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UNITED STATES GOVERNMENT SPECIFICATION FOR  
GREEN PAINT—SEMIPASTE AND READY MIXED

FEDERAL SPECIFICATIONS BOARD

STANDARD SPECIFICATION NO. 15

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 materials covered by it.

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1. GENERAL

The paint contemplated by this specification is a chrome green paint, and it may be ordered either in the form of semipaste pigment ground in linseed oil or as ready-mixed paint.

The basis of purchase may be either net weight or volume (231 cubic inches to the gallon).

(a) PIGMENT.—The pigment in both semipaste and ready-mixed paints should be a chrome green containing about 23 per

cent of color (sum of lead chromate and insoluble Prussian blue), about 10 per cent of magnesium silicate, aluminum silicate, or similar siliceous material, and about 67 per cent of barium sulphate. It should be made by precipitating the color on the proper base rather than by mixing the individual materials. It must yield on analysis:

	Maximum	Minimum
	Per cent	Per cent
Color (total lead chromate and insoluble Prussian blue).....		20
Material soluble in water, including soluble Prussian blue.....	0.2	.....
Acid-soluble or water-soluble calcium in any form, calculated as CaO.....	0.5	.....
Material other than color and barium sulphate.....	15	.....
The remainder must be barium sulphate.		

(b) 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 90 per cent pure raw linseed oil, the balance to be combined drier and thinner. The thinner shall be turpentine, volatile mineral spirits, or a mixture thereof.

(c) SEMIPASTE.—Semipaste paint 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 that of a sample mutually agreed upon by buyer and seller. The weight per gallon shall be not less than 16 pounds. The paste shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment.....	72	68
Linseed oil.....	32	28
Moisture and other volatile matter.....	0.7	.....
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment).....	2.5	.....

(d) **READY-MIXED PAINT.**—Ready-mixed paint 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 12 pounds. The paint shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment.....	55	50
Liquid (containing at least 90 per cent linseed oil).....	50	45
Water.....	0.5	.....
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment).....	2.5	.....

## 2. SAMPLING

It is mutually agreed by buyer and seller that a single package out of each lot of not more than 1000 packages 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. This sample shall be placed in a clean, dry metal or glass container, which it must 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.

Samples will, in general, be tested by the following methods, but the purchaser reserves the right to apply any additional tests, or use any available information to ascertain whether the material meets the specification.

## 3. 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 diffi-

cult 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 5 cm in diameter, spreading the paste over the bottom. Heat at 105 to 110° C for one hour, 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 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 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 an excess of sulphuric acid of specific gravity 1.2 (8 to 10 cc will usually suffice), 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 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 CaCl<sub>2</sub> 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 acids (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. 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 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 screen, cool, and weigh accurately. Weigh an amount of semipaste containing 10 g of pigment (see 3(*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.

#### 4. ANALYSIS OF PIGMENT

(a) QUALITATIVE ANALYSIS.—Test for Prussian blue 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 a mixture of ferric and ferrous salts to the filtrate, and render acid with dilute hydrochloric acid. A blue color indicates Prussian blue in the sample. Ignite another portion very gently to decompose the Prussian blue and make a qualitative analysis of the residue.

(b) **MATTER SOLUBLE IN WATER.**—Transfer 2.5 g of the pigment to a graduated 250 cc flask, add 100 cc of water, boil for 5 minutes, cool, fill to mark with water, mix, and allow to settle. Pour the supernatant liquid through a dry paper and discard the first 20 cc. Then evaporate 100 cc of the clear filtrate to dryness in a weighed dish, heat for one hour at 105 to 110° C, cool, and weigh. The residue should not exceed 0.002 g.

(c) **BARIUM SULPHATE AND SILICEOUS MATERIAL.**—Heat a 1 g portion of the pigment very gently in a small porcelain dish. The heat must be so regulated by moving the burner that the Prussian blue is thoroughly decomposed without rendering the iron difficultly soluble. Allow to cool, transfer to a 400 cc beaker, add 20 cc of concentrated hydrochloric acid, heat on steam bath for 30 minutes, boil for 5 minutes, dilute with hot water to about 250 cc, filter on paper while hot, wash thoroughly with hot water until the washings are free from lead and chlorine, and ignite and weigh the residue, which will be barium sulphate and siliceous material. Mix the ignited residue with about 10 times its weight of anhydrous sodium carbonate (grinding the mixture in an agate mortar if necessary), and fuse the mixture in a covered platinum crucible, heating about one hour. Let cool, place crucible and cover in a 250 cc beaker, add about 100 cc of water, and heat until the melt is disintegrated. Filter on paper (leaving crucible and cover in beaker) and wash the beaker and filter thoroughly with hot water to remove soluble sulphates. Place the beaker containing the crucible and cover under the funnel, pierce the filter with a glass rod, and wash the carbonate residue into the beaker by means of a jet of hot water. Wash the paper with hot, dilute hydrochloric acid (1:1), and then with hot water. If the carbonate residue is not completely dissolved, add sufficient dilute hydrochloric acid to effect solution, and remove crucible and cover, washing them with a jet of water. Heat the solution to boiling and add 10 to 15 cc of dilute sulphuric acid, and continue the boiling for 10 or 15 minutes longer. Let the precipitate settle, filter on a weighed Gooch crucible, wash with hot water, ignite, cool, and weigh as BaSO<sub>4</sub>. Subtract from the result of the previous determination to obtain the siliceous material.

(d) **LEAD AND CHROMIUM.**—Unite the filtrate and washings from barium sulphate and siliceous material (see (c)), dilute to 500 cc, nearly neutralize with ammonium hydroxide, and pass in a rapid stream of hydrogen sulphide until all the lead is precipitated as PbS; filter, wash with water containing a little hydro-

gen sulphide, dissolve in hot nitric acid (1:3), and determine lead as sulphate in usual manner, weighing as  $\text{PbSO}_4$ . Boil the filtrate from the lead sulphide to expel hydrogen sulphide. Add sodium peroxide in sufficient amount to render the solution alkaline and to oxidize the chromium to chromate. Boil until the hydrogen peroxide is driven off, cool, acidify with sulphuric acid (1:4), add a measured excess of a freshly prepared solution of ferrous sulphate, 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  $\text{PbCrO}_4$ . From the  $\text{PbCrO}_4$  found, calculate the equivalent of  $\text{PbSO}_4$  by multiplying by the factor 0.938. Subtract this value from the total  $\text{PbSO}_4$  found above and report the remainder as lead compounds other than chromate, calculated as  $\text{PbSO}_4$ .

(e) CALCIUM.—Ignite 2 g of the pigment and dissolve the residue in hydrochloric acid as in 4(c). Then, without filtering from the insoluble matter, transfer to a 500 cc volumetric flask, saturate with hydrogen sulphide, make alkaline with ammonia, fill to the mark, mix, and filter through a dry paper, discarding the first 20 cc. Then determine the calcium in 250 cc of the filtrate (corresponding to 1 g pigment) by precipitation as oxalate and weighing as calcium oxide. The calcium oxide should weigh not more than 0.005 g.

(f) COLOR.—Add the percentages of matter soluble in water 4(b), barium sulphate and siliceous material 4(c), lead compounds other than chromate calculated as  $\text{PbSO}_4$  4(d), and calcium oxide 4(e) and subtract the sum from 100. Call the difference the percentage of color.

## 5. 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 should be dry and free from streaks.

(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. Material complying with the specification should yield less than 0.5 cc.

(f) VOLATILE THINNER.—Follow the procedure outlined in 3(e). Correct the result for any water found (see 5(e)) and report the remainder as volatile thinner.

(g) PERCENTAGE OF PIGMENT.—Follow the procedure outlined in 3(f).

(h) TESTING NONVOLATILE VEHICLE.—Follow the procedure outlined in 3(g), 3(h), and 3(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 3(j).

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

## 6. REAGENTS

(a) EXTRACTION MIXTURE.—

- 5 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 crystallized sodium thiosulphate to 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 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 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 1000 cc.

(f) HANUS SOLUTION.—Dissolve 13.2 g of iodine in 1000 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.

(g) 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, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g to 1000 cc), kept at about 50° C for 15 days, and then distilled.

(h) 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  $\text{H}_2\text{SO}_4$  has been added, and then dilute to 1000 cc. This solution should be freshly standardized when needed, as it does not keep well.

(i) STANDARD POTASSIUM DICHROMATE SOLUTION.—Dissolve 4.903 g of pure dry crystallized potassium dichromate in water and dilute to 1000 cc. One cubic centimeter of this solution corresponds to 0.0108 g  $\text{PbCrO}_4$ , or 0.0101 g  $\text{PbSO}_4$ .

(j) POTASSIUM FERRICYANIDE SOLUTION.—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.

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