U. S. Gov't
Master
Specification
No. 375

DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
George K. Burgess, Director

CIRCULAR OF THE BUREAU OF STANDARDS, No. 302

[Issued February 9, 1926]

UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR SHELLAC, FLAKE ORANGE

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 375

This specification was officially promulgated by the Federal Specifications Board on January 25, 1926, for the use of the departments and independent establishments of the Government in the purchase of shellac, flake orange.

[The latest date on which the technical requirements of this specification shall become mandatory for all departments and independent establishments of the Government is April 26, 1926. They may be put into effect, however, at any earlier date, after promulgation.]

CONTENTS

I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

II. TYPES

Orange shellac shall be furnished in one of the four types as follows: A, B, C, or D.

III. MATERIAL

Orange shellac shall be the manufactured product of stick lac (the secretion of the Tacchardia Lacca) freed from most of the lac dye and prepared in flake form. Seed, garnet, and button lac are not admissible under this specification.

IV. GENERAL REQUIREMENTS

Unless specifically waived in the contract, flake orange shellac must be "free"; that is, any flakes that may have stuck together must separate readily under hand pressure.

V. DETAIL REQUIREMENTS

Flake orange shellac shall conform to the requirements for the respective types given below:

	A	B	C	D
Iodine number (maximum)	18.0	18.0	18.0	24.5
Matter insoluble in hot 95 per cent alcohol (maximum) per cent	1.75	2.50	3.00	3.00
Moisture and volatile matter (maximum) do	2.0	2.0	2.0	2.0
Matter soluble in water (maximum) do	.5	.5	.5	.5
Wax (maximum) do	5.5	5.5	5.5	5.5
Ash (maximum) do	1.0	1.0	1.0	1.0

Color when specified shall be no darker than a sample mutually agreed upon by the buyer and seller when tested as per Section VI 2 (*g*).

VI. METHODS OF 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 specification.

1. Sampling.—It is mutually agreed by the buyer and seller that double handfuls shall be taken from 10 per cent of the packages taken at random from the delivery. The composite samples taken as above shall be thoroughly mixed, quartered down to approximately 3 pounds and divided into three equal portions which shall be separately sealed and marked. The inspector shall send one of the sealed samples to the testing laboratory, retain one for umpire test in case of dispute, and, if requested, shall deliver the third to the seller.

2. Laboratory Examination.—Thoroughly mix the sample of shellac to be tested and grind not less than a 100 g portion to entirely pass a 20-mesh screen and keep in a tightly stoppered container. Make the necessary determinations on this sample with as little delay as possible.

(*a*) *Iodine number.*—Introduce 0.2 g of ground shellac into a 250 cc dry bottle of clear glass with a ground-glass stopper, add 20 cc of glacial acetic acid (see Reagents) and warm the mixture gently on top of a hot-water bath until solution is complete (except for the wax). A pure shellac is rather difficultly soluble; solution is quicker according to the proportion of rosin present. Add 10 cc of chloroform (see Reagents) and cool the solution to 21.5 to 22.5° C. The

bottles should be allowed to stand half immersed in a shallow pan of water, well insulated or equipped with a suitable thermostat, at least 30 minutes at 21.5 to 22.5° C. before the Wijs solution (see Reagents) is added. Add 20 cc of Wijs solution (which shall be at a temperature of 21.5 to 22.5° C.) from a pipette, having a rather small delivery aperture (about 30 seconds). Close the bottle, place it back into the pan of water, and note the time. The bottles must be kept half immersed in water at 21.5 to 22.5° C. during the one hour that the shellac is exposed to the Wijs solution. Agitate the bottles occasionally during that hour.

NOTE.—If a number of samples are being run, at least five minutes should be allowed between additions of the Wijs solution.

After exactly one hour add 10 cc of freshly prepared 10 per cent potassium iodide water solution (see Reagents), washing into the bottle any Wijs solution on the stopper with the same. Titrate the solution immediately with the 0.1 *N* sodium thiosulphate solution (see Reagents), allowing the solution to run in slowly (about 25 to 30 cc) with vigorous shaking until the solution becomes a straw color. Now add 15 cc of freshly prepared starch solution (see Reagents) and finish titrating. The end point is sharp, as the reaction products of shellac remain dissolved in the chloroform; any color returning after one-half minute or so is disregarded.

A blank determination shall be run at the same time on the reagents. The blank is necessary on account of the well-known effect of temperature changes on the volume and possibly loss of strength of the Wijs solution.

From the volumes of thiosulphate solution required for the sample and the blank, respectively, and the iodine value of the thiosulphate solution, calculate the iodine number (centigrams of iodine per gram of sample) of the shellac.

The analyst should run in parallel a sample of pure shellac of known iodine number as a check upon the reliability of the Wijs and thiosulphate solutions.

(*b*) *Matter insoluble in hot 95 per cent alcohol.*[1]—Weigh accurately 2 g of the sample, transfer to a small beaker and heat the shellac with 25 cc of 95 per cent alcohol (see Reagents). Prepare a Gooch crucible with an asbestos pad in the customary manner and dry to constant weight. Arrange the crucible for filtration by suction and pour sufficient boiling alcohol through it to thoroughly heat the crucible.

NOTE.—A cold crucible will congeal the wax and hinder filtration.

Immediately filter the boiling shellac solution, using suction, transfer the insoluble matter from the beaker to the crucible, using

[1] The continuous extraction method (A. S. T. M. Standards, 1924, p. 829, Method D 29-24) may be used.

a "policeman" if necessary and a wash bottle containing hot alcohol. Wash the residue in the crucible with boiling alcohol five times, nearly filling the crucible each time.

NOTE.—It will be necessary to shut off the suction momentarily to fill the crucible.

Wash off any film of shellac on the sides or bottom of the crucible with hot alcohol and dry to constant weight in an oven at 105 to 110° C. The weight of the residue in the crucible, multiplied by 100 and divided by the weight of the sample, is the percentage of material insoluble in hot alcohol.

(*c*) *Moisture and volatile matter.*—Weigh accurately approximately 5 g of the sample and heat in a flat-bottomed dish about 4 inches in diameter in a well ventilated air bath for three to six hours at 38 to 43° C. Do not allow the temperature to rise above 43° C.

NOTE.—With poorly ventilated ovens the drying may take much longer. Completeness of drying should be ascertained by continuing the treatment to constant weight. Calculate the percentage loss in weight.

(*d*) *Matter soluble in water.*—Weigh 10 to 25 g of sample accurately and stir thoroughly with 100 cc of distilled water in a suitable sized flask or beaker. Cover with a watch glass and allow to stand at room temperature (approximately 21° C.) for four hours, stirring occasionally. Decant the water through a 12.5 cm filter paper into a weighed evaporating dish, washing the shellac and paper with at least 50 cc more of water. Evaporate the water and dry the extract at 105 to 110° C. for one hour or more to constant weight. Cool, weigh, and calculate the percentage of matter soluble in water.

(*e*) *Wax.*—Dissolve 10 g of the sample in 200 cc of 95 per cent alcohol (see Reagents) with agitation at a temperature of 24° C.± 3° C. Allow the solution to stand for several hours, preferably overnight, in a tall cylindrical vessel maintained at a temperature of 24° C.±3° C. until the wax has settled to a small layer a tthe bottom of the container. Decant the clear solution through a 12.5 cm folded filter paper, taking care not to disturb the wax layer. Finally, transfer the wax to the filter, using 25 cc of alcohol at the prescribed temperature. Wash the container and the wax with four 15 cc portions of alcohol. The final washings should be colorless. Allow most of the alcohol to evaporate from the filter paper and dissolve the wax by pouring successive small portions of boiling chloroform (see Reagents) through the paper. Also wash the container with boiling chloroform and pour on the filter. Collect the filtrate, which should contain all the wax, in a tared dish, evaporate the chloroform and dry to constant weight at 105 to 110° C. Weigh the wax residue and calculate the percentage of wax.

The removal of the wax from the filter paper may also be accomplished in any suitable continuous extraction apparatus.

(*f*) *Ash.*—Transfer an accurately weighed portion of from 2 to 3 g of the sample to a weighed porcelain crucible and ignite at as low a temperature as possible until all organic matter has been destroyed. Cool, weigh, and calculate the percentage of ash.

(*g*) *Color.*—Digest a weighed portion of the sample in a closed container with twice its weight of cold 95 per cent alcohol (see Reagents), shaking at intervals until the shellac is entirely "cut." Prepare in the same manner a shellac varnish with exactly the same proportion of resin and alcohol, using the sample of shellac mutually agreed upon for color by the buyer and seller. Compare the color of these two varnishes in clear glass test tubes of the same diameter. The color of the sample under examination shall not be darker than that of the sample mutually agreed upon for color by the buyer and seller.

(*h*) *Appearance.*—Examine the shellac as received in the laboratory and note whether the material is "free" or not. If flakes are stuck together, ascertain whether hand pressure will separate them or not.

3. Reagents.—(*a*) *Acetic acid.*—99 per cent acetic acid having a melting point of 14.8° C. free from reducing impurities, as shown by its action on bichromate-sulphuric acid mixture.

(*b*) *Wijs solution.*—Dissolve 13 g of pure iodine (resublimed grade) in a liter of acetic acid (see (*a*) above) using gentle heat if necessary. Cool and determine the strength by titration with thiosulphate solution (see (*d*) below). Set aside 50 to 100 cc of the solution and introduce dry chlorine gas into the remainder until a characteristic color change occurs and the halogen content has been doubled. By titration ascertain if the halogen content has been more than doubled, and, if so, reduce it by adding a requisite quantity of the iodine-acetic acid solution. The final product should contain an amount of iodine and chlorine corresponding to iodine-monochloride; a slight excess of iodine does no harm, but any excess of chlorine over the theory for ICl must be avoided.

(*c*) *Chloroform.*—U. S. P. chloroform should be used.

(*d*) *Sodium thiosulphate solution.*—Dissolve pure sodium thiosulphate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g crystallized sodium thiosulphate will be present in 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. (See Analytical Chemistry, Treadwell-Hall, vol. II, 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 strength. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(*e*) *Starch solution.*—Dissolve 0.2 g of starch per 100 cc of distilled water and boil.

(*f*) *Potassium iodide solution.*—Dissolve potassium iodide free from iodate in distilled water in the proportion of 1 part salt to 10 parts of water.

(*g*) *Ninety-five per cent alcohol.*—Either 95 per cent ethyl alcohol or 95 per cent specially denatured alcohol, U. S. Internal Revenue Bureau Formula No. 1 or No. 30. (Formula No. 1 calls for the addition to 100 gallons ethyl alcohol (190 proof) of 5 gallons of approved wood alcohol, the wood alcohol to be subject to the same specifications as are imposed upon wood alcohol used in completely denatured alcohol.) (Formula No. 30 calls for the addition to 100 gallons of pure ethyl alcohol of 190 proof of 10 gallons of pure methyl alcohol, which methyl alcohol is to have a specific gravity of not more than 0.810 at 60° F.)

VII. PACKING AND MARKING

There are no packing and marking requirements applicable to this specification.

VIII. NOTES

This specification covers the requirements for orange flake shellac for use in ship bottom paints and in the preparation of orange shellac varnish. In general, type A will include the grades of shellac known in the trade as "Double triangle G," "Diamond I," "Superfine," and the highest grades "D. C." and "V. S. O."

In general, type B will include the grades considered in the trade as lower than "superfine," but higher than "pure T. N.," such as "fine," "good," and "heart."

Type C represents the grade known in the trade as "pure T. N." This material is rosin free, but is darker and contains more insoluble material than types A or B.

Type D represents the grade known in the trade as "U. S. S. A. T. N." It usually contains rosin up to a maximum of 3 per cent, and, in addition, it is generally darker and contains more insoluble material than types A or B, but is usually a little lighter in color than type C.

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