.

**12.5.2** Place the tube on the agitator (12.3.2) 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 (32 r/min is satisfactory). Mix at this rate for 25 min.

Place the tube in the centrifuge (12.3.4) 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  $23 \pm 2$  °C. This may be done by admitting air to the centrifuge casing.

**12.5.3** 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, extra care must be taken to remove the oil without disturbing this layer. To this end a modified siphon can be used to advantage.

Add 25 ml of the acetone (12.2.2) 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.

**12.5.4** Prepare the sintered glass crucibles (12.3.1) by adding 0,3 to 0,6 g of the filter aid (12.2.3) 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 the acetone. Remove the acetone by applying a vacuum to the filter. Dry the crucibles in an oven at  $100 \pm 5$  °C for 1 h. Allow to cool for 1 h in the desiccator (12.3.6) 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.

**12.5.5** 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 the acetone using a wash-bottle.

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

**12.5.6** After washing, continue applying suction until the crucible is free from acetone, dry it at  $100 \pm 5$  °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 12.5.3) in the same way as for the original oil. Weigh any additional sediment obtained as before.



## 12.6 Expression of results

### 12.6.1 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 supernatant oil on second phosphoric acid treatment.

Report the results to two decimal places.

### 12.6.2 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 % ( $m/m$ ).

## 13 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 sub-clause 6.1 of ISO 1517)

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 thorough rinsing with distilled water.

- b) Coating the panel (see sub-clause 6.2 of ISO 1517)

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.

## 14 Liebermann-Storch-Morawski test for presence of colophony

Mix 3 to 5 drops of the sample with 3 ml of acetic anhydride. Add 1 drop of sulphuric acid,  $\rho = 1,54$  g/ml, 65 % ( $m/m$ ). The reaction for colophony is positive if a fugitive violet colour appears which, at the moment of its maximum intensity, is stronger than the colour of potassium permanganate solution,  $c(\text{KMnO}_4) = 0,0002$  mol/l.

## 15 Test for presence of fish oil

### 15.1 Principle

Saponification of a test portion and liberation of the fatty acids.

Treatment of the fatty acids with bromine. If fish oil is present, the resulting bromides are insoluble in an acetic acid/chloroform mixture, whereas bromides of vegetable oils are soluble.

### 15.2 Reagents

15.2.1 Acetic acid, glacial.

15.2.2 Nitric acid, 252 g/l solution.

15.2.3 Hydrochloric acid, 37 g/l solution.

15.2.4 Potassium hydroxide, 56 g/l ethanolic solution.

15.2.5 Glacial acetic acid/chloroform, mixture of equal volumes.

15.2.6 Bromine.

15.2.7 Light petroleum, boiling range 40 to 60 °C.

15.2.8 Diethyl ether,  $\rho = 0,712$  to 0,716 g/ml, yielding a non-volatile residue at 80 °C of not more than 0,001 % ( $m/m$ ).

### 15.3 Procedure

Saponify approximately 5 g of the sample in a 250 ml boiling flask, complying with the requirements of ISO 1773, using 50 ml of the potassium hydroxide solution (15.2.4) and boiling under reflux for 1 h, with swirling at intervals.

Remove the flask from the condenser and evaporate to a syrupy paste on a water or steam bath.

To ensure removal of ethanol, add 10 ml of water and again evaporate, almost to dryness.

Dissolve the soap in about 100 ml of boiling water and transfer to a 500 ml separating funnel. Add an excess of the hydrochloric acid solution (15.2.3) and shake vigorously to decompose the soap. Cool and add about 200 ml of the light petroleum (15.2.7). Shake well and allow to stand for several hours. Run off the acid layer. Filter the light petroleum layer through a fast filtering, fat-free paper, pouring the liquid from the top of the separating funnel. Wash the filtered solution with 10 ml of the nitric acid solution (15.2.2). Allow to settle, run off the acid layer and wash several times with water. Repeat the acid and subsequent water washes until no trace of metal is present. Evaporate the light petroleum and carefully dry the residual fatty acids.

Take 1,5 ml of the residual fatty acids and dissolve in 40 ml of the diethyl ether (15.2.8) and 5 ml of the glacial acetic acid