U. S. Gov't
Standard
Specification,
No. 66.

DEPARTMENT OF COMMERCE.

BUREAU OF STANDARDS.

George K. Burgess, Director.

CIRCULAR OF THE BUREAU OF STANDARDS, NO. 146.

[Issued September 25, 1923.]

UNITED STATES GOVERNMENT SPECIFICATION FOR WATER-RESISTING RED ENAMEL.

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION NO. 66.

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 water-resisting red enamel.

CONTENTS.

1. GENERAL.

The material desired under this specification is an extremely durable, highest quality red enamel, suitable primarily for outside use. It should be made by grinding pure high color strength toluidine red toner (metanitro-paratoluidine-azo-betanaphthol), free from any base or substratum, with the very best water-resisting long oil spar varnish. The color and hiding power when specified shall be equal to those of a sample mutually agreed upon by buyer and seller. It must meet the following requirements:

WEIGHT PER GALLON.—Not less than 7½ pounds.

PIGMENT.—Not less than 6 per cent by weight; pigment to be composed entirely of pure high color strength toluidine red toner, free from any other organic coloring matter, base, or substratum.

61692°—23

Coarse Particles and "Skins" (total residue retained on No. 325 sieve).—Not more than 0.5 per cent.

Nonvolatile Matter.—Not less than 60 per cent by weight.

Set to Touch.—In not more than 18 hours.

Dry Hard and Tough.—In not more than 48 hours.

Working Properties.—Enamel must have good brushing, flowing, covering, and leveling properties and must not cake in the container.

Water Resistance.—Dried film must withstand cold water for 18 hours and boiling water for 15 minutes without whitening, dulling, or change in color.

Toughness.—Enamel must pass a 50 per cent Kauri reduction test at 24° C. (75° F.).

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 be taken as representative of the whole. Whenever possible, an original unopened container shall be sent to the laboratory, and when for any reason this is not done the inspector shall thoroughly mix the contents of the container sampled, transfer not less than 1 quart to a clean, dry glass bottle or tin can, which must be nearly filled with the sample, securely stoppered with a new, clean cork or well-fitting cover or cap, sealed and distinctly labeled by the inspector. The inspector should take a duplicate from the container sampled to be held for check in case of dispute, and, when requested, should take a sample for the seller.

3. LABORATORY EXAMINATION.

The tin panels used in the following tests shall all be cut from bright tin plate weighing not more than 25 nor less than 19 g per square decimeter (0.51 to 0.39 pound per square foot). (Commercial No. 31 gauge bright tin plate should weigh about 0.44 pound per square foot. It is important that the tin plate used shall be within the limits set.) The panels shall be about 7.5 by 13 cm (3 by 5 inches) and must be thoroughly cleaned with benzol immediately before using.

(*a*) Caking in Container and Working Properties.—When an original package is received in the laboratory, it shall be

weighed, opened, and stirred with a stiff spatula or paddle. The enamel must be no more difficult to break up than a normal good grade of enamel paint. The enamel 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. Apply some of the thoroughly mixed enamel, both by brushing and flowing, to clean glass plates. It should work easily under the brush. Dry both plates in a nearly vertical position. They should both dry without streaking, separating, or showing brush marks. A portion of thoroughly mixed enamel shall be placed in a clean container and the portions for the remaining tests promptly weighed out.

(*b*) COLOR AND HIDING POWER.—Place some of the enamel 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 by transmitted and reflected light.

(*c*) WEIGHT PER GALLON.—Weigh a clean, dry, 100 cc graduated flask. Fill to the mark with the thoroughly mixed enamel 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*) COARSE PARTICLES AND SKINS.—Dry in an oven at 105 to 110° C. a No. 325 sieve, cool, and weigh accurately. Weigh accurately about 50 g of the enamel, 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 for one hour at 105 to 110° C., cool, and weigh.

(*e*) PIGMENT.—*Qualitative examination.*—Pour about 1 g of the thoroughly stirred enamel, previously strained through a No. 200 sieve in a 50 cc beaker. Add about 40 cc of chloroform (U. S. P.) and warm on the steam bath, stirring with a glass rod. A clear orange-red solution should result in a few minutes. Take another portion of the enamel and spread it with a spatula on a smooth, white surface, such as a piece of milk glass. Touch a few drops of alcoholic sodium hydroxide solution to the center of the film and rub well with a glass spatula. There should be no change in color.[1]

[1] The presence of para nitraniline red is indicated by a violet color.

Quantitative determination.—Weigh 1 g (± 10 mg) of the enamel and 6 g (± 10 mg) of pure zinc oxide, place on a large glass plate, add 2 cc of linseed oil and rub up with a flat-bottomed glass pestle or muller, grinding with a circular motion 50 times. Gather up with a sharp-edge spatula and grind out twice more in like manner, giving the pestle a uniform pressure. Next weigh to ± 1 mg an amount of pure high color strength toluidine red toner equal to 6 per cent of the weight of enamel taken, add 4 drops of linseed oil and rub up with the glass pestle. Then add 6 g of pure zinc oxide and 2 cc of linseed oil and treat in exactly the same manner as described above. Transfer portions of each paste to a clean microscope slide quite close together, and then draw a palette knife across both samples, so as to make them meet in a line. Compare the tints as shown on both sides of the glass. The color of the sample tested shall be not less than that of the selected standard, and the tone shall be not materially different from it.

(*f*) Nonvolatile Matter.—Place a portion of the sample in a stoppered bottle or weighing pipette. Weigh container and sample. Transfer about 1.5 g of the sample to a weighed flat-bottomed metal dish about 8 cm in diameter (a friction-top can plug). Weigh container again and by difference calculate the exact weight of the portion of sample transferred to the weighed dish. Heat dish and contents in an oven maintained at 105 to 110° C. (221 to 230° F.) for three hours. Cool and weigh. From the weight of the residue left in the dish and weight of the sample taken calculate the percentage of nonvolatile residue.

(*g*) Drying Time.—Pour the enamel on one of the tin panels described above. Place the panel in a nearly vertical position in a well-ventilated room, but not in the direct rays of the sun. The atmosphere of this room must be free from products of combustion or laboratory fumes. The temperature of the room should be from 21 to 32° C. (70 to 90° F.). The film is tested at points not less than 2.5 cm (1 inch) from the edges of the film by touching lightly with the finger. The enamel is considered to have set to touch when gentle pressure of the finger shows a tacky condition, but none of the enamel adheres to the finger. The enamel is considered to have dried hard when the pressure that can be exerted between the thumb and finger does not move the film or leave a mark which remains noticeable after the spot is lightly polished. If rapid, light rubbing breaks the surface, the sample is considered not to have satisfactorily dried hard. In case the test shows time of setting to touch or drying hard more than 18 and 48 hours,

respectively, two additional tests shall be run on different days, and if the enamel does not meet the above drying and hardening requirements on both of these additional tests it shall be considered unsatisfactory. In cases where different laboratories fail to agree on the drying test, due to different atmospheric conditions, and umpire tests are necessary, such tests shall be made in a well-ventilated room maintained at a temperature of 70° F. and relative humidity of 65 per cent saturation.

(*h*) WATER RESISTANCE.—Pour the enamel on two of the tin panels described above and allow to dry under the conditions described in paragraph (*g*) for 48 hours. Place one of these panels in a beaker containing about 2.5 inches of distilled water at room temperature (immersing the end of the panel which was uppermost during the drying period) and leave in water for 18 hours. The enamel shall show no whitening and no more than very slight dulling either when observed immediately after removing from the water or after drying for 2 hours. Place the other panel in a beaker containing about 2.5 inches of boiling distilled water (immersing the end of the panel which was uppermost during the drying period) and allow to remain in the boiling water for 15 minutes. The enamel shall show no whitening, no more than a very slight dulling, and no material change in color, either when observed immediately after removing from the water or after drying for 2 hours.

(*i*) TOUGHNESS.—The toughness of the enamel is determined by the Kauri reduction test, as follows: By proportionately reducing its toughness by the addition of a standard solution of "run-Kauri" gum in pure spirits of turpentine.

(1) *Preparation of the "run Kauri."*—Arrange a distillation flask, water-cooled condenser, and a tared receiver on a balance. Place in the flask about one-third of its volumetric capacity of clear, bright hard pieces of Kauri gum broken to pea size. Carefully melt and distil until 25 per cent, by weight, of the gum taken is collected in the tared receiver. (At the end of the distillation the thermometer in the distillation flask with the bulb at the level of the discharging point of the flask should register about 316° C. (600° F.).) Pour the residue into a clean pan, and when cold break up into small pieces.

(2) *Preparation of standard "run-Kauri" solution.*—Place a quantity of the small broken pieces of run-Kauri, together with twice its weight of freshly redistilled spirits of turpentine, using only that portion distilling over between 153 and 170° C. (308 and 338° F.) in a carefully tared beaker. Dissolve by heating

to a temperature of about 149° C. (300° F.) and bring back to correct weight when cold by the addition of the amount of redistilled spirits of turpentine necessary to replace the loss by evaporation during the dissolving of the gum.

(3) *Reduction of the enamel.*—Having carefully determined the nonvolatile content of the enamel according to the method under paragraph (*f*) of this specification, take 100 g of the enamel and add to it an amount of the standard run-Kauri solution equivalent to 50 per cent, by weight, of the nonvolatile matter in the enamel. Mix the enamel and the solution thoroughly.

(4) *Application of the enamel.*—Flow a coat of the enamel thus reduced on one of the tin panels described above and let stand in a nearly vertical position at room temperature for one hour. Next place the panel in a horizontal position in a properly ventilated oven and bake for five hours at 95 to 100° C. Remove the panel from the oven and allow to cool at room temperature, preferably 24° C. (75° F.) for one hour.

(5) *Bending the panel.*—Place the panel with the enameled side uppermost over a 3-mm (⅛ inch) rod, held firmly by suitable supports, at a point equally distant from the top and bottom edges of the panel and bend the panel double rapidly. The enamel must show no cracking whatsoever at the point of bending. For accurate results the bending of the panel should always be done at 24° C. (75° F.), for a lowering of the temperature will lower the percentage of reduction that the enamel will stand without cracking, while an increase in the temperature increases the percentage of reduction that the enamel will stand.

4. BASIS OF PURCHASE.

Enamel shall be purchased by volume, the unit being a gallon of 231 cubic inches at 15.5° C. (60° F.). The volume may be determined by measure or, in case of large deliveries, it may be easier to determine the net weight and specific gravity at 15.5/15.5° C. (60/60° F.) of the delivery. The weight per gallon in pounds can then be determined by multiplying the specific gravity by 8.33. The net weight in pounds divided by the weight per gallon gives the number of gallons.

ADDITIONAL COPIES
OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.
AT
5 CENTS PER COPY