DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

S. W. STRATTON, Director

CIRCULAR OF THE BUREAU OF STANDARDS

No. 104

[OCTOBER 18, 1920]

RECOMMENDED SPECIFICATION FOR ASPHALT VARNISH

PREPARED AND RECOMMENDED BY THE U. S. INTERDEPARTMENTAL COMMITTEE ON PAINT SPECIFICATION STANDARDIZATION, SEPTEMBER 27, 1920; P. H. WALKER, BUREAU OF STANDARDS, CHAIRMAN; J. W. GINDER, TREASURY DEPARTMENT, SECRETARY

[This committee was appointed at the suggestion of the Secretary of Commerce, and consisted of representatives of the War, Navy, Agriculture, Interior, Post Office, Treasury, and Commerce Departments, the Panama Canal, and the Educational Bureau of the Paint Manufacturers' Association of the United States. The committee submitted a preliminary draft of the specification to a large number of representatives of the paint and varnish manufacturers, and gave careful consideration to the replies received.]

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1. GENERAL

This varnish shall be composed of a high grade of asphalt fluxed and blended with properly treated drying oil and thinned to the proper consistency with a volatile solvent. It must be resistant to air, light, lubricating oil, water, and mineral acids of the concentration hereinafter specified, and must meet the following requirements:

APPEARANCE.—Smooth and homogeneous; no livering or stringiness.

COLOR.—Jet black.

FLASH POINT (CLOSED-CUP).—Not below 30° C (86° F).

ACTION WITH LINSEED OIL.—Varnish must mix freely to a homogeneous mixture with an equal volume of raw linseed oil.

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Insoluble in Carbon Bisulphide.—Not more than 1 per cent.

Nonvolatile Matter.—Not less than 40 per cent by weight.

Fatty Matter.—Not less than 20 per cent of the nonvolatile. Must be liquid and not show any rosin by the Liebermann-Storch test.

Set to Touch.—Within 5 hours.

Dry Hard and Tough.—Within 24 hours.

Toughness.—Film on metal must withstand rapid bending over a rod 3 mm (⅛ inch) in diameter.

Working Properties.—Varnish must have good brushing, flowing, covering, and leveling properties.

Resistance to Water.—Dried film must withstand cold water for 18 hours.

Resistance to Oil.—Dried film must withstand lubricating oil for 6 hours.

Resistance to Mineral Acids.—Dried film must withstand action of the following acids for 6 hours: Sulphuric acid, specific gravity 1.3 (about 40 per cent). Nitric acid, specific gravity 1.22 (about 35 per cent). Hydrochloric acid, specific gravity 1.09 (about 18 per cent).

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. When for any reason this can not be done, the inspector shall select a package and thoroughly mix its contents. He shall fill a 1 quart, clean, dry container from this package, securely stopper it with a new clean cork or well-fitting cover or cap, seal, and distinctly label it.

The inspector shall take a duplicate from the container sampled to be held for check in case of dispute, and, when requested, shall take a sample for the seller.

3. LABORATORY EXAMINATION

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:

(*a*) Appearance and Color.—Pour some of the thoroughly mixed sample on a clean, clear glass plate and stand in a vertical position until the excess varnish has drained off. Examine by

transmitted light. The varnish must be smooth and homogeneous and must not show any separation or segregation of the constituents. Examine by reflected light. The film must be jet black in color.

(*b*) FLASH POINT.—Determine with either the "Tag" or Elliott closed-cup tester. The former is preferred and directions for its use are found in A. S. T. M. Standards D 56–19. For the method of determining with the Elliott cup see Proceedings A. S. T. M., 1917, part 1, p. 414.

(*c*) ACTION WITH LINSEED OIL.—Pour 10 cc of the varnish into a test tube and add an equal volume of raw linseed oil conforming to Bureau of Standards Circular No. 82. Stopper the test tube and shake vigorously for several minutes. Then pour some of the mixture on a clear glass plate and stand in a vertical position. After the excess varnish has drained off, examine by transmitted light. There shall be no separation of the oil and varnish.

(*d*) INSOLUBLE IN CARBON BISULPHIDE.—Weigh about 5 g of the varnish into a small beaker, add 25 cc of carbon bisulphide, and allow to stand for 15 minutes. Filter through a weighed Gooch crucible, prepared with a medium thick mat of asbestos, using suction if necessary to aid in filtration. Wash the residue in the crucible with carbon bisulphide until the washings are colorless. Dry in air at room temperature until the odor of carbon bisulphide has almost disappeared, and then for one hour in an oven at 110° C. Cool and weigh. From the weight of the insoluble left in the crucible and the weight of sample taken, calculate the percentage of insoluble in carbon bisulphide.

(*e*) NONVOLATILE MATTER.—Place a portion of the sample in a stoppered bottle or weighing pipette. Weigh the 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 the container again and by difference calculate the exact weight of the portion of sample tranferred to the weighed dish. Heat the dish with its contents in an oven maintained at 105 to 110° C. for three hours. Cool and weigh. From the weight of the residue left in the dish and the weight of the sample taken, calculate the percentage of nonvolatile residue.

(*f*) FATTY MATTER.—Weigh about 5 g of the varnish into a wide-mouthed flask, add 50 cc of benzol, and heat under a reflux condenser on a steam bath until the varnish is entirely dissolved. Add 25 cc of a 0.5 *N* alcoholic caustic soda solution and continue

boiling under the reflux condenser for one-half hour. Remove the condenser and evaporate the solution to dryness.

Add to the residue in the flask 50 cc of distilled water and heat until the residue is disintegrated. Filter the water solution of the soaps. Repeat this operation with 25 cc portions of water until the residue is completely disintegrated and the wash water is clear and colorless.

Combine the filtrates (the soap solution and washings), acidify with hydrochloric acid, and heat until the fatty acids and any emulsified asphalt separate and rise to the top, and the water below is clear.

Cool, transfer to a separatory funnel, and extract twice with 50 cc portions of ether. Combine the ether extracts and wash with water until free from acid. Filter the ether extracts through paper into a beaker and wash the residue on the paper with ether until the washing runs through colorless. Evaporate the ether solutions to dryness.

Add 25 cc of 95 per cent ethyl alcohol to the residue in the beaker and warm on the steam bath. Cool to room temperature and filter through paper into a tared flask or dish. Repeat this operation with 10 cc portions of 95 per cent ethyl alcohol until the alcohol remains colorless. Finally wash the residue on the paper with 95 per cent ethyl alcohol until the washings run through colorless.

Evaporate to dryness on a steam bath and heat for an hour in an oven at 105° C (221° F). Cool and weigh. From the weight of the residue in the flask and the weight of the original sample calculate the percentage of fatty matter.

(Sometimes the residue obtained after saponification and the evaporation of the benzol and alcohol from the saponifying mixture is not completely disintegrated by boiling with water. In that case, extract with water until nothing further dissolves and then dry. Dissolve in benzol, using heat if necessary, and wash the benzol solution several times with water. Heat the washings until the odor of benzol has disappeared and add to the soap solution before acidifying.)

The fatty matter obtained above must be a clear amber and liquid.

A fugitive violet color shall not be obtained when the fatty matter is subjected to the following tests: Dissolve a small amount of the fatty matter in 5 cc of acetic anhydride, warming if

necessary to aid solution. Cool, draw off the acetic anhydride solution, and add a drop of sulphuric acid, 1.53 specific gravity.

(*g*) Drying Time.—Pour the varnish on a clean glass plate not less than 15 cm (6 inches) long and 10 cm (4 inches) wide. Place the plate in a nearly vertical position in a well-ventilated room, but not in the direct rays of the sun. 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 varnish is considered to have set to touch when gentle pressure of the finger shows a tacky condition, but none of the varnish adheres to the finger. The varnish 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 not considered to have satisfactorily dried hard.

(*h*) Toughness.—Flow the varnish on one side of a dry steel plate that has previously been cleaned of all scale, rust, and grease. This plate should be about 0.4 mm (0.016 inch) thick; and 10 by 15 cm (4 by 6 inches) will be found of convient size. Let it dry in a vertical position not in the direct rays of the sun, in a well-ventilated room at a temperature not below 21° C (70° F) for a period of not less than six days. Now bring the test piece to a temperature between 21 and 24° C. (70 to 75° F) and with the varnish film on the outside, bend rapidly over a rod 3 mm (⅛ inch) in diameter. The film must show no evidence of cracking or flaking.

(*i*) Working Properties.—A clean piece of sheet steel similar to that used for testing the toughness of the varnish shall be used for determining the working properties. The plate shall be thoroughly cleaned of all grease and rust and dried. It shall then be laid in a horizontal position and one coat of the varnish applied by brushing. The varnish shall work easily under the brush, showing no tendency to draw or pull, and shall flow out to a smooth, glossy, jet-black film, free from brush marks, blisters, pinholes, or other defects.

Test pieces prepared in the above manner and allowed to dry in a horizontal position, not in the direct rays of the sun, in a well-ventilated room, at a temperature not below 21° C (70° F) for a period of not less than 6 days, shall be used for testing the resistance of the varnish to cold water, lubricating oil, and mineral acids.

(*j*) RESISTANCE TO WATER.—A test piece, prepared and dried as under (*i*), shall be inclined at an angle of 45° to the vertical, and a gentle stream of cold tap water with a temperature of about 25° C (77° F) allowed to flow for 18 hours down the middle of the varnished surface. After wiping off with a soft cloth or chamois skin any deposit due to the tap water, the varnish must show no whitening, dulling, softening, or other visible defects.

(*k*) RESISTANCE TO LUBRICATING OIL.—A test piece prepared and dried as under (*i*) shall be laid flat, and in at least two different places several drops of lubricating oil allowed to stand in contact with the film for 6 hours. After wiping off the oil with cotton waste, no softening or other deterioration of the film due to the lubricating oil shall be perceptible.

(*l*) RESISTANCE TO MINERAL ACID.—Test pieces prepared and dried as under (*i*) shall be laid flat, and in different places several drops each of sulphuric acid (specific gravity 1.3), nitric acid (specific gravity 1.22), and hydrochloric acid (specific gravity 1.09) allowed to remain in contact with the film for 6 hours. The acid spots shall be covered with watch glasses to prevent evaporation. After washing off the acids and drying with a soft cloth the varnish must show only very slight hardening and dulling, and the metal beneath shall show no corrosion. (In order to examine the metal the varnish film can be removed with chloroform or carbon bisulphide.)

WASHINGTON, September 27, 1920.

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