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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR OUTSIDE WHITE TITANIUM-ZINC PAINT, SEMIPASTE AND READY-MIXED

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 278

This specification was officially promulgated by the Federal Specifications Board on May 8, 1925, for the use of the Departments and Independent Establishments of the Government in the purchase of outside white titaniumzinc paint, semipaste and ready-mixed.

CONTENTS

	~ ~ ~ ~ ~ ~
I. Classes	1
II. Material	2
III. General requirements	2
IV. Detail requirements	2
1. Pigment	2
2. Liquid	2
3. Semipaste	2
4. Ready-mixed paint	3
V. Methods for sampling and testing	3
1. Sampling	3
2. Laboratory examination, semipaste	4
3. Analysis of pigment	6
4. Laboratory examination, ready-mixed paint	8
5. Reagents	10
VI. Packing and marking	12
VII. Additional information	12
VIII. General specifications	12

I. CLASSES

Outside white titanium-zinc paint shall be of the following classes: Semipaste in linseed oil and ready-mixed.

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II. MATERIAL

No details specified.

III. GENERAL REQUIREMENTS

No details specified.

IV. DETAIL REQUIREMENTS

I. PIGMENT

The pigment shall be composed of:

Ingredients	Maximum	Minimum
Titanium pigment Zinc oxide	Per cent	Per cent 50
Extending pigments Matter soluble in water	10 0.8	
Lead Sulphide sulphur	None. None.	

In no case shall the sum of the titanium pigment and zinc oxide be less than 90 per cent. The titanium pigment shall contain 25 per cent titanium oxide, the remainder to be blanc fixe (precipitated barium sulphate).

2. LIQUID

The liquid in semipaste paint shall be entirely linseed oil; in ready-mixed paint it shall contain not less than 85 per cent linseed oil, the remainder to be combined drier and thinner. The drier shall be free from lead. 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 those of a sample mutually agreed upon by buyer and seller. The weight per gallon shall be not less than $17\frac{1}{4}$ pounds. The semipaste shall consist of:

Ingredients	Maximum	Minimum
Pigment	Per cent 75 30 None. 0. 7 2. 0	Per cent 70 25

4. 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 dried film shall be resistant to sulphide fumes. 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 14 pounds. The paint shall consist of:

Ingredients	Maximum	Minimum
Pigment Liquid (containing at least 85 per cent linseed oil) Lead in any form. Water Coarse particles and "skins" (total residue retained on No. 325 sieve based on pigment)	Per cent 62 42 None. 0.5 2.0	Per cent 58 38

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 specifications.

I. SAMPLING

It is mutually agreed by buyer and seller that a singlé 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.—Place some of the paste 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.

(e) MOISTURE AND OTHER VOLATILE MATTER.—Weigh accurately from 3 to 5 g of the paste in a tarred flat-bottomed dish about 8 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 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 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 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, wash once with water, then add 50 cc of water and 50 cc of ether. Shake very gently with a whirling action to disolve 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 CaCl, 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. 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 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 onehalf 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.—Make qualitative analysis following ordinary methods. The pigment should show negative test for sulphide sulphur and appreciable water-soluble matter; ash a sample of the semipaste or ready-mixed paint and test for lead.

(b) MATTER SOLUBLE IN WATER.—Transfer 2.5 g of the pigment to a graduated 250 cc flash, add 100 cc of water, boil for five minutes, cool, fill to the mark with water, mix, and allow to settle. Pour the supernatant liquid through a dry filter 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.

(c) TITANIUM OXIDE.—Weigh accurately about 1 g of the pigment, transfer to a 250 cc Pyrex beaker, add 25 cc of concentrated sulphuric acid and 8 g of ammonium sulphate. Mix well and heat on a hot plate until fumes of sulphuric acid are evolved, and then continue the heating over a strong flame until solution is complete (usually not over five minutes of boiling) or it is apparent that the residue is composed of silica or silicaous matter. Caution should be observed in handling this hot acid solution. Cool the solution, dilute with 100 cc of water, stir, heat carefully to boiling while stirring, settle, filter through paper and transfer the precipitate completely to the paper. Wash the insoluble residue with cold 5 per cent (by volume) sulphuric acid until titanium is removed.

Dilute the filtrate to 200 cc and add about 30 cc of ammonia, specific gravity 0.90, to lower the acidity to approximately 5 per cent sulphuric acid (by volume).

Wash out a Jones reductor ¹ with dilute 5 per cent (by volume) sulphuric acid and water, leaving sufficient water in the reductor to fill to the upper level of the zinc. (These washings should require not more than one or two drops of 0.1 N potassium permanganate solution to obtain the pink color.) Empty the receiver, and put in it 25 cc (measured in a graduate) of ferric sulphate solution. (See Reagents.) Reduce the prepared titanium solution as follows: (1) Run 50 cc of the 5 per cent sulphuric acid solution through the reductor at a speed of about 100 cc per minute, (2) follow this with the titanium solution, (3) wash out with 100 cc of 5 per cent sulphuric acid, (4) finally run through about 100 cc of water.

Care should be observed that the reductor is always filled with solution or water to the upper level of the zinc.

Gradually release the suction, wash thoroughly the glass tube that was immersed in the ferric sulphate solution, remove the receiver, and titrate immediaely with 0.1 N potassium permanganate solution. (See Reagents.)

1cc 0.1 N KMn0₄=0.00481 g Ti=0.00801 g TiO₂

Run a blank determination, using the same reagents, washing the reducer as in the above determination. Subtract this permanganate reading from the original reading and calculate the final reading to titanium dioxide (TiO_2) (which will include iron, chromium, arsenic, and any other subtance which is reduced by zinc and acid).

¹Directions for preparing a Jones reductor may be found in Blair, The Chemical Analysis of Iron, 8th ed., Lippincott & Co., or Treadwell-Hall, Analytical Chemistry, 5th ed., J. Wiley & Sons, p. 368.

(d) BARIUM SULPHATE.—Ignite and weigh the insoluble matter obtained in separating the titanium (see V, 3, (c)). Mix the ignited residue with about 10 times its weight of anhydrous sodium carbonate (grind the mixture in an agate mortar if necessary), fuse the mixture in a covered platinum crucible, heating about one hour. Let cool, place the 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 the crucible and cover in the 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 the 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 BaSO₄. Subtract the result from the original determination to obtain the siliceous material.

(e) ZINC ONDE.—Weigh accurately about 0.5 g of the pigment, transfer to a 400 cc beaker, add 30 cc of hydrochloric acid (1:2), boil for two or three minutes, add 200 cc of water and a small piece of litmus paper; add strong ammonia until slightly alkaline, render just 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.

(f) CALCULATIONS.—In case the percentage of barium sulphate (V, 3, (d)) is not more than 3.17 times as great as the percentage of titanium oxide (V, 3, (c)), add the two together and call the sum the percentage of titanium pigment. If the percentage of barium sulphate is greater than this amount, take 3.17 times the percentage of titanium oxide as the percentage of barium sulphate to be included in the percentage of titanium pigment and include the remainder in the percentage of extending pigments. Subtract the sum of the percentages of titanium pigment, zinc oxide (V, 3, (e)), and matter soluble in water (V, 3, (b)) from 100. Call the remainder percentage of extending 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 wellmixed paint on a clean tin plate panel. 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) RESISTANCE TO SULPHIDE FUMES.—Apply a sufficient number of coats of the paint to two glass plates to completely hide the surface, and expose one of the plates in a saturated atmosphere of hydrogen sulphide for 18 hours. Compare the color with the unexposed plate. The exposed plate should be practically no darker than the unexposed one. There shall be no greater difference in the color of the two plates than there will be with similar plates coated with a paint made with titanium pigment, lead free zinc oxide, and raw or refined linseed oil with sufficient cobalt added for drying.

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

(f) WATER.—Mix 100 g of the paint in a 250 or 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.

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

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

(i) 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.

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

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

5. REAGENTS

(a) URANYL INDICATOR.—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 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.² 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 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.

² Treadwell-Hall, Analytical Chemistry, 2, 6th ed., p. 551.

(\hbar) 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 SOLUM 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.

(i) 0.1 N POTASSIUM PERMANGANATE Solution.—Dissolve 3.161 g of pure potassium permanganate in a liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: In a 400 cc beaker dissolve 0.25 to 0.30 g of Bureau of Standards sodium oxalate in 250 cc of hot water (80 to 90° C.) and add 15 cc of dilute sulphuric acid (1:1). Titrate at once with the potassium permanganate solution, stirring the liquid vigorously and continuously. The permanganate must not be added more rapidly than 10 to 15 cc per minute, and the last 0.5 to 1 cc must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The solution should not be below 60° C. by the time the end point is reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small asbestos covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.8334 gives its iron equivalent, or multiplied by 1.1954 gives its titanium dioxide (TiO₂) equivalent.³

The permanganate solution should be kept in a glass-stoppered bottle painted black to keep out light.

(k) FERRIC SULPHATE SOLUTION.—A solution containing 2 per cent of iron as ferric sulphate is desired and may be prepared as follows: Dissolve 20 g of pure iron or plain carbon steel in a slight excess of hydrochloric acid, oxidize with nitric acid, heat with about 80 cc of sulphuric acid until fumes are evolved, finally cool, and dilute to 1,000 cc set on steam bath, until dissolved, and filter if necessary. Add 0.1 N permanganate solution until a faint pink color shows that any ferrous iron has been oxidized. Ferric ammonium sulphate may also be used.⁴

⁸ International Atomic Weights, 1925.

⁴ Gooch, Methods in Chemical Analysis, 1st ed., p. 426.

VI. PACKING AND MARKING

No details specified.

VII. ADDITIONAL INFORMATION

This specification covers the requirements for a high-grade white paint for outside and general use, intended particularly to be used wherever excessive amounts of sulphide fumes would quickly discolor the average outside white paint. For marine use, the addition of 1 pint of water-resisting spar varnish to each gallon of paint, just before application, is recommended. It may be ordered either in the form of semipaste pigment ground in linseed oil or of readymixed paint, and the purchaser shall state which is desired.

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

VIII. GENERAL SPECIFICATIONS

No details specified.

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