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No. 67.

DEPARTMENT OF COMMERCE.

BUREAU OF STANDARDS.
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(Issued September 19, 1923.)

UNITED STATES GOVERNMENT SPECIFICATION FOR
GLOSS INTERIOR LITHOPONE PAINT, WHITE AND
LIGHT TINTS.

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION NO. 67.

This specification was officially adopted by the Federal Specifications Board on September 1, 1923, for the use of the Departments and Independent Establishments of the Government in the purchase of gloss interior lithopone paint, white and light tints.

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1. GENERAL.

This specification covers ready-mixed lithopone paints, frequently known as gloss mill white, in white and a variety of light tints. Paints under this specification are not intended for outside exposure. They shall dry to gloss opaque coats that will adhere well to wood, metal, and plaster, stand washing with soap and water, and show no material change in color on exposure to light or material yellowing when kept in the dark.

The paint shall be purchased by volume (231 cubic inches to the gallon).

(a) PIGMENT.—The pigment shall consist of :

	Maximum.	Minimum.
	Per cent.	Per cent.
Lithopone ¹		65
Zinc oxide.....		20
Tinting and extending pigments.....	5.0	
Material soluble in water.....	0.8	

¹ The lithopone used must contain not less than 26 per cent of zinc sulphide and must not darken on exposure.

In no case shall the sum of zinc oxide and lithopone be less than 95 per cent.

(b) LIQUID.—The liquid portion of the paint shall consist of treated drying oils or varnish, or a mixture thereof, and turpentine or volatile mineral spirits, or a mixture thereof, in such proportions as to insure not less than 60 per cent of nonvolatile vehicle. The nonvolatile vehicle shall dry to a tough and elastic film.

(c) PAINT.—The paint shall be well ground, shall not settle badly, cake, or thicken in the container, shall be readily broken up with a paddle to a smooth, uniform paint of brushing consistency, and shall dry within 24 hours to a varnish gloss finish without streaking, running, or sagging, and free from laps and brush marks. The color and hiding power when specified shall be equal to those of a sample mutually agreed upon by buyer and seller. After drying for not less than five days marks made on the painted surface with a soft lead pencil (No. 2 Mogul) shall be easily removed by washing with soap and warm water without appreciably marring the paint surface. The weight per gallon shall be not less than 12½ pounds. The paint shall consist of:

	Maximum.	Minimum.
	Per cent.	Per cent.
Pigment.....	60	50
Liquid (containing at least 60 per cent nonvolatile matter).....	50	40
Water.....	1	
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment).....	0.5	

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.

2. 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. 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.

3. LABORATORY EXAMINATION.

(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 paint must be no more difficult to mix to a uniform consistency than a good grade of gloss mill white. The paint 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 the thoroughly mixed paint shall be placed in a clean container and 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.**—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, TIME OF DRYING, AND RESISTANCE TO WASHING.**—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 it stand for 24 hours. The paint should be dry and free from streaks. Let the panel stand for five days, then make marks on it with a soft lead pencil (No. 2 Mogul) and wash these marks off with warm (75° C.) distilled water and white floating soap, using

a sponge or soft rag. The marks must be removed by this treatment without appreciably marring the paint film.

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) **FASTNESS TO LIGHT.**—Apply a sufficient number of coats of the paint to a ground-glass plate to completely hide the surface, cover half of this painted surface with opaque black paper, and expose indoors in a well-lighted room for five days. Remove the black paper and examine the surface. The exposed portion should be no darker than the portion protected by the black paper.

(f) **YELLOWING.**—Apply a sufficient number of coats of the paint to two ground-glass plates to completely hide the surface; after applying the last coat let dry in a well-lighted room for five days. Place one of the plates in a dark room or cabinet¹ with a warm, very humid atmosphere, for 96 hours. Remove from cabinet and compare with the plate that has been kept in a light room. The plate kept in the dark shall be only slightly yellower than the plate kept in the light. There shall be no greater difference in color of the two plates than there will be with similar plates coated with the best grade of paint of this general nature.

(g) **WATER.**—Mix 100 g of the paint in a 300 cc flask with 75 cc of toluol. Connect with a condenser and distil 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.

(h) **VOLATILE THINNER.**—Weigh accurately from 3 to 5 g of the paint into a tared flat-bottomed dish about 5 cm in diameter, spreading the paint over the bottom. Heat at 105 to 110° C. for one hour, cool, and weigh. Calculate the loss in weight as percentage of water and volatile thinner, subtract from this the percentage of water (3, g), and report the remainder as volatile thinner.

(i) **PERCENTAGE OF PIGMENT.**—Weigh accurately about 15 g of the paint into a weighed centrifuge tube. Add 20 to 30 cc of "extraction mixture" (see Reagents), mix thoroughly with a

¹ A convenient cabinet for this test is described in Circular No. 152, Educational Bureau, Scientific Section, Paint Manufacturers' Association of the United States.

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 sieve to remove any skins, and preserve in a stoppered bottle. Preserve the extracted vehicle for 3 (*k*).

(*j*) PERCENTAGE OF NONVOLATILE VEHICLE.—Add together the percentages of water (3, *g*), of volatile thinner (3, *h*), and of pigment (3, *i*), and subtract the sum from 100. The remainder is the percentage of nonvolatile vehicle.

(*k*) NATURE OF NONVOLATILE VEHICLE.—Evaporate the extracted vehicle and extraction mixture from 3 (*i*) to about 5 cc. Thoroughly clean with benzol a piece of bright sheet iron, tin plate, or terneplate. Spread a portion of the concentrated extracted vehicle on the sheet of metal, allow to dry for 30 minutes at room temperature in a vertical position, bake for three hours at 100 to 110° C. (212 to 221° F.), remove from the oven, and keep at room temperature for three days. Place the panel with the coated side uppermost over a 3 mm ($\frac{1}{8}$ -inch) rod, held firmly by suitable supports, at a point equally distant from the top and bottom edges of the panel and bend double rapidly. The dried vehicle must show no cracking whatever at the point of bending. Test the film with a knife blade at a place not less than 2.5 cm (1 inch) from the edge. The film should be tough and elastic. If it powders, or if particles fly under the test, it will be considered brittle, which will be cause for rejection. The film must also stand light, vigorous rubbing with the finger without powdering or disintegrating.

(*l*) 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 paint containing 10 g of pigment (see 3, *i*), add 50 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 for one hour at 105 to 110° C., cool, and weigh.

4. ANALYSIS OF PIGMENT.

Use the pigment extracted in 3 (i).

(a) *QUALITATIVE ANALYSIS.—Make qualitative analysis, following ordinary methods.

(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 five minutes, cool, fill to 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) BARIUM SULPHATE AND SILICEOUS MATERIAL.—Transfer 1 g of pigment to a porcelain casserole or dish, moisten with a few drops of alcohol, add 40 cc of hydrochloric acid (1.1 specific gravity), cover, and boil to expel hydrogen sulphide. Remove the cover and evaporate to dryness on the steam bath, moisten with hydrochloric acid, dilute with water, filter through paper, and wash with dilute hydrochloric acid and then with hot water until the washings are free from zinc and chlorine. 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 (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 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 BaSO_4 . Subtract from the result of the previous determination to obtain the siliceous material.

(d) TOTAL ZINC CALCULATED AS ZINC OXIDE.—With material containing no interfering elements (iron, for example) weigh accurately about 1 g of pigment, transfer to a 400 cc beaker, moisten with alcohol, add 30 cc of hydrochloric acid (1:2), boil for two to 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.

When iron or other interfering elements are present (see 4, a), take the filtrate containing the zinc from 4 (c), add a slight excess of bromine water and 2 g of ammonium chloride, heat to nearly boiling, add an excess of ammonia, heat for about two minutes, filter, dissolve the precipitate in hydrochloric acid, add 2 g of ammonium chloride, and reprecipitate with ammonia as above. Filter, wash the precipitate with hot 2 per cent ammonium-chloride solution, unite the two filtrates, and determine zinc as above.

(e) ZINC OXIDE.—Weigh accurately 2.5 g of pigment, transfer to a 250 cc graduated flask, moisten with a few drops of alcohol, add about 200 cc of 1 to 3 per cent acetic acid, shake vigorously, and let stand for 30 minutes, shaking once very five minutes. Fill to the mark with 1 to 3 per cent acetic acid, mix, filter through a dry paper, discard the first 25 cc, and determine zinc in 100 cc of the filtrate (corresponding to 1 g) as in 4 (d). Calculate the percentage of zinc oxide.

(f) CALCULATIONS.—Subtract the percentage of zinc oxide (4, e) from the percentage of total zinc as zinc oxide (4, d) and multiply the remainder by 1.2 to convert to percentage of zinc sulphide. In case the percentage of barium sulphate (4, c) is not more than 2.86 times as great as the percentage of zinc sulphide, add the two together and call the sum the percentage of lithopone. If the percentage of barium sulphate is greater than this amount, take 2.86 times the percentage of zinc sulphide as the percentage of barium sulphate to be included in the percentage of lithopone and include the remainder in the percentage of tinting and extending pigments. Subtract the sum of the percentages of zinc oxide

(4, *e*), lithopone, and matter soluble in water (4, *b*) from 100. Call the remainder percentage of tinting and extending pigments.

5. REAGENTS.

(a) EXTRACTION MIXTURE.—

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

(b) ONE TO THREE PER CENT ACETIC ACID.—Dilute 20 cc of glacial acetic acid to 1,000 cc with distilled water.

(c) 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.

(d) 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. The amount of ferrocyanide solution required for the blank should be subtracted from the amount used in standardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtain when the sample is titrated.

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