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BUREAU OF STANDARDS

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RECOMMENDED SPECIFICATION FOR LINSEED OIL—RAW, REFINED, AND BOILED

PREPARED AND RECOMMENDED BY THE U. S. INTERDEPARTMENTAL COMMITTEE ON PAINT SPECIFICATION STANDARDIZATION APRIL 16, 1919

[This committee was appointed at the suggestion of the Secretary of Commerce, and consisted of representatives of the War, Navy, Agriculture, Interior, Post Office, Treasury, and Commerce Departments, the Railroad Administration, the Panama Canal, and the war service committee of the Paint Manufacturers Association of the United States. The committee submitted a preliminary draft of this specification to more than 300 representatives of the paint and varnish industry, including all of the large manufacturers of linseed oil, and gave careful consideration to the large number of replies received in time]

CONTENTS

Page
1. General 1
    - Raw linseed oil 1
    - Refined linseed oil 2
    - Boiled linseed oil 2
2. Sampling 3
3. Laboratory examination 4
4. Reagents for testing 8
5. Basis of purchase 9

1. GENERAL

Linseed oil, raw, refined, or boiled, as specified in contract, shall be pure and shall conform to the following requirements:

RAW LINSEED OIL

	Maximum	Minimum
Loss on heating at 105 to 110° C (per cent)	0.2	
Foots by volume (per cent)	2.0	
Specific gravity 15.5/15.5° C	.936	0.932
Acid number	6.0	
Saponification number	195.0	189.0
Unsaponifiable matter (per cent)	1.5	
Iodine number (Hanus)[a]		170.0
Color	Not darker than a freshly prepared solution of 1.0 g potassium bichromate in 100 cc pure strong (1.84 specific gravity) sulphuric acid.	

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[a] When raw linseed oil from North American seed is specified by the purchaser, the iodine number must be not less than 180 and the oil shall conform to all the other requirements as above.

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REFINED LINSEED OIL

Contract shall state whether acid refined or alkali refined is desired.

	Maximum	Minimum
Loss on heating at 105 to 110° C (per cent)	0.2	
Foots by volume (per cent)	.2	
Specific gravity at 15.5/15.5° C	.936	0.932
Acid number (acid refined oil)	9.0	3.0
Acid number (alkali refined oil)	3.0	
Saponification number	195.0	189.0
Unsaponifiable matter (per cent)	1.5	
Iodine number (Hanus) [a]		170.0
Color	Not darker than a freshly prepared solution of 0.1 g potassium bichromate in 100 cc pure strong (1.84 specific gravity) sulphuric acid.	

[a] When refined linseed oil from North American seed is specified by the purchaser, the iodine number must be not less than 180 and the oil shall conform to all the other requirements as above.

BOILED LINSEED OIL

Boiled oil shall be pure, well-settled linseed oil that has been boiled with oxides of manganese and lead. It shall conform to the following requirements:

	Maximum	Minimum
Loss on heating at 105 to 110° C (per cent)	0.2	
Specific gravity at 15.5/15.5° C	.945	0.937
Acid number	8.0	
Saponification number	195.0	189.0
Unsaponifiable matter (per cent)	1.50	
Iodine number (Hanus) [a]		168.0
Ash (per cent)	.7	.2
Manganese (per cent)		.03
Lead (per cent)		.1
Time of drying on glass (hours)	20.0	

[a] When boiled linseed oil from North American seed is specified by the purchaser, the iodine number must be not less than 178 and the oil shall conform to all the other requirements as above.

2. SAMPLING

The method of sampling given under (*a*) below should be used whenever it is feasible to apply it. To meet conditions when (*a*) is not applicable, method (*b*), (*c*), or (*d*) is to be used, according to the special conditions that obtain.

(*a*) DURING LOADING OF TANK CARS OR FILLING OF CONTAINERS FOR SHIPMENT AT THE FACTORY.—The purchaser's inspector shall draw a sample at the discharge pipe where it enters the receiving vessel or vessels. The total sample shall be not less than 5 gallons and shall be a composite of small samples of not more than 1 pint each taken at regular intervals during the entire period of loading or filling.

The sample thus obtained shall be thoroughly mixed, and from this composite sample three portions of not less than 1 quart each shall be placed in clean dry glass bottles or tin cans which must be filled with the sample and securely stoppered with new clean corks or well-fitting metal covers or caps. These shall be sealed and labeled distinctly by the inspector, and one delivered to the buyer, one to the seller, and the third held for check in case of dispute.

(*b*) FROM LOADED TANK CARS OR OTHER LARGE VESSELS.—The total sample shall be not less than 5 gallons and shall be a composite of numerous small samples of not more than 1 pint each taken from the top, bottom, and intermediate points by means of a glass or metal container with removable stopper or top. This device attached to a suitable pole is lowered to the various desired depths when the stopper or top is removed and the container allowed to fill. The sample thus obtained is handled as in (*a*).

(*c*) BARRELS AND DRUMS.—Not less than 5 per cent of the packages in any shipment or delivery of barrels and drums shall be sampled. The packages shall be shaken, rolled, and stirred to thoroughly mix the contents. The samples from the individual containers shall be taken through the bung hole or holes not less than 1¼ inch in diameter bored in the head or side for the purpose. The apparatus for drawing the sample shall consist of a glass tube about 1 inch in diameter and somewhat longer than the length or diameter of the oil container, a conical stopper that will fit the glass tube and is not more than ½ inch long fastened to a stiff metal rod not more than ¼ inch in diameter and not less than 4 inches longer than the glass tube. The stopper is lowered by the rod until it rests on the bottom of the cask, the tube slipped down slowly over the rod, and finally pressed on the stopper. By holding tube and rod the column of oil can then be removed. This process is repeated until the required amount of sample is obtained, which must be not less than 2 gallons. This is mixed and handled as in (*a*)

(*d*) Small Containers, Cans, etc., of 10 Gallons or Less.—Small containers, cans, etc., of 10 gallons or less should be sampled while filling by method (*a*) whenever possible. When method (*a*) is not applicable, it is mutually agreed that: In all cases the total sample taken shall not be less than 3 quarts. This shall be obtained by taking at least one package from each lot of not more than 300 packages. The sample thus taken shall be thoroughly mixed and subdivided as in (*a*).

3. LABORATORY EXAMINATION

Samples shall, in general, be tested by the following methods, but the purchaser reserves the right to apply any additional tests such as specific tests for foreign oils, rosin, etc., or use any available information to ascertain whether the material meets the specification. The laboratory sample shall be thoroughly mixed by shaking, stirring, or pouring from one vessel to another and the samples for the individual tests taken from this thoroughly mixed sample.

(*a*) Loss on Heating at 105 to 110° C.—Place 10 g of the oil in an accurately weighed 200 cc Erlenmeyer flask; weigh. Heat in an oven at a temperature between 105 and 110° C for 30 minutes; cool and weigh. Calculate the percentage loss. This determination shall be made in a current of dry carbon dioxide gas.

(*b*) Foots.—With all materials at a temperature between 20 and 27° C mix, by shaking in a stoppered flask for exactly one minute, 25 cc of the well-shaken sample of oil, 25 cc of acetone (see 4(*a*)) and 10 cc of the acid calcium chloride solution (see 4(*b*)). Transfer the mixture to a burette where settling can take place for 24 hours. The temperature during this period should be between 20 and 27° C.

The volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture is read in tenths of a cubic centimeter or a fraction thereof. This reading multiplied by four expresses the amount of foots present as percentage by volume of the oil taken.

(*c*) SPECIFIC GRAVITY.—Use a pyknometer accurately standardized and having a capacity of at least 25 cc or any other equally accurate method, making the test at 15.5° C, water being unity at 15.5° C.

(*d*) ACID NUMBER.—Weigh from 5 to 10 g of the oil. Transfer to a 350 cc Erlenmeyer flask. Add 50 cc of neutral 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask. Heat on a steam bath for 30 minutes. Cool and add phenolphthalein indicator. Titrate to a faint permanent pink color with the standard sodium hydroxide solution. Calculate the acid number (milligrams KOH per gram of oil).

(*e*) SAPONIFICATION NUMBER.—Weigh about 2 g of the oil in a 350 cc Erlenmeyer flask. Add 25 cc alcoholic sodium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with half normal sulphuric acid. Run two blanks with the alcoholic sodium hydroxide solution. (See 4(*h*).) These should check within 0.1 cc N/2 H_2SO_4. From the difference between the number of cubic centimeters of N/2 H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g of oil).

(*f*) UNSAPONIFIABLE MATTER.—Weigh 8 to 10 g of the oil. Transfer to a 250 cc, long-neck flask. Add 5 cc of strong solution of sodium hydroxide (equal weights of NaOH and H_2O), and 50 cc 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid but do not project the liquid onto the sides of the flask. At the end of two hours remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500 cc glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc, add 100 cc redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500 cc separatory funnel and repeat the process using 60 cc of ether. After thorough separation draw off the aqueous solution into a 400 cc beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 cc of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the accumulated water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock, and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portionwise if necessary) into a 250 cc flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool and weigh. (*The unsaponifiable oil from adulterated drying oils is volatile and will evaporate on long heating. Therefore heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.*)

(*g*) IODINE NUMBER.—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g (0.10 to 0.20 g) to a 500 cc bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc of chloroform to two empty bottles like that used for the sample. Add to each bottle 25 cc of the Hanus solution (see 4 (*g*)) and let stand with occasional shaking for one-half hour. Add 10 cc of the 15 per cent potassium iodide solution and 100 cc of water, and titrate with standard sodium thiosulphate using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titration and the titration on the samples and the iodine value of the thiosulphate solution, calculate the iodine number of the samples tested. (Iodine number is centigrams of iodine to 1 g of sample.)

(*h*) ASH.—Tare a porcelain crucible or dish. Add 10 to 25 cc of oil, carefully weighing the amount added. Place on a stone slab on the floor of a hood. Ignite by playing the flame of a burner on the surface of the oil and allow to burn quietly until most of the oil is burned off; then transfer to a muffle or over a flame and continue heating at a very low temperature (not over a dull red) until all carbonaceous matter is consumed. Cool, weigh, and calculate the percentage of ash. Moisten the ash with a few drops of water and test with litmus paper. Record whether neutral or alkaline. Wash any ash adhering to the test paper back into the crucible. Dissolve the ash in dilute nitric acid to which a little hydrogen peroxide has been added. After solution is complete make up the volume to about 50 cc with nitric acid and water so that the final volume will contain about 1 volume of concentrated nitric acid and 3 volumes of water. Boil to remove excess of hydrogen peroxide. Determine manganese by the bismuthate method as described in Treadwell-Hall, Analytical Chemistry, third edition, volume 2, page 617.

Ash another portion of the oil and dissolve the ash as above in nitric acid and hydrogen peroxide. Transfer to a 250 cc beaker and dilute to about 200 cc. This volume of solution should contain 15 to 20 cc of concentrated nitric acid. Electrolyze this solution using platinum electrodes (the anode being previously weighed) with a current density of about 0.5 ampere and 2 to 2.5 volts. It is best to pass the current overnight (about 15 hours). On removing the anode, it is carefully washed in clear water, dried in a steam oven, transferred to an oven where it is heated to 180° C, cooled and weighed. The increase in weight of the anode multiplied by 0.86 gives the weight of lead in the sample. Calculate to percentage. If desired, the lead may be determined by the sulphate or any other accurate method in place of the electrolytic method given above.

(*i*) TIME OF DRYING ON GLASS.—Flow the oil over a perfectly clean glass plate and allow to drain in a vertical position in a well-ventilated room at a temperature between 15 and 39° C. After about 2 hours the film is tested at intervals with the finger at points not less than 2½ cm from the edges. The film will be considered dry when it adheres no longer to the finger and does not rub up appreciably when the finger is rubbed lightly across the surface. With boiled linseed oil this usually occurs in from 5 to 18 hours.

(*j*) COLOR.—Prepare a fresh solution of pure potassium bichromate in pure strong (1.84 specific gravity) colorless sulphuric acid. For raw oil, this solution should be in the proportion of 1.0 g potassium bichromate to 100 cc (184.0 g) sulphuric acid. For refined oil, the solution should be in the proportion of 0.1 g potassium bichromate to 100 cc sulphuric acid. Place the oil and colored solution in separate thin-walled, clear glass tubes of the same diameter (1 to 2 cm) to a depth of not less than 2.5 cm and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

4. REAGENTS FOR TESTING

The following reagents will be required:

(*a*) Acetone that will pass the specification of the United States Pharmacopoeia.

(*b*) Acid Calcium Chloride Solution.—Saturate with calcium chloride a mixture of 90 parts water and 10 parts concentrated hydrochloric acid (specific gravity 1.2).

(*c*) Standard Sodium Thiosulphate Solution.—Dissolve pure sodium thiosulphate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g crystallized sodium thiosulphate will be present in 1000 cc of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Analytical Chemistry, Treadwell-Hall, Vol. II, 3d ed., p. 646.) This solution will be approximately decinormal, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly decinormal strength. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(*d*) Starch Solution.—Stir up 2 to 3 g of potato starch or 5 g soluble starch with 100 cc of 1 per cent salicylic acid solution, add 300 to 400 cc boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

(*e*) Standard Iodine Solution.—Dissolve 13 g of resublimed iodine and 18 g of pure potassium iodide (free from iodates) in 50 cc of distilled water, and dilute to 1000 cc. Determine its exact value by titrating with the standard sodium thiosulphate solution.

(*f*) Potassium Iodide Solution.—Dissolve 150 g of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

(*g*) Hanus Solution.—Dissolve 13.2 g of iodine in 1000 cc of glacial acetic acid (99.5 per cent) that will not reduce chromic acid. Add enough bromine to double the halogen content, determined by titration (3 cc of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

(*h*) STANDARD SODIUM HYDROXIDE SOLUTION.—Prepare a stock concentrated solution of sodium hydroxide by dissolving sodium hydroxide in water in the proportion of 200 g NaOH to 200 cc water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 cc and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolphthalein as indicator. (See Bureau of Standards Scientific Paper 183.) This solution will be approximately one-fourth normal, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

(*i*) ALCOHOLIC SODIUM HYDROXIDE SOLUTION.—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle, and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g to 1000 cc), kept at about 50° C for 15 days, and then distilled.

(*j*) HALF NORMAL SULPHURIC ACID SOLUTION.—Add about 15 cc sulphuric acid (1.84 specific gravity) to distilled water, cool and dilute to 1000 cc. Determine the exact strength by titrating against freshly standardized sodium hydroxide or by any other accurate method. Either adjust to exactly half normal strength or leave as originally made, applying appropriate correction.

5. BASIS OF PURCHASE

Material is to be purchased by weight or volume as specified in the contract. When purchased by weight, the price shall be quoted per pound or per hundred pounds. When purchased by volume, a gallon of oil shall mean 231 cubic inches, at 15.5° C.

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