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DEPARTMENT OF COMMERCE
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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR
OLIVE DRAB PAINT (SEMIPASTE AND READY-MIXED)

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 137

This specification was officially adopted by the Federal Specifications Board on May 1, 1924, for the use of the Departments and Independent Establishments of the Government in the purchase of olive drab paint (semipaste and ready-mixed)

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I. CLASSES

Olive drab paint shall be of the following classes: Semipaste in linseed oil and ready mixed.

II. MATERIAL

No details.

III. GENERAL REQUIREMENTS

No details.

IV. DETAIL REQUIREMENTS

1. PIGMENT

The pigment shall be composed of:

Ingredients	Maximum	Minimum
	<i>Per cent</i>	<i>Per cent</i>
White lead (basic carbonate, basic sulphate, or a mixture thereof)		35
Zinc oxide (ZnO)		30
White mineral pigments (containing no lead or zinc compounds), pure tinting colors, or any mixture thereof	30	
Organic colors	None.	
Sulphide sulphur	None.	

In no case shall the sum of the basic lead carbonate, basic lead sulphate, and zinc oxide be less than 70 per cent. The lead and zinc pigments may be introduced in the form of any mixture preferred of basic carbonate white lead, basic sulphate white lead, zinc oxide, or leaded zinc, provided the above requirements as to composition are met.

The difference between the total lead weighed as lead sulphate and the lead sulphate equivalent to the chromium found, multiplied by the factor 0.883 shall be considered white lead. It is not possible to determine the amount of lead carbonate and lead sulphate when carbonates or sulphates of other metals, such as calcium, are present. Also neither basic lead carbonate nor basic lead sulphate is a definite compound. The factor to convert PbSO_4 to $(\text{PbCO}_3)_2 \text{Pb}(\text{OH})_2$ is 0.854, to convert PbSO_4 to $\text{PbSO}_4 \text{PbO}$ is 0.868, and to convert PbSO_4 to $(\text{PbSO}_4)_2 \text{PbO}$ is 0.913. The arbitrary factor used under this specification is the mean of the largest and smallest of these three factors.

2. LIQUID

The liquid in semipaste paint shall be entirely pure raw or refined linseed oil; in ready-mixed paint it shall contain not less than 85 per cent pure raw linseed oil, the remainder 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 pure raw or refined 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 those of a sample mutually agreed upon by buyer and seller. The weight per gallon shall be not less than 18 pounds. The paste shall consist of:

Ingredients	Maximum	Minimum
	<i>Per cent</i>	<i>Per cent</i>
Pigment.....	77	73
Linseed oil.....	27	23
Moisture and other volatile matter.....	0.7	-----
Coarse particles and "skins" (total residue retained on No. 325 sieve based on pigment).....	2.0	-----

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 be not less than 15 pounds. The paint shall consist of:

Ingredients	Maximum	Minimum
	<i>Per cent</i>	<i>Per cent</i>
Pigment.....	66	62
Liquid (containing at least 85 per cent linseed oil).....	38	34
Water.....	0.5	-----
Coarse particles and "skins" (total residue retained on No. 325 sieve based on pigment).....	2.0	-----

V. 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 must be filled with the sample, closed with a tight cover, sealed, marked, and sent to the laboratory for test with the inspector's report on caking in container.

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) **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 vessel 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 plus or minus 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 plus or minus 2 in the second decimal place.

(c) **MIXING WITH LINSEED OIL.**—One hundred grams of the paste shall be placed in a cup, 18 cc of 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.

(d) **COLOR.**—To the mixture made, in (c), add 3 cc of drier (F. S. B. No. 20) and mix thoroughly. Prepare a mixture of the standard paste with 18 cc of linseed oil and 3 cc of drier, using the same linseed oil and drier for both lots of paint. Apply both paints on clean metal or glass so that the edges touch one another. Let dry and compare the colors.

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

(f) **PERCENTAGE OF PIGMENT.**—Weigh accurately about 15 g of the paste in 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 enough 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 counter-balance 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 twice with 40 cc of extraction mixture and once with 40 cc of ether. After drawing off the ether, 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 110 to 115° C. for two hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 sieve 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 and lead sulphate precipitate, wash once with water, then add 50 cc of water and 50 cc of ether. Shake very gently with a whirling action 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 CaCl₂ 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.

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 about 0.15 g (0.10 to 0.20 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. Re-weigh 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 Reagents) 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 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 170, the oil does not meet the specification.

(*j*) COARSE PARTICLES AND SKINS.—Dry in an oven at 105 to 110° C. a No. 325 sieve, cool, and weigh accurately. Weigh an amount of semipaste containing 10 g of pigment (see V 2 (*f*)), add 100 cc of kerosene, mix thoroughly, and wash with kerosene through the sieve, breaking up all lumps, but not grinding. After washing with kerosene until all but the particles too coarse to pass the sieve have been washed through, wash all kerosene from the sieve with ether or petroleum ether, heat the sieve and contents for one hour at 105 to 110° C., cool and weigh.

3. ANALYSIS OF PIGMENT

(a) **QUALITATIVE ANALYSIS.**—A complete qualitative analysis, following the well-established methods, is always advisable. Test a portion of the pigment with hydrochloric acid (1:1). No odor of hydrogen sulphide should develop. Boil, dilute, filter, and test the filtrate for metals other than lead and zinc (especially calcium and barium). The absence of calcium in this filtrate indicates that the extending pigments contain no calcium carbonate or calcium sulphate; the absence of barium indicates that the extending pigments contain no barium carbonate. To test for chromate, boil another small portion of the pigment with dilute nitric acid, filter, cool, and add to the filtrate a few cubic centimeters of ether and a few drops of hydrogen peroxide; stir, let stand until the ether layer separates. If this layer is deep blue, chromium is indicated. Test for Prussian blue (which is rarely present) by boiling a portion of the pigment with sodium hydroxide solution. A yellow or yellow-brown precipitate with a yellow liquid above it should result. Filter, add to the filtrate a mixture of ferric and ferrous salts, and render acid with dilute hydrochloric acid. A blue color indicates the presence of Prussian blue in the sample.

(b) **WHITE LEAD.**—Weigh accurately about 1 g of the pigment, transfer to a 250 cc beaker, moisten with a few drops of alcohol, add slowly 25 cc of concentrated hydrochloric acid, cover, and boil for 5 to 10 minutes. Dilute to about 150 cc with hot water and boil for 5 to 10 minutes. Filter on paper or on a Gooch crucible, and wash the insoluble residue thoroughly with hot water. Discard the residue for quantitative purposes. Nearly neutralize with ammonia the filtrate from the insoluble matter, dilute to about 300 cc and pass into the clear solution a rapid current of hydrogen sulphide to complete precipitation, let the sulphide of lead settle, filter on paper, wash with water containing some hydrogen sulphide, dissolve the sulphide in hot nitric acid (1:3), add 10 cc of sulphuric acid (1:1), evaporate until copious fumes of sulphuric anhydride are evolved; cool, add about 75 cc of water, and then about 75 cc of 95 per cent ethyl alcohol. Stir, let settle, filter on a Gooch crucible, wash with dilute alcohol, dry, ignite, and weigh as $PbSO_4$. Subtract the lead sulphate equivalent of the total chromium as found below, (c), multiply the remaining $PbSO_4$ by the factor 0.883 and report as white lead.

(c) **TOTAL CHROMIUM.**—Heat the filtrate from the lead sulphide to expel hydrogen sulphide. Cool, add sodium peroxide, keeping the beaker covered, in sufficient amount to render the solution alkaline and to oxidize the chromium to chromate. Boil until all the hydrogen peroxide is driven off, cool, acidify with sulphuric acid (1:4), add a measured excess of a freshly prepared solution of ferrous sul-

phate, and titrate the excess of ferrous iron with standard potassium dichromate, using potassium ferricyanide solution as outside indicator. Titrate a blank of an equal volume of the ferrous sulphate solution with the standard potassium dichromate. From the difference between the titration on the blank and on the sample, calculate the chromium in the sample to PbCrO_4 . From the PbCrO_4 found, calculate the equivalent of PbSO_4 by multiplying by the factor 0.938.

(d) ZINC OXIDE.—Weigh accurately about 1 g of the pigment, transfer to a 250 cc beaker, moisten with alcohol, add 30 cc of hydrochloric acid (1:2), boil for two or three minutes, add about 100 cc of water, let settle, and filter on paper; to the filtrate add about 2 g of ammonium chloride and strong ammonia until slightly alkaline (the latter to precipitate out any iron present), set on the steam bath to settle, filter into a 400 cc beaker, wash the precipitate once with water, remove the beaker and dissolve the iron hydroxide with dilute hydrochloric acid, catching the ferric chloride in a 250 cc beaker, add to this filtrate 1 g of ammonium chloride and make ammoniacal, let settle, filter and wash thoroughly with hot water, catching the filtrate and washings in the original 400 cc beaker, reserved from the first precipitation. Add a small piece of litmus paper. Render the filtrate first acid with hydrochloric acid, then add 3 cc of strong hydrochloric acid, heat nearly to boiling, and titrate with standard ferrocyanide, as in standardizing that solution (see Reagents). Calculate total zinc as zinc oxide.

(e) ORGANIC COLORING MATTER.—(*A. S. T. M. Standards, 1921, p. 690*).—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.

(f) CALCULATIONS.—Add the percentage of white lead (see V 3 (b)), zinc oxide (see V 3 (d)), and subtract from 100; the remainder is reported as extending and tinting pigments.

4. LABORATORY EXAMINATION, READY-MIXED PAINT

(a) CAKING IN CONTAINER.—Follow the procedure outlined in V 2 (a), noting that the paint should be no more difficult to break up than a good grade of mixed paint.

(b) 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.

(c) BRUSHING PROPERTIES AND TIME OF DRYING.—Brush this 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 should be dry 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.

(d) COLOR.—Paint the sample and the standard on clean metal or glass so that the edges touch one another. Let dry and compare colors.

(e) WATER.—Mix 100 g of the paint in a 300 cc flask with 75 cc of toluol. Connect with a condenser and distill until about 50 cc of distillate has been collected in a graduate. The temperature in the flask should be then about 105 to 110° C. The number of cubic centimeters of water collecting under the toluol in the receiver is the percentage of water in the paint.

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

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

(h) TESTING NONVOLATILE VEHICLE.—Follow the procedure outlined in V 2 (g), (h), and (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 V 2 (j).

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

5. REAGENTS

(a) URANYL INDICATOR FOR ZINC TITRATION.—A 5 per cent solution of uranyl nitrate in water or a 5 per cent solution of uranyl acetate in water made slightly acid with acetic acid.

(b) STANDARD POTASSIUM FERROCYANIDE.—Dissolve 22 g of the pure salt in water and dilute to 1,000 cc. To standardize transfer about 0.2 g (accurately weighed) of pure metallic zinc or freshly ignited pure zinc oxide to a 400 cc beaker. Dissolve in 10 cc of hydrochloric acid and 20 cc of water. Drop in a small piece of litmus paper, add ammonium hydroxide until slightly alkaline, then add hydrochloric acid until just acid, and then 3 cc of strong hydrochloric acid. Dilute to about 250 cc with hot water and heat nearly to boiling. Run in the ferrocyanide solution slowly from

a burette with constant stirring until a drop tested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing one minute. A blank should be run with the same amounts of reagents and water as in the standardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated.

(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, 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. 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 of boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

(e) EXTRACTION MIXTURE.—

10 volumes ether (ethyl ether).

6 volumes benzol.

4 volumes methyl alcohol.

1 volume acetone.

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

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

(h) HANUS SOLUTION.—Dissolve 13.2 g of iodine in 1,000 cc of 99.5 per cent glacial acetic acid, which 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.

(i) 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.

(j) STANDARD FERROUS SULPHATE SOLUTION.—Dissolve 14 g of pure crystallized ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in about 500 cc of water to which 25 cc of concentrated H_2SO_4 has been added, and then dilute to 1,000 cc. This solution should be freshly standardized when needed, as it does not keep well.

(k) STANDARD POTASSIUM DICHROMATE SOLUTION.—Dissolve 4.903 g of pure, dry, crystallized potassium dichromate in water and dilute to 1,000 cc. One cubic centimeter of this solution corresponds to 0.0108 g PbCrO_4 , or 0.0101 g PbSO_4 . This solution may be checked by determining its iron value on Bureau of Standards Standard Sample No. 27a, Sibley Iron Ore.

(l) POTASSIUM FERRICYANIDE SOLUTION.—Dissolve a piece of potassium ferricyanide 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.

VI. PACKING AND MARKING

No details.

VII. ADDITIONAL INFORMATION

This specification covers the requirements for a high-grade olive drab paint for outside and general use. It may be ordered either in the form of semipaste pigment ground in linseed oil or of ready-mixed paint, and the purchaser shall state which is desired.

The semipaste shall be purchased by net weight, and the ready-mixed paint either by weight or volume (231 cubic inches to the gallon).

VIII. GENERAL SPECIFICATIONS

No details.

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