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ISO RECOMMENDATION
R 276

LINSEED STAND OILS

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BRIEF HISTORY

The ISO Recommendation R 276, *Linseed Stand Oils*, was drawn up by Technical Committee ISO/TC 35, *Raw Materials for Paints, Varnishes and Similar Products*, the Secretariat of which is held by the Nederlands Normalisatie-instituut (NNI).

Work on this question by the Technical Committee began in 1950 and led in 1959 to the adoption of a Draft ISO Recommendation.

In November 1959, this Draft ISO Recommendation (No. 328) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

Austria	Greece	New Zealand
Belgium	India	Portugal
Burma	Ireland	Spain
Chile	Israel	Sweden
Czechoslovakia	Italy	United Kingdom
Denmark	Japan	U.S.A.
Germany	Netherlands	U.S.S.R.

One Member Body opposed the approval of the Draft:

France

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided in November 1962, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 276 November 1962

LINSEED STAND OILS

1. SCOPE

This ISO Recommendation defines the important requirements of linseed stand oils and the methods of test for these requirements.

2. DEFINITION

Linseed stand oils are polymerized linseed oils obtained only by heat from linseed oil and conforming to the requirements given below.

NOTE. — Certain of these materials are also referred to as "lithographic varnishes".

3. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

The linseed stand oils should have the following characteristics:

Property	Stand oil 1 extra low	Stand oil 2 low	Stand oil 3 medium	Stand oil 4 high	Stand oil 5 extra high	Clause describing test method
Viscosity,* poises at 20 °C (at 25 °C)	max. 10 (8)	10 to 40 (8 to 30)	40 to 80 (30 to 60)	80 to 160 (60 to 110)	min. 160 (110)	5.1
Ash, max. %	0.10	0.10	0.10	0.10	0.10	5.2
Acid value, max.	6	10	15	20	20	5.3
Saponification value	186 to 200	186 to 200	186 to 200	186 to 200	186 to 200	5.4
Unsaponifiable matter, max. %	2.0	2.0	2.0	2.0	2.0	5.5
Polybromide test	negative	—	—	—	—	5.6
Test for presence of colophony	negative	negative	negative	negative	negative	5.7
Test for presence of blown oils	negative	negative	negative	negative	negative	5.8
Clearness and colour	To be agreed between purchaser and vendor					—
Odour	Similar to that of a reference sample agreed between purchaser and vendor **					—

* For lithographic varnishes, the viscosity to be specified should be agreed between purchaser and vendor. A deviation of 10% from the specific viscosities is permitted.

** The arbitration method should be agreed beforehand between purchaser and vendor.

4. SAMPLING

See ISO Recommendation R , * *Sampling Raw Materials for Paints and Varnishes.*

5. TEST METHODS

5.1 Viscosity

Any method by which can be measured the viscosity of liquids by means of a falling sphere.

5.2 Ash

Refer to the relevant method described in the Annex to ISO Recommendation R 150 **.

5.3 Acid value

Refer to the relevant method described in the Annex to ISO Recommendation R 150. **

5.4 Saponification value

Refer to the relevant method described in the Annex to ISO Recommendation R 150. **

5.5 Unsaponifiable matter

Refer to the relevant method described in the Annex to ISO Recommendation R 150. **
The solvent to be used should be agreed between purchaser and vendor.

5.6 Polybromide test

5.6.1 *Reagents*

5.6.1.1 Sulphuric acid, 4N.

5.6.1.2 Potassium hydroxide, 1N ethanolic solution.

5.6.1.3 Sodium sulphate, anhydrous.

5.6.1.4 Bromine, analytical grade.

5.6.1.5 Diethyl ether, $d_{20} = 0.712$ to 0.716 having a residue non-volatile at 80 °C of not more than 0.001 %.

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

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* At present Draft ISO Recommendation No. 731.

** ISO Recommendation R 150, *Raw, Refined and Boiled Linseed Oil*, gives in the Annex extracts from the "Standard Methods of the Oils and Fats Section" and "Standard Methods of the Organic Coatings Section" of the International Union of Pure and Applied Chemistry (IUPAC).

5.6.2 *Preparation of the fatty acids*

Saponify about 15 g of the oil with 70 ml of the ethanolic potassium hydroxide solution by boiling for one hour under reflux.
Transfer the soap solution in a porcelain dish and, after addition of 50 ml of hot distilled water, expel the ethanol by evaporating on a steam bath.

Dissolve the soap in hot distilled water, transfer to a tall beaker and acidify with 25 ml of sulphuric acid. Boil the solution whilst passing a slow current of carbon dioxide through it, until the layer of fatty acids is clear. After cooling to room temperature, add 150 ml of the light petroleum. Filter the light petroleum layer through anhydrous sodium sulphate (the filtrate should not contain water).
Evaporate the solvent on the steam bath, removing the last traces with a slow current of carbon dioxide.

5.6.3 *Procedure*

Dissolve 1 g of the fatty acids mentioned in clause 5.6.2 in 10 ml diethyl ether, and cool to −10 °C in a 150 ml conical flask with a ground-glass stopper.

Subsequently add 0.3 ml of bromine carefully, while stirring. After swirling thoroughly, place the flask in an ice water bath and allow to stand for 5 min at 0 °C, until any sediment present has settled. Experience has shown that one of the following phenomena takes place:

(*a*) a crystalline sediment is produced . . . . . see clause 5.6.3.1,
(*b*) a crystalline sediment and dark emulsion are produced . . . see clause 5.6.3.2,
(*c*) a dark emulsion is formed. . . . . see clause 5.6.3.3,
(*d*) the solution is clear . . . . . see clause 5.6.3.3.

5.6.3.1 The immediate formation of a crystalline sediment reveals the presence of linseed oil (or of another non-polymerized polybromide-forming oil). The test is then reported as being positive.

5.6.3.2 If, in addition, an emulsion or a heavy liquid layer is formed on the bottom of the flask—as usually occurs in the case of stand oil of higher viscosity—add 5 ml of diethyl ether at 0 °C and swirl the flask again in the ice water bath, in order to facilitate the detection and identification of the crystalline sediment. If this is not sufficient to dissolve the heavy liquid phase, make further additions of 5 ml of cooled diethyl ether and cool again until the heavy liquid layer dissolves. Finally, examine the contents of the flask again for the presence of crystalline sediment.

5.6.3.3 If only a dark emulsion or a heavy liquid layer is formed on the bottom of the flask, or if the solution is clear, allow the mass to stand in an ice water bath for 12 to 16 hours. If crystals are formed after this period, remove the liquid phase by additions of diethyl ether (5 ml at a time, as under clause 5.6.3.2), and examine again for the presence of crystals.

No crystalline sediment and a clear solution denote either the absence of linseed oil or an amount too small for detection. Report the test then as being negative.