

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS  
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CIRCULAR OF THE BUREAU OF STANDARDS, No. 93

[3d edition, issued April 29, 1927]

UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR  
PAINTS, IRON OXIDE AND IRON HYDROXIDE

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 13b

[Revised September 2, 1925]

This specification was officially adopted by the Federal Specifications Board on February 3, 1922, for the use of the departments and independent establishments of the Government in the purchase of iron-oxide and iron-hydroxide paints.

[The date on which the technical requirements of this revision of this specification became mandatory for all departments and independent establishments of the Government was September 2, 1925. The changes included in this revision were authorized by the Federal Specifications Board and promulgated in a circular letter on above revision date]

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## I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

## II. TYPES

Iron oxide and iron hydroxide paints shall be of the following types: Semipaste in linseed oil and ready mixed.

## III. MATERIAL AND WORKMANSHIP

See detail requirements.

## IV. GENERAL REQUIREMENTS

See detail requirements.

## V. DETAIL REQUIREMENTS

### 1. PIGMENT

The pigment in both semipaste and ready-mixed paint shall be very finely ground iron oxide, iron hydroxide, siliceous minerals or a mixture thereof, to which carbon pigment may be added, if necessary, to produce the required color. It must be free from organic coloring matter (dyes or lakes). The pigment shall show on analysis not less than 30 per cent of ferric oxide ( $\text{Fe}_2\text{O}_3$ ). The total of the ferric oxide, insoluble siliceous matter, and loss on ignition shall be not less than 90 per cent.

### 2. LIQUID

The liquid in semipaste paint shall be entirely linseed oil; in ready-mixed paint it shall contain not less than 75 per cent linseed oil, the balance to be combined drier and thinner. The thinner shall be turpentine, volatile mineral spirits, or a mixture thereof.

### 3. SEMIPASTE

Semipaste shall be made by thoroughly grinding the pigment with linseed oil.

The semipaste as received and three months thereafter shall be not caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances, in all proportions without curdling. The color and hiding power when specified shall be equal to that of a sample mutually agreed upon by buyer and seller. The weight per gallon shall be not less than  $13\frac{1}{2}$  pounds. The paste shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment.....	72.0	68.0
Linseed oil.....	32.0	28.0
Moisture and other volatile matter.....	.7	-----
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment).....	3.5	-----

#### 4. READY-MIXED PAINT

Ready-mixed paints shall be well ground, shall not settle badly or cake in the container, shall be readily broken up with a paddle to a smooth, uniform paint of good brushing consistency, and shall dry within 18 hours to a full oil gloss, without streaking, running, or sagging. The color and hiding power when specified shall be equal to those of a sample mutually agreed upon by buyer and seller. The weight per gallon shall not be less than 12 pounds. The paint shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment.....	57.0	53.0
Liquid (containing at least 75 per cent linseed oil).....	47.0	43.0
Water.....	.5	-----
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment).....	3.5	-----

### VI. METHODS FOR SAMPLING AND TESTING

*Deliveries will, in general, be sampled and tested by the following methods, but the purchaser reserves the right to use any additional available information to ascertain whether the material meets the specification.*

#### 1. SAMPLING

It is mutually agreed by buyer and seller that a single package out of each lot of not more than 1,000 packages shall be taken as representative of the whole. Whenever possible an original unopened container shall be sent to the laboratory, and when this is for any reason not done the inspector shall determine by thorough testing with a paddle or spatula whether the material meets the requirement regarding caking in the container. He shall then thoroughly mix the contents of the container and draw a sample of not less than 5 pounds of the thoroughly mixed paint, place it in a clean, dry metal or glass container, which it shall nearly fill. The container shall be closed with a tight cover, sealed, marked, and sent to the laboratory for test with the inspector's report on caking.

When requested a duplicate sample may be taken from the same package and delivered to the seller, and the inspector may take a third sample to hold for test in case of dispute.



## 2. LABORATORY EXAMINATION—SEMIPASTE

(a) **CAKING IN CONTAINER.**—When an original package is received in the laboratory it shall be weighed, opened, and stirred with a stiff spatula or paddle. The paste must be no more difficult to break up than a normal good grade of semipaste paint. The semipaste shall finally be thoroughly mixed, removed from the container, and the container wiped clean and weighed. This weight subtracted from the weight of the original package gives the net weight of the contents. A portion of thoroughly mixed semipaste shall be placed in a clean container and the portions for the remaining tests promptly weighed out.

(b) **COLOR.**—Place some of the paint on a clean, clear glass plate. Place some of the standard agreed upon beside the sample on the plate, turn the glass over, and compare the colors.

(c) **WEIGHT PER GALLON.**—From the weight of a known volume of the paste calculate the specific gravity, which multiplied by 8.33 gives the weight in pounds per gallon. Any suitable container of known volume may be used for the purpose, but a short cylinder of heavy glass with rounded bottom about 75 mm high and having a capacity of from 125 to 175 cc (a glass cap to keep dust from reagent bottle stopper) is a convenient apparatus for the purpose. The capacity of this vessel is determined to within 1 cc. The paste is packed into it until completely full, the top leveled off smooth with a spatula, and weighed to  $\pm 0.5$  g. Subtract the weight of the empty container and divide the remainder by the number of cubic centimeters representing the capacity of the container. The quotient is the specific gravity, which can be thus determined within  $\pm 2$  in the second decimal place.

(d) **MIXING WITH LINSEED OIL.**—One hundred grams of the paste shall be placed in a cup, 30 cc linseed oil added slowly, with careful stirring and mixing with a spatula or paddle. The resulting mixture must be smooth and of good brushing consistency.

(e) **MOISTURE AND OTHER VOLATILE MATTER.**—Weigh accurately from 3 to 5 g of the paste into a tared flat-bottomed dish about 8 cm in diameter, spreading the paste over the bottom. Heat at 105 to 110° C. for three hours, cool, and weigh. Calculate loss in weight as percentage of moisture and volatile matter.

(f) **PERCENTAGE OF PIGMENT.**—Weigh accurately about 15 g of the paste into a weighed centrifuge tube. Add 20 to 30 cc of "extraction mixture" (see Reagents), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add sufficient of the reagent to make a total of 60 cc in the tube. Place the tube in the container of a centrifuge, surround with water, and counterbalance the container of the opposite arm with a similar tube or a tube with water. Whirl at a moderate speed until well settled.

Decant the clear supernatant liquid. Repeat the extraction three times with 40 cc of extraction mixture. After drawing off the extraction mixture set the tube in a beaker of water at about 80° C. or on top of a warm oven for 10 minutes, then in an oven at 105 to 110° C. for two hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 screen to remove any skins, and preserve in a stoppered bottle.

(g) PREPARATION OF FATTY ACIDS.—To about 25 g of the paste in a porcelain casserole add 15 cc of aqueous sodium hydroxide (see Reagents) and 75 cc of ethyl alcohol, mix and heat uncovered on a steam bath until saponification is complete (about one hour). Add 100 cc of water, boil, add sulphuric acid of specific gravity 1.2 (8 to 10 cc in excess), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer, wash once with water, then add 50 cc of water and 50 cc of ether. Shake very gently with a whirling motion to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw off the aqueous layer and wash the ether layer with one 15 cc portion of water and then with 5 cc portions of water until free from sulphuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g of anhydrous sodium sulphate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sodium sulphate. Decant this clear solution, if necessary, through a dry filter paper, into a dry 100 cc Erlenmeyer flask. Pass a rapid current of dry air (pass through a  $\text{CaCl}_2$  tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75° C. on a dry hot plate until the ether is entirely driven off.

NOTE.—It is important to follow all of the details, since ether generally contains alcohol, and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulphate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(h) TEST FOR MINERAL OIL AND OTHER UNSAPONIFIABLE MATTER.—Place 10 drops of the fatty acid (g) in a 50 cc test tube, add 5 cc of alcoholic soda (see Reagents), boil vigorously for five minutes, add 40 cc of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present. If the solution is not clear, the oil is not pure linseed oil.

(i) IODINE NUMBER OF FATTY ACIDS.—Place a small quantity of the fatty acids (*g*) in a small weighing burette or beaker. Weigh accurately. Transfer by dropping from 0.09 g to 0.15 g into 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 amount of sample used. Add 10 cc of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 cc of the Wijs solution (see Reagents) and let stand with occasional shaking for one hour in a dark place at a temperature of from 21 to 23° C. 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 titrations and the titration on the sample and the iodine value of the thiosulphate solution, calculate the iodine number of the sample tested. (Iodine number is centigrams of iodine to 1 g of sample.) If the iodine number is less than 175, the oil does not meet the specification.

(j) COARSE PARTICLES AND SKINS.—Dry in an oven at 105 to 110° C. a No. 325 screen, cool, and weigh accurately. Weigh an amount of semipaste containing 10 g of pigment (see 2 (*f*)), add 50 cc of kerosene, mix thoroughly, and wash with kerosene through the screen, breaking up all lumps, but not grinding. After washing with kerosene until all but the particles too coarse to pass the screen have been washed through, wash all kerosene from the screen with ether or petroleum ether, heat the screen for one hour at 105 to 110° C., cool, and weigh.

### 3. ANALYSIS OF PIGMENT

(a) ORGANIC COLORING MATTER.—(A. S. T. M. Standards, 1918, p. 656). Test the pigment successively with hot water, 95 per cent alcohol, alcoholic sodium hydroxide, and acetic acid. Chloroform, sodium hydroxide, sulphuric acid, hydrochloric acid-stannous chloride solution, and other reagents may be tried. The solutions should remain colorless. The presence of an organic color may often be detected by the characteristic odor given off on ignition.

(b) TOTAL IRON OXIDE.—Ignite 1 g of the pigment in a porcelain crucible at a dull red heat to destroy organic matter. Transfer to a 400 cc beaker and add 25 cc of concentrated hydrochloric acid. Cover with a watch glass and digest just short of boiling (80 to 90° C.) till no dark specks can be seen in the insoluble residue. The addition of a few drops of 5 per cent stannous chloride solution after adding the acid greatly assists dissolving of the iron. When the residue is light in color, the solution of iron may be considered complete. This



may take from 15 minutes to an hour or longer. Then add 25 to 50 cc of water and heat to gentle boiling (avoid vigorous prolonged boiling), drop in stannous chloride solution (see Reagents) slowly until the last drop makes the solution colorless or free from any tinge of yellow. It is best to keep the watch glass on the beaker while adding the tin solution, with agitation of the hot iron solution after each addition. If too much stannous chloride is added by mistake, add permanganate to the solution until a yellow color appears, then again add stannous chloride drop by drop until the yellow color just disappears. Dilute with 200 cc of cold water, then add all at once with vigorous stirring 15 cc of mercuric chloride solution. Let stand three or four minutes. A slight white precipitate should form. If none, or a heavy grayish precipitate forms, the determination should be discarded and repeated. Add 15 cc of phosphoric acid mixture (see Reagents) and 3 drops of diphenylamine solution (see Reagents). Titrate with standard potassium dichromate solution (see Reagents) taking the change of the dark-green color to a sudden blue-black color as the end point. Calculate the total iron found to  $\text{Fe}_2\text{O}_3$ . If preferred, potassium ferricyanide may be used as an external indicator. In this case omit the addition of the phosphoric acid mixture and the diphenylamine indicator, but add the dichromate solution, and toward the end take out a very small drop of the solution which is being titrated and touch this to a drop of dilute potassium ferricyanide solution (see Reagents), best placed on a paraffined surface. Toward the end point the blue is replaced by a bluish-green coloration, perceptible at the junction of the two solutions. Take as the end point when no trace of the bluish-green coloration can be detected. Any other accurate method for determining iron may be used, at the option of the analyst.

(c) LOSS ON IGNITION.—Ignite 1 g of the pigment to constant weight. It is safest to use a porcelain crucible for this purpose.

(d) INSOLUBLE SILICEOUS MATTER.—Transfer 1 g of the pigment to a porcelain dish, ignite at a dull red heat to destroy organic matter, cool, add 20 cc of hydrochloric acid, 1:1, cover and heat on a steam bath until no dark specks can be seen in the insoluble residue. Remove cover, add 10 cc of strong hydrochloric acid, evaporate to dryness on a steam bath, moisten with hydrochloric acid, add water, and wash on to a filter paper with hot water; wash, ignite, and weigh the insoluble siliceous matter.

#### 4. LABORATORY EXAMINATION—MIXED PAINT

(a) CAKING IN CONTAINER.—Follow the procedure outlined in 3 (a), noting that the paint should be no more difficult to break up than a good grade of mixed paint.

(b) COLOR.—Follow the procedure outlined in 3 (b).

(c) WEIGHT PER GALLON.—Weigh a clean, dry, 100 cc graduated flask. Fill to the mark with the thoroughly mixed paint and weigh again. The increase in weight expressed in grams, divided by 100, gives the specific gravity, which multiplied by 8.33 gives the weight in pounds per gallon.

(d) BRUSHING PROPERTIES AND TIME OF DRYING.—Brush the well-mixed paint on a suitable panel, which may be ground glass, steel, or well-filled wood. Note whether the paint works satisfactorily under the brush. Place the panel in a vertical position in a well-ventilated room and let stand for 18 hours. The paint shall be dry, smooth, and free from streaks. Flow a portion of the paint on a clean glass plate. Let dry in a nearly vertical position at room temperature (65 to 100° F.). The film shall show no streaking or separation within a distance of 4 inches from the top.

(e) WATER.—Mix 100 g of the paint in a 500 cc short-neck glass flask<sup>1</sup> with 75 cc of toluol (free from water). Connect with the distilling trap and condenser, and heat so that the condensed distillate falls from the end of the condenser at the rate of from two to five drops per second. Continue the distillation at the specified rate until no water is visible on any part of the apparatus except at the bottom of the trap. This operation usually requires less than an hour. A persistent ring of condensed water in the condenser tube should be removed by increasing the rate of distillation for a few minutes. The number of cubic centimeters of condensed water measured in the trap at room temperature is the percentage of water in the paint.

(f) VOLATILE THINNER.—Follow the procedure outlined in 2 (e). Correct the result for any water found (see 4 (e)) and report the remainder as volatile thinner.

(g) PERCENTAGE OF PIGMENT.—Follow the procedure outlined in 2 (f).

(h) TESTING NONVOLATILE VEHICLE.—Follow the procedure outlined in 2 (g), 2 (h), and 2 (i), except that in the preparation of the fatty acids the mixture of paint and alkali is heated on the steam bath until all volatile thinner is driven off.

(i) COARSE PARTICLES AND SKINS.—Follow the procedure outlined in 2 (j).

(j) TESTING PIGMENT.—Follow the procedure outlined in 3 (a) to 4 (d), inclusive.

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<sup>1</sup> The apparatus for determining water is that described in "Standard method of test for water in petroleum products and other bituminous materials," Serial Designation D-95-24, A. S. T. M. Standards, 1924, p. 901, and Figure 1 (b) and (c), p. 902.



## 5. REAGENTS

## (a) EXTRACTION MIXTURE.—

- 10 volumes ether (ethyl ether).
- 6 volumes benzol.
- 4 volumes methyl alcohol.
- 1 volume acetone.

(b) AQUEOUS SODIUM HYDROXIDE.—Dissolve 100 g of sodium hydroxide in distilled water and dilute to 300 cc.

(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 of 24.83 g of crystallized sodium thiosulphate to 1,000 cc of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall, *Analytical Chemistry*, vol. 2, 6th ed., p. 551.) 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. 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 of 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, then dilute to 1 liter.

(e) POTASSIUM IODIDE SOLUTION.—Dissolve 150 g of potassium iodide free from iodate in distilled water and dilute to 1,000 cc.

(f) WIJS SOLUTION.—The preparation of the iodine monochloride solution presents no great difficulty, but it should be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine over that required to form the monochloride. This condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using gentle heat to assist the solution, if it is found necessary. Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15° C. and is free from reducing impurities in the proportion so that 13 g of iodine will be present in 1,000 cc of solution. Set aside a small portion of this solution while pure, and pass dry chlorine into the remainder until the halogen content of the solution is doubled. Ordinarily it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

(g) ALCOHOLIC SODIUM HYDROXIDE SOLUTION.—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g per 1,000 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, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g to 1,000 cc), kept at about 50° C. for 15 days, and then distilled.

(h) STANDARD POTASSIUM DICHROMATE SOLUTION.—Dissolve 4.903 g of pure, dry, crystallized potassium dichromate in water and dilute to 1,000 cc (1 cc = 0.008 g  $\text{Fe}_2\text{O}_3$ ). Determine its exact iron value with Bureau of Standards Standard Sample No. 27a, Sibley Iron Ore.

(i) PHOSPHORIC ACID MIXTURE.—Mix 150 cc of sulphuric acid (specific gravity 1.84) with 150 cc of phosphoric acid (specific gravity 1.7), and dilute the mixture with water to 1,000 cc.

(j) DIPHENYLAMINE INDICATOR.—Dissolve 1 g of diphenylamine in 100 cc of concentrated sulphuric acid. Use three drops of the solution as indicator.

(k) STANNOUS CHLORIDE SOLUTION.—Dissolve 50 g of the crystallized salt ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) in 300 cc of concentrated hydrochloric acid and dilute with water to 500 cc. Keep the clear solution in a tightly stoppered bottle containing some metallic tin.

(l) MERCURIC CHLORIDE SOLUTION.—A saturated solution of  $\text{HgCl}_2$  (60 to 100 g per liter).

(m) POTASSIUM FERRICYANIDE SOLUTION.—A 1 per cent solution is satisfactory. Dissolve a piece half as big as a small pea in 50 cc of water. This solution must be made fresh when wanted, because it does not keep.

## VII. PACKING AND MARKING OF SHIPMENTS

Shall be in accordance with commercial practice unless otherwise specified.

## VIII. NOTES

This specification applies to iron-oxide and iron-hydroxide paints of red and brown colors. The paint may be ordered in the form of either semipaste paint or ready-mixed paint. The semipaste may be purchased by net weight or by volume. The ready-mixed paint should be purchased by volume (231 cubic inches to the gallon).

For formulas and methods of using this material and information regarding the use of other specification paint materials see Bureau of Standards Technologic Paper No. 274, entitled "Use of United States Government Specification Paints and Paint Materials."

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